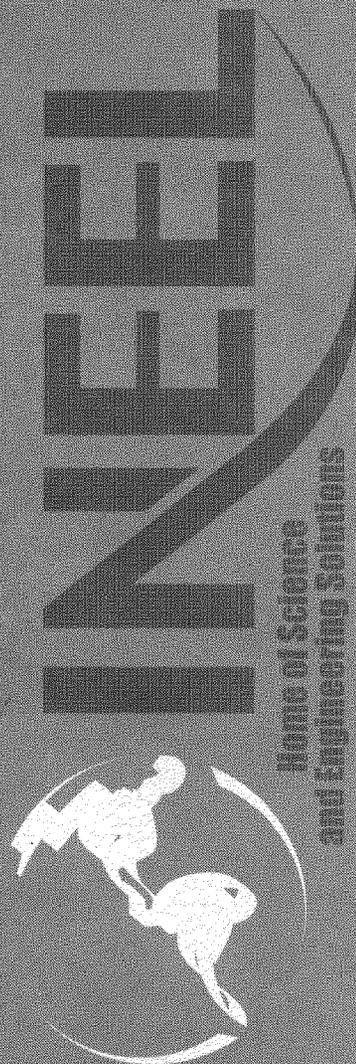


***Final Results Report,
In Situ Grouting Technology
for Application in Buried
Transuranic Waste Sites***

***Volume 1, Technology Description
and Treatability Study Results for
Operable Unit 7-13/14***

*Guy G. Loomis
James J. Jessmore
Jerry R. Weidner
Christopher M. Miller
Allen L. Sehn*

August 2002



DISCLAIMER

This information was prepared as an account of work sponsored by an agency of the U.S. Government. Neither the U.S. Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. References herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the U.S. Government or any agency thereof. Views and opinions of authors expressed herein do not necessarily state or reflect those of the U.S. Government or any agency thereof.

**Final Results Report,
In Situ Grouting Technology for Application
in Buried Transuranic Waste Sites**

**Volume 1, Technology Description and
Treatability Study Results for Operable Unit 7-13/14**

**Guy G. Loomis
James J. Jessmore
Jerry R. Weidner
Christopher M. Miller^a
Allen L. Sehn^a**

August 2002

**Idaho National Engineering and Environmental Laboratory
Idaho Falls, Idaho 83415**

**Prepared for the
U.S. Department of Energy
Assistant Secretary for Environmental Management
Under DOE Idaho Operations Office
Contract DE-AC07-99ID13727**

a. University of Akron in Akron, Ohio.

Final Results Report, In Situ Grouting Technology for Application in Buried Transuranic Waste Sites

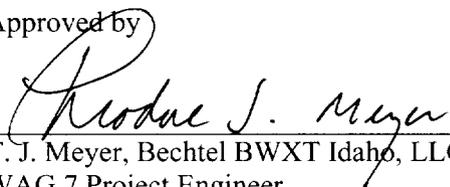
Volume 1, Technology Description and
Treatability Study Results for Operable Unit 7-13/14

INEEL/EXT-02-00233

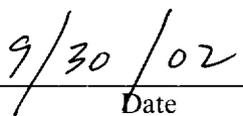
Revision 0

August 2002

Approved by



T. J. Meyer, Bechtel BWXT Idaho, LLC
WAG 7 Project Engineer



Date

ABSTRACT

This report discusses in situ grouting in two volumes. Volume 1 summarizes the technology and presents results of a treatability study conducted by the Idaho National Engineering and Environmental Laboratory. Volume 2 gives the results of analytical calculations on the long-term durability of monoliths created by in situ grouting and on the long-term performance of a treated buried waste site. Volume 2 uses analytical techniques and support data discussed in Volume 1. In situ grouting involves the injection of grout at high pressure (jet grouting) into a buried waste site. The grouting action creates a solid monolith with reduced permeability and increased subsidence control. Testing described in Volume 1 involves three phases: bench testing, implementability testing, and full-scale field testing. The treatability study is being performed to determine the efficacy of using in situ grouting as a buried waste treatment at the Idaho National Engineering and Environmental Laboratory's Waste Area Group 7, Operable Unit 13/14, located in the Subsurface Disposal Area of the laboratory's Radioactive Waste Management Complex. Data presented in this report will be used in the Waste Area Group 7-13/14 Comprehensive Remedial Investigation/Feasibility Study, which is part of the Comprehensive Environmental Response, Compensation, and Liability Act process for Superfund sites such as the Subsurface Disposal Area.

EXECUTIVE SUMMARY

This report discusses in situ grouting in two volumes. Volume 1 summarizes the technology and presents the results of testing conducted for an in situ grouting treatability study performed by the Idaho National Engineering and Environmental Laboratory (INEEL). Volume 2 contains the results of analytical calculations estimating the long-term durability of the monoliths created by the grouting technology and on the long-term performance of a treated buried waste site relative to containment release. Volume 2 uses analytical techniques and the support data discussed in Volume 1. In situ grouting involves applying grout at high pressure (jet-grouting) to a buried waste site, creating solid monoliths with reduced permeability and increased subsidence control. Testing described in this volume involves three phases: bench testing, implementability testing, and full-scale field testing. The treatability study is being performed to determine the efficacy of using in situ grouting as a buried waste treatment at the Waste Area Group 7 (WAG-7), Operable Unit 13/14, located in the Subsurface Disposal Area (SDA) of the INEEL's Radioactive Waste Management Complex (RWMC). Data presented herein will be used in the WAG 7-13/14 Comprehensive Remedial Investigation/Feasibility Study, which is part of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) process for Superfund sites such as SDA.

In situ grouting creates solid monoliths from unconsolidated buried waste sites using jet grouting of specialty grouts. The technology was developed for remediation of the transuranic pits and trenches at the INEEL. However, the technology has also been applied to stabilize contaminated soil sites, specifically the Acid Pit at the SDA. The process involves high-pressure (nominally 400 bar [6,000 psi]) injection of specialty grouts directly into the waste via a drill string with injection nozzles at the bottom. The drill string is driven into the waste on a nominally 50-cm (20-in.) triangular pitch, and the rotating drill string is withdrawn in precise increments creating a column of grouted waste. By interconnecting the columns, a solid monolith is formed that eliminates the potential for subsidence, reduces the local hydraulic conductivity, and, by using specialty grouts, can retard or eliminate the release of contaminants to the ground water. The unique feature of the in situ grouting technology is the inclusion of a contamination control system involving a thrust block and drill string shroud that contains any transuranic contaminants mobilized during grouting.

Specific results of the in situ grouting treatability study are given below and include those from bench, implementability, and field studies. Bench studies involved a complicated testing protocol designed to first choose three grouts from six candidates for use in implementability testing that involved full-scale field grouting equipment. Bench data are also obtained to support monolith durability estimates and transport modeling efforts discussed in Volume 2. Implementability testing compared three grouts, and one was chosen for use in field testing. Finally, a limited field demonstration of the technology was performed in which contamination control data are discussed in detail.

Six candidate grouts for bench testing included TECT HG™ (cementitious), U.S. Grout (cementitious-pozzlonic), GMENT™-12 (cementitious-pozzlonic), Enviro-Blend® (phosphate based), Waxfix™ (molten paraffin based), and Saltstone (mostly pozzlonic). All of these grouts displayed the potential to be jet-groutable. In fact, TECT HG and Waxfix had been applied before at the INEEL. The bench tests first screened the grouts for basic grouting parameters including gel time, temperature of set, viscosity, and density. The only grout eliminated due to an early gel time (less than 2 hours) on this initial screening was Saltstone. A gel time of 2 hours was chosen to avoid “flash setting” of the grout in pumping equipment.

It was concluded that with some reformulation effort (beyond the scope of this study) that the Saltstone could be considered for application. Waxfix had special screening criteria because criticality

concerns required that a neutron absorber be uniformly distributed in the grout during a multiday cooldown period. Specifically, there was a requirement that boron-10 be suspended in a cooling column of Waxfix at a concentration of 1 g/L. Using a glycerin solution of sodium tetraborate as the source of boron-10, there was almost a complete settling of the boron during cooldown, which eliminated Waxfix from further consideration in this study. However, subsequent analytical studies show that a uniform suspension is possible. Including Waxfix as a candidate grout is desirable in that past studies demonstrated that extensive penetration of grout into the soil/waste matrix is achievable. Additionally, Waxfix could be applied as an aid to retrieving buried transuranic waste as a contamination control measure during waste excavation and subsequent processing. This initial screening left TECT HG, U.S. Grout, Enviro-Blend, and GMENT-12 for further testing.

These four remaining grouts were tested as a neat grout for compressive strength, hydraulic conductivity, leaching using American Nuclear Society (ANS) 16.1, measurement of oxidation-reduction potential (eH) and acid-base properties (pH) during the ANS 16.1 leaching procedure, tensile strength, and effect on compressive strength due to the presence of interferences (commonly seen interferences that might affect grout curing in the transuranic pits and trenches include organic sludge, soil, and nitrate salts). Based on the interference tolerance testing, a selected weight-percent mixture of neat grout and the interferences (either 50 wt% soil, 9 wt% organic, or 12 wt% nitrate salts) was formulated and tested for ANS 16.1 leaching with pH and eH measured for each leachate, compressive and tensile strength, and hydraulic conductivity. Other special testing designed for the in situ grouting application included a microencapsulation test in which neat grout is mixed with an organic sludge containing trichloroethane (TCA), trichloroethylene (TCE), carbon tetrachloride (CCl₄), and tetrachloroethylene (PCE). Once mixed, the cured sample is inserted into a special control volume chamber in which the air space is sampled every 10 days for a total of 90 days to establish a rough order of magnitude of the rate of diffusion for the various volatile organic compounds (VOCs). In addition, a special macroencapsulation test was performed in which a cylindrical sample of cured grout with a cavity in the center was filled with organic sludge and sealed at the end to measure the diffusion of VOCs through the matrix. This macroencapsulation test was performed in the same special control column chamber used in the microencapsulation test.

Based on an evaluation of the results from the testing protocol discussed above, three of the grouts were further down-selected including TECT HG, U.S. Grout, and GMENT-12 (Enviro-Blend was eliminated). The three chosen grouts displayed excellent compressive and tensile strength characteristics, with neat grout values in the 161–466 bar (2,500–7,000 psi) range for compressive strength. Enviro-Blend displayed poor cured strength only in the “hundreds” of psi range. More importantly, the three grouts displayed good tolerance to interference materials such as organic sludge, soil, and nitrate salts relative to compressive strength. In general, compressive strengths above 1,000 psi were obtained with up to 9 wt% organic sludge, 50 wt% soil, and 12 wt% nitrate salts. On the other hand, Enviro-Blend had no tolerance to the interferences.

ANS 16.1 leach data (as leach index), showed that Enviro-Blend was superior to the three down-selected grouts due to the presence of phosphate in the grout (the three selected grouts displayed ANS 16.1 leach indexes in the range from 10 to 13, with Enviro-Blend measured at 15). During these leaching tests, the eH and pH were measured in the leachate water and found to be in the range of 9.6 to 11.4 for pH and less than 313 mV for eH, suggesting compatibility with the INEEL basic soil. Hydraulic conductivity was measured for the selected grouts in the e-9 cm/s range. For Enviro-Blend, the value was e-7 cm/s, which is two orders of magnitude lower than for the selected grouts. Interestingly, even with the presence of the interferences (nitrate at 12 wt%, soil at 50 wt%, and organic sludge at 9 wt%), hydraulic conductivity for the chosen grouts was not degraded much below the e-9 cm/s range. Based on an elaborate weighting criterion, all of the test results were applied to the four candidate grouts, resulting in a score of 4,184 for TECT HG, 4,150 for U.S. Grout, 3,862 for GMENT-12, and 3,010 for Enviro-Blend.

Based on this ranking system, TECT HG, U.S. Grout, and GMENT-12 were recommended for implementability testing.

Other data obtained during the bench study were results from a specially designed micro and macro encapsulation study for VOC release from the grout. The micro test involved intimately mixing neat grout with organic sludge and measuring the gas release rate of the various VOCs integral to the sludge. In the macro test, a neat grout cylinder with a hollow core was created, and the organic sludge was placed inside the cylinder and sealed in place. For this case, the grout is assumed to be surrounding the sludge, which is a condition observed in previous field-scale demonstrations in simulated waste, and the movement of the VOCs is primarily one of diffusion. Results of this test were surprising in that it was found in the micro encapsulation test that there was only a release of VOC source term of the order of “hundredths” of a percent per 10-day testing period. This suggests that TECT HG, U.S. Grout, and GMENT-12 retard the flow of VOCs for possibly hundreds of years, which may be on the order of natural disintegration of the compounds in nature.

Volume 2 of this report takes the detailed data from the bench studies and formulates predictive models of contaminant transport and mechanisms of transport. Data important to these studies are the leach indexes, the physical properties, and the eH and pH of the leachate water in the ANS 16.1 testing.

Implementability full-scale field testing was performed to down-select the three grouts recommended from the bench study. These studies also gave performance data such as mixability, groutability (ease of jet grouting) and cleanup properties for those grouts that had never been grouted before. This testing involved creating triplex columns on a 50-cm (20-in.) triangular pitch in INEEL-like silty-clay soils. During this testing, it was demonstrated with full-scale field equipment that the three grouts recommended from the bench testing (TECT HG, U.S. Grout, and GMENT-12) could be applied for in situ grouting. All three grouts could be mixed and delivered at 400 bar (6,000 psi) via jet grouting. U.S. Grout and GMENT-12 required using a 2.4-mm nozzle to achieve the desired (ease of mixing) 400 bar (6,000 psi), and the third grout could pressurize the system using a 3-mm nozzle. The size of the nozzle is important in that the larger the nozzle the less prone to plugging from small debris in the system or the effects of filter-caking in a stagnant condition. Also demonstrated at the implementability testing was the ability to place a 7-cm (2.75-in.) polyethylene rod into a just-grouted hole for proof of concept that removal of the rod after grout cure would result in a complete borehole for performing hydraulic conductivity tests.

GMMENT-12 was chosen from the three grouts based on factors such as basic cost, ease of mixing and cleanup of the grout, minimized grout returns in creating a triplex column, and formation of the monolith. All three grouts displayed the capability to be jet grouted and form solid stand-alone monoliths in an INEEL-type soil condition (tightly packed silty-clay soils). This soil condition is thought to be the most restrictive for jet grouting due to a lack of voids compared with the buried debris case, where the voids are much increased over a soil-only condition. U.S. Grout had noticeably higher grout returns due to a lower specific gravity than the other grouts (U.S. Grout 1.6, GMENT-12 1.84, TECT HG 2.16). After grouting two holes with U.S. Grout, the space under the simulated thrust block was filled with grout and the third hole could not be grouted. With a lower specific gravity grout, there is not as much kinetic energy imparted to the surrounding medium as with the higher specific gravity grouts, the velocity of the grout being the same. An evaluation of ease of mixing and cleanup properties for TECT HG and GMENT-12 showed GMENT-12 with a slight edge; therefore, GMENT-12 was selected as the single grout to be carried into field testing.

During the field test, a total of 12 holes were grouted using the thrust block/shroud concept. This concept involves a glovebox-like structure placed over the pit called a thrust block. Plastic sleeves are attached to the thrust block for each predetermined hole. Prior to grout injection, the plastic sleeves are

attached to the drill string shroud, forming a seal. The drill string is inserted through a plastic diaphragm in the thrust block to allow drilling, then grouting. When finished, the drill string is withdrawn, and the plastic sleeve is “J” sealed using duct tape.

Even though an injury accident occurred after successfully grouting only 12 holes, considerable data on using the thrust block concept and actual data on the capability of the thrust block to contain the terbium tracer were obtained. It was planned to grout 114 holes and perform an elaborate excavation of the monolith; however, the project was not completed. The main reason was the need to redirect remaining budget for more pressing INEEL projects. At the time, the cost of restart would have been prohibitive, requiring new pressure relief systems and verification of operability, new procedures, and a vigorous operational readiness process.

As the test proceeded, operating procedures were perfected for using the thrust block concept. Since a trickle flow of grout through the nozzles had been utilized on all other grouting studies at the INEEL, this test represented the first attempt at grouting without allowing a continuous flow. During implementability testing, it was observed that following discontinuation of high-pressure flow, the drain of fluid in the drill stem was noted to be on the order of minutes. In fact, this knowledge was applied for the first two holes. For the first hole, the process worked as planned. When moving from the second hole to the third hole, the sack formed by the “J seal” twist and tape action on the thrust block sleeve filled with draining grout. Gravity pulled the sack full of fluid off the stinger, and the potentially terbium-contaminated neat grout flowed onto the top of the thrust block. This led to measurable terbium tracer on some of the thrust block smears.

This occurrence led to two corrective actions. One action was to separate the high-pressure hose at the fitting near the weather structure wall and relieve the vacuum in the drill stem (caused by the draining fluid that holds up material in the drill stem). In fact, compressed air was introduced to blow the grout out through the nozzles. The other action was to provide a separate bag at the bottom of the sack to help contain any dripping that may occur at the J seal. In an actual radioactive application, however, it would be desirable to have a special self-cleaning relief valve in the system to relieve the vacuum and the possible automatic actuation of compressed air to blow out the remaining grout.

Another major issue was the amount of nozzle plugging and time spent using rotopercussion to unstick plugged nozzles. This issue may be related to the grout chosen for the test (GMENT-12). In prior studies using TECT HG, there was an allowed trickle flow for most of the grouting; however, there were times when the grout was stopped and startup was accomplished without significant plugging of the nozzles.

Prior to discontinuation of testing, all systems were working as planned, with minor modifications required. These modifications include the need for a better view of the void space under the thrust block using remote TV cameras. Another minor modification to the thrust block design would be to provide a deeper Lexan well in that the TV cameras were not deep enough in the various camera wells to get a perfect wide-angle view of the spaces under the thrust blocks. Another minor modification would be to provide a hard pipe for the inlet and outlet of the thrust block high-efficiency particulate air filtration system to avoid collapse of the hose. It was obvious that a better weld connection of the shroud to the top bracket was required as well as an engineered twist in the shroud material itself to avoid the rotating drill steel from touching the inner shroud as the drill string was inserted and withdrawn from the test pit.

During the test, grout was mixed in Idaho Falls at a Ready Mix plant and transported 80 km (50 miles) to the INEEL Cold Test Pit South three times a day (3,024 L [800 gal] per trip). This distance led to poor utilization of mixed grout in that many loads were dumped unused, having begun to set before they could be injected. When the grout actually arrived at the Cold Test Pit, the grouting system had not

been functioning for the entire 2 hours, and a full truck was still available. The obvious solution is to utilize a mobile ready-mix plant at the Cold Test Pit.

During the limited field demonstration, several lessons were learned. Some of the lessons were related to operations of the system and others were system related due to the experienced catastrophic failure of a high-pressure fitting.

During operations in the field, the most basic problem with the system was nozzle plugging related to the fact that no trickle flow of grout was allowed by the thrust block/shroud contamination control system. Because this system disallows a trickle flow of grout (in past studies trickle flow was the technique to keep the nozzles clear), a completely new design of a vacuum relief system within the drill string is needed. This vacuum relief system is needed to allow complete draining of the drill string of neat grout immediately after grouting and prior to moving the drill string to a new hole. Following grouting, simply letting the drill string drain its fluid was not sufficient in that the vacuum created by partially draining the drill string held up fluid that once jostled upon moving the system, causing fluid to drain into the plastic sleeve that had been taped off.

Lessons learned relative to the high-pressure system failure include the following: The grouting subcontractor should install a high-pressure relief valve and a redundant-pressure relief plug to allow emergency bleeding of the system. The primary system would be a valve, and the secondary system could be a simple plug located in an easily and safely accessed area. This emergency plug should allow safe, easy access for tools in the event of a system pressurized by nozzle plugging. Once the plug has been forced open in an emergency, it should be replaced with a new plug and/or fitting. In addition, as part of the emergency procedure, the relief system should be cleaned or replaced to allow proper operation. The grouting contractor should also reevaluate the position of personnel working on the high-pressure equipment and perhaps employ shielding from high-pressure fittings. The grouting subcontractor should check the setting on the automatic shutoff feedback switch prior to each use. This will require a pressurization procedure using water and may require special plumbing to accomplish the testing.

The incident at the Cold Test Pit suggests rapid uncontrolled overpressurization by the triplex pump not shown on the gauges. Specifically, if a nozzle plugs, the operator needs overpressurization protection independent of the gauges. The grouting contractor should use only pressure gauges that operate smoothly at all pressures. It is speculated that the gauge used during the field test sticks at lower pressures and unsticks at higher ones. For instance, the gauge can read 20 bar when it is really 500 bar, about to go to 1,000 bar with a few more strokes of the triplex pump. It is recommended that there be two gauges, one used during low-pressure operations and another used during high-pressure ones. It is suggested that the low-pressure gauge be valved in to operate at low pressures and valved out when operating at high pressures. The most obvious lesson learned is that the grouting contractor should use only rated equipment and fittings such as valves, hoses, and whip-checks. Whip-check and fitting documentation should accompany the fittings and indicate an operating pressure at the design pressure of the pump with an appropriate safety factor. A shield should be installed around the outlet to the high-pressure pump to deflect any future blowout due to catastrophic failure of any fittings in the vicinity of the high-pressure pump.

Data quality objectives were listed in the test plans covering the bench, implementability, and field testing phases of the treatability study. Most of the data quality objectives discussed in the test plans were addressed by the treatability study. However, there are definite, missing gaps in data due to truncation of the field-testing program. All of the data quality objectives were met for the bench and implementability testing phases. With limited testing in the field testing phase, many of the objectives associated with the field testing involving the thrust block and contamination control system were addressed. Overall, the main data quality objective relating to implementability of the in situ grouting process using the thrust

block contamination control system was demonstrated. Only minor design changes are required as discussed above.

The overall grouting process is not as rapid on a time-per-hole basis compared with that expected using alternative grouting concepts (the x-y positional system discussed later in the report). However, the thrust block design could be applied for a variety of applications in buried waste regions. For instance, the thrust block concept could be used to grout a series of interconnected columns (say 10-hole columns) at various regions within a pit to support a cap and leave the thrust block in place. Another application would be to grout small very specific hot spots within a buried waste region. The time issue only becomes important when treating large areas on the order of hundreds of thousands of holes over a 10-year period. Finally, to fully evaluate the missing data quality objectives (those relating to the characteristics of the emplaced monolith like void filling, and monolith durability), would require completion of the grouting in the pit followed by hydraulic conductivity testing and excavation of the monolith with further chemical and physical testing of samples from the resultant monolith.

The following conclusions stem from the in situ grouting studies:

In situ grouting of buried transuranic waste using the thrust block concept is technically feasible at the INEEL with several modifications to the system. Modifications include developing a better pressure relief system to facilitate draining of fluid in the drill stem. Inclusion of an additional plastic shield over the J-seal layer would avoid minor dripping of grout when moving the system. By using double screening in the grout preparation phase, potential debris in the grout that could block nozzles can be avoided. Finally, modifications to the shroud assembly that would prevent wear on the inner shroud and disallow detachment at the upper bracket are required.

A variety of grouts are available for application to jet grouting. Grouts tested in this study that were shown to be jet groutable include TECT HG, U.S. Grout, and GMENT-12. With minor modifications, the paraffin-based Waxfix and Saltstone grout could most likely also be candidate grout materials. By reformulation of American Minerals, Inc.'s Enviro-Blend grout, it too could be considered a candidate grout.

Bench studies of U.S. Grout, TECT HG, Enviro-Blend, and GMENT-12 show excellent retention of constituent elements and tracer materials during ANS 16.1 leach testing. Bench studies suggest that U.S. Grout, TECT HG, and GMENT-12 show a strong tolerance to interferences commonly occurring within the buried waste including organic sludge (up to 9 wt% tolerance), soil (up to 50 wt% tolerance), and nitrate salts (up to 12 wt% tolerance). Bench studies of VOC retention show that there is only a few hundredths of a percent of source term lost per 10-day interval in special microencapsulation testing involving cured mixtures of neat grout and 9 wt% organic sludge (for U.S. Grout, TECT HG, and GMENT-12).

The contamination control features of the thrust block/drill string shroud concept worked as planned. There was no terbium tracer spread to the high-volume air monitors, even though neat grout with potential terbium contamination was spilled onto the top of the thrust block (when the sack containing grout drippings fell off the drill string stinger). Inductively coupled plasma-mass spectroscopy of smears taken on the top of the thrust block following cleanup of the spill showed terbium contamination. Even with extensive foot traffic and movement of the drill rig over the spill location, there was no spread to the high-volume filters. It is hypothesized that the grout locks the tracer material up in larger, less easily aerosolizable particles. It is speculated that if the bag had not dropped, there would only have been terbium tracer within the containment of the drill string shroud and under the negative pressure of the thrust block in the grout returns.

Examination of the limited monolith created by grouting 12 holes showed a solid monolith with the usual inclusions of compacted clay soil. Embedded in the monolith was a drum containing nitrate salts partially filled with grout. Parts of the drum were filled with neat grout (where the voids were), and voids in the nitrate salt material had cured grout.

The following recommendations grow out of results of the in situ grouting studies:

There should be a tradeoff study comparing the thrust block concept and the x-y positional system remote grouting idea. The x-y positional system has been proposed as an alternative grout delivery system, which involves the drill rig mobilized by a remotely controlled gantry crane. In principle, the x-y positional system answers all the problems encountered with the thrust block concept. With the x-y positional system, a trickle flow of grout can be allowed and there are no real limitations on grout returns, which improves the chances of complete pit void filling. In addition, the x-y positional system has more flexibility when encountering large hard objects that might refuse the drill bit.

CONTENTS

ABSTRACT.....	v
EXECUTIVE SUMMARY.....	vii
ACRONYMS.....	xix
1. INTRODUCTION.....	1
2. IN SITU GROUTING AND PAST EXPERIENCE	2
2.1 Technology Description.....	2
2.2 Grouting with Single-Component Material	3
2.3 Grouting with Dual-Component Material.....	8
2.4 Grouting as a Pretreatment Prior to Retrieval.....	10
2.4.1 Retrieval of a Monolith Grouted with Portland Cement	10
2.4.2 Retrieval of a Monolith Grouted with Acrylic Polymer.....	11
2.4.3 Retrieval of a Monolith Grouted with Waxfix	11
2.5 Stabilization of the Acid Pit Using Jet Grouting.....	12
2.6 Contamination Control During Grouting.....	12
3. BENCH STUDIES	13
3.1 Background.....	13
3.1.1 TECT HG	14
3.1.2 Saltstone	14
3.1.3 Tank Closure Grout.....	14
3.1.4 Waxfix	14
3.1.5 U.S. Grout (Ultrafine Grout)	14
3.1.6 Enviro-Blend—American Minerals (Phosphate)	14
3.2 Test Objectives	15
3.2.1 Bench Testing.....	15
3.2.2 Special Testing	17
3.3 Bench Testing Protocol.....	18
3.4 Screening Test Results.....	20
3.5 Screening of Grout/Interference Mixtures	21
3.5.1 Soil as an Interference	22
3.5.2 Organic Sludge as an Interference.....	22

3.5.3 Nitrate Salt as an Interference.....	23
3.6 Testing of Neat Grouts.....	23
3.6.1 Physical and Chemical Testing of Neat Grouts.....	23
3.6.2 Physical and Chemical Testing of Interference/Grout Mixtures.....	30
3.7 VOC Encapsulation Testing.....	33
3.7.1 Microencapsulation Testing.....	33
3.7.2 Macroencapsulation Testing.....	35
3.8 Special Testing for Wax-Based Grouts.....	38
3.9 Use of Powdered Activated Carbon in Grouts.....	39
3.10 Down-Selection of Grouts for Implementability Testing.....	40
3.10.1 Monolith Implementability Variables (Weighting Factor = 5).....	41
3.10.2 Physical Stabilization of the Waste Site (Weighting Factor = 4.5).....	41
3.10.3 Long-Term Durability (Weighting Factor = 4).....	43
3.10.4 Waste Site Permeability (Weighting Factor = 4.0).....	43
3.10.5 Chemical Stabilization (weighting factor = 3.0).....	43
3.10.6 Numerical Value of the Down-Selection.....	43
4. IMPLEMENTABILITY STUDIES.....	47
4.1 Introduction/Background.....	47
4.2 Objectives.....	47
4.2.1 Test Objective 8.....	47
4.2.2 Noncritical Test Objective C.....	47
4.3 Test Hardware Description, Procedures.....	48
4.3.1 Hardware.....	48
4.3.2 Procedures.....	50
4.4 RESULTS OF IMPLEMENTABILITY TESTING.....	55
4.4.1 Special Nozzle Testing for U.S. Grout and GMENT-12.....	55
4.4.2 Creating a Triplex Column Using the Thrust Block.....	56
4.4.3 Observation of Halliburton Reading Versus Jean Lutz Reading.....	58
4.4.4 Special Drill Steel Drain Test.....	59
4.4.5 Ease of Mixing of Grout and Clean-out.....	59
4.4.6 Grout Returns Under Thrust Block.....	60
4.4.7 Curing Temperature Results.....	61
4.4.8 Polyethylene Rod Removal Results.....	61
4.4.9 Destructive Examination of Resultant Columns.....	61
4.4.10 Down-Selection of Grouts for Field Testing.....	68

5.	FIELD TESTING	69
5.1	Preparing for Grouting	69
5.1.1	Site Preparation	69
5.1.2	Simulated Waste Preparation	69
5.1.3	Waste Pit Construction	72
5.2	Equipment	76
5.2.1	Grouting System	76
5.2.2	Thrust Block/Contamination Control Features	76
5.3	Grouting Procedures	82
5.3.1	Special Procedures for Plugged Nozzles	87
5.4	Evaluation of Grouting Operations	90
5.4.1	Debris in Delivered Grout	92
5.4.2	Draining, Spillage, Cleaning	92
5.4.3	Excess Time to Perform Grouting/Wasted Grout	93
5.4.4	Plugging of Injection Nozzles	94
5.4.5	Plugging of Injection Nozzle with Lost Time to Relieve Pressure	94
5.4.6	Detachment of Shroud, Wearing of Material on Shroud, HEPA Filter Clogging	94
5.4.7	Collapse of Air Line in Thrust Block HEPA Filter	95
5.4.8	Limited View of Volume Under Thrust Block	95
5.5	Evaluation of Contamination Control	95
5.5.1	Results of Thrust Block and Drill String Smears	96
5.5.2	Results of Contaminant Spread to the HEPA Filter	98
5.5.3	Results of Air Monitoring	98
5.5.4	Results of Contamination in the Grout Returns	100
5.5.5	Comparison of Clean-out to Background Water Samples	101
5.5.6	Discussion of Contamination Control Results	101
5.6	Destructive Examination of the In Situ Grouting Pit	101
6.	LESSONS LEARNED	108
6.1	General Lessons	108
6.2	Lessons from the Accident	109
7.	VENDOR BID ESTIMATE	111
8.	DISCUSSION OF RESULTS	112
8.1	Bench Studies	112
8.1.1	Boron-10 Suspension in Waxfix	112

8.1.2	Too Early Gel Time for Saltstone.....	113
8.1.3	Poor Performance of Phosphate-Based Grout	113
8.1.4	Cracked End Plug for Macroencapsulation Testing	113
8.1.5	Durability	113
8.2	Implementability Testing	114
8.3	Field Testing.....	114
9.	DATA EVALUATION RELATIVE TO DATA QUALITY OBJECTIVES	117
10.	CONCLUSIONS AND RECOMMENDATIONS	129
11.	REFERENCES	131
Appendix A—	Preconceptual Design for In Situ Grouting	A-1
Appendix B—	Data for Interference Tolerance Testing	B-1
Appendix C—	Neat Grout ANS 16.1 Individual Sample Data	C-1
Appendix D—	Cement Chemistry and Durability	D-1
Appendix E—	Grout with Interferences ANS 16.1 Individual Sample Data	E-1
Appendix F—	Carbon Additive to Reduce Migration of VOCs	F-1
Appendix G—	Evaluation of Void Space in a Pit	G-1
Appendix H—	Grouting Pit Construction Details.....	H-1
Appendix I—	Conceptual Design and Cost Estimate.....	I-1
Appendix J—	Grouting Vendor Bid.....	J-1
Appendix K—	Thermocouple Data During Implementability Testing	K-1
Appendix L—	ICP-MS Evaluation of Smears and Air-Filter Samples.....	L-1

ACRONYMS

ANS	American Nuclear Society
ANSI	American National Standards Institute
API	American Petroleum Institute
ASTM	American Society for Testing and Materials
BBWI	Bechtel BWXT Idaho, LLC
BWXT	BWX Technologies, Inc.
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
DE	Department of Energy
DOE	Department of Energy
DOE-ID	Department of Energy Idaho Operations Office
EWR	early waste retrieval
EXT	external
FL	Florida
HEPA	high-efficiency particulate air (filter)
HLW	high-level waste
HVAC	heating, ventilation, and air conditioning
ICP	inductively coupled plasma
ICP-MS	inductively coupled plasma-mass spectroscopy
IDR	initial drum retrieval
INEEL	Idaho National Engineering and Environmental Laboratory
INEL	Idaho National Engineering Laboratory
LI	Leach Index
LLC	Limited Liability Company
NA	not applicable
NRC	Nuclear Regulatory Commission

NUREG/CR	Nuclear Regulatory Commission/contractor report
NW	northwest
PAC	powdered-activated carbon
PCE	perchloroethylene
PCE	tetrachloroethylene
PNNL	Pacific Northwest National Laboratory
PPE	personal protective equipment
PVC	polyvinyl chloride
RADCON	radiological control
RCRA	Resource Conservation and Recovery Act
ROD	Record of Decision
RPD	relative percent difference
RWMC	Radioactive Waste Management Complex
SDA	Subsurface Disposal Area
SE	southeast
SW	southwest
TBD	to be determined
TCA	trichloroethane
TCE	trichloroethylene
TCLP	toxicity characteristic leaching procedure
TV	television
WAG	Waste Area Group
WSRC	Westinghouse Savannah River Company

Final Results Report, In Situ Grouting Technology for Application in Buried Transuranic Waste Sites

Volume 1, Technology Description and Treatability Study Results for Operable Unit 7-13/14

1. INTRODUCTION

This report discusses in situ grouting in two volumes. Volume 1 summarizes the technology and presents the results of testing conducted for an in situ grouting treatability study performed by the Idaho National Engineering and Environmental Laboratory (INEEL). Volume 2 gives the results of analytical calculations on the long-term durability of monoliths created by the grouting technology and on the long-term performance of a treated buried waste site. Volume 2 uses analytical techniques and data discussed in Volume 1.

In situ grouting is the injection of grout at high pressure (jet grouting) to a buried waste site, creating solid monoliths for reduced permeability and increased subsidence control. Testing described in this volume involves three phases: bench, implementability, and full-scale field testing. The overall treatability study is being performed to determine the efficacy of using in situ grouting as a buried waste treatment at the Waste Area Group 7 (WAG-7), Operable Unit 13/14, located at the Subsurface Disposal Area (SDA) of the INEEL's Radioactive Waste Management Complex (RWMC). Data presented in this report will be used in the WAG 7-13/14 Comprehensive Remedial Investigation/Feasibility Study, which is part of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) process for Superfund sites such as the SDA.

Volume 1 of this report includes a summary of past technology development efforts related to in situ grouting as well as findings from the bench testing phase outlined in the bench test plan (Grant et al. 2000) and the implementability and field findings presented in the field and implementability test plan (Loomis 2001). The primary objective of both the bench and implementability studies was to down-select a single grout from a list of products under consideration for the final field test. These tests also provide key data for use in modeling the long-term risk of the resulting monolith created by in situ grouting. In addition to the test results, an estimate is given of the cost of application for the technology for remediation of transuranic pits and trenches.

Bench studies were performed at the University of Akron in Akron, Ohio, and involved a series of screening, physical, and chemical tests on six promising grouts applicable to the jet-grouting process. The study produced important data such as hydraulic conductivity, leaching, and dissolution information on the grouts as well as the tolerance of the grouts to interferences common in SDA buried waste. These data are essential to modeling efforts to predict long-term durability and performance of a grouted buried waste site. In Volume 2 of this report, the data are used to assess these long-term predictions.

The implementability tests were performed at the Richland, Washington, jet-grouting contractor site (Applied Geotechnical Engineering and Construction). In the implementability tests, three grouts down-selected from the bench testing were jet grouted at full scale, and a single grout was recommended for the full-scale field test, which was performed at the INEEL's Cold Test Pit South. This report describes the in situ grouting, provides the bench and field data, and evaluates results. In addition to giving test results, the report considers expected cost of operation in a radioactive mixed waste environment.

2. IN SITU GROUTING AND PAST EXPERIENCE

A series of in situ technologies have been developed for stabilizing mixed waste buried in landfills and contaminated soil sites. The technologies involve nonreplacement jet grouting to create a solid monolith from buried waste such that subsidence abatement is achieved while significantly reducing contamination migration. The monolith is created by jet grouting in a relatively tight pattern directly into the soil/waste matrix. The process has also been applied to buried waste as a pretreatment for eventual waste retrieval. The grout agglomerates fine geological media containing contaminants such that the normally dusty retrieval operation is performed relatively dust free. Full-scale field demonstrations have been performed in numerous simulated mixed waste sites. In addition, the technology has been applied to a contaminated mixed waste site.

Historically, subsurface containment strategies involve creating a vertical barrier wall and in some cases a horizontal barrier under the waste to create a “bathtub” around the contaminated zone. The jet-grouting technology creates a simultaneous horizontal and vertical barrier by forming a solid monolith of the buried waste as a form of in situ remediation. Another option is to retrieve the waste and process it for final disposal separately. The technology of jet grouting to create a monolith supports both of these potential remedial options. For the in situ disposal option, the resultant monolith is immune from subsidence, which can compromise any capping actions. In addition, the monolith lowers the water permeability through the material, thus reducing contaminant transport. If specially formulated grouting agents are used, some contaminants can also be chemically stabilized such that they are not soluble in water and thus not prone to leaching and migration.

Grouting agents considered by the INEEL are those that produce a solid matrix and are chemically neutral in the applied environment, thus representative of natural geological analogs. In addition, for the long-term disposal option, the grouting material is designed to be chemically and thermodynamically stable in the present burial environment, which would include a cap to eliminate freeze-thaw effects. For the in situ disposal option, it is assumed that as long as environmental effects do not change the chemical and thermodynamic equilibrium, the monolith can be considered stable for geological times (thousands to millions of years). This concept is important for transuranic waste with materials that have radiological half-lives on the order of 24,000 years in that modeling for these timeframes appears difficult.

For the retrieval option, the monolith produced by jet grouting causes contaminants and fine soils to be agglomerated into larger less aerosolizable particles, which improves the chances of controlling the spread of contaminants during retrieval and handling, especially for the plutonium-239/americium-241 particles. The first studies (Loomis and Thompson 1995) involved only the grout/retrieval concept. Later studies focused on the monolith concept for the in situ disposal option. Later studies involved testing a variety of grouting materials and strategies (Loomis, Thompson, and Heiser 1995; Loomis, Zdinak, and Bishop 1997). Most recently, the technology was extended to the creation of monoliths in contaminated soil zones (Loomis et al. 1999). Basically, the early studies resulted in an understanding of the need for balance between grout physical characteristics, jet grouting parameters, and resultant monolith development. What follows is a detailed description of how the technology is applied, followed by results of various full-scale studies performed on simulated mixed waste sites called pits.

2.1 Technology Description

The grouting apparatus consists of a CASA GRANDE JET-5 class high-pressure positive displacement pump, low-pressure feed pump with hopper assembly, CASA GRANDE C-6 class track-mounted drilling/grouting rig, and associated high-pressure hoses. A 9-cm-diameter drill stem is driven into the soil waste matrix using rotopercussion. Most insertions into buried debris are

accomplished within 1-2 minutes of drill time. While drilling, a low-volume flow of grout is injected at the bit end of the nozzle to reduce friction and allow easier insertion.

The technology involves driving a drill stem through the waste and injecting grout at 400 bar (6,000 psi) through the rotating drill stem while withdrawing the drill stem in precise increments. Repeated applications on a nominal 50-cm triangular pitch matrix form a series of interconnected columns that eventually turn the soil/waste seam into a solid monolith. Contamination spread at the surface during drilling and grouting is reduced by using a specially designed “thrust block” and shroud assembly. This equipment contains the grout returns due to the high-pressure grouting process shown in Figures 1-3.

Figure 1 shows the basic glovebox nature of the thrust block assembly. Each hole has a diaphragm seal and a double plastic bag plus a metal recessed lid. Additionally, the thrust block is kept at negative pressure by using a high-efficiency particulate air (HEPA) filter also shown in Figure 1. Figure 2 shows the shroud around the drill stem and a plastic glove port in the thrust block with an “o” ring seal on the drill string housing that eliminates the spread of contaminants from the rotating contaminated drill stem. Figure 3 shows that following grouting, the drill steel is withdrawn and the plastic sack is twisted, taped, and cut. Besides providing a volume to collect grout returns during grouting, the thrust block offers a clean area for worker protection and adds a degree of shielding in the case of radioactive waste. In addition, the preformed holes through the thrust block have a pipe wiper material to clean the drill stem of contaminated material during withdrawal. The thrust block concept is applicable for large surface areas or small “surgical” applications of jet grouting.

An alternative concept for applying jet-grouting technology for buried transuranic waste is to use the x-y positional system, in which the drill string is suspended above a bermed area on a bridge crane. Use of this system has more widespread application for either buried transuranic waste or buried low-level but high gamma activity waste. This concept is described in full in Appendix A.

During field studies, a variety of grout materials were injected, including both single and dual materials as well as molten waxes. What follows is a description of test results.

2.2 Grouting with Single-Component Material

A series of materials have been jet grouted while successfully minimizing return of material to the surface. The jet-grouting action mixes the grout with the waste and interstitial soils to create monoliths in the buried wastes (Loomis and Thompson 1995; Loomis, Thompson, and Heiser 1995; Loomis, Zdinak, and Bishop 1997), thus providing for final in situ disposal. The test pits were typically constructed of containerized simulated waste material.

For these studies, the transuranic pits and trenches at the INEEL were used as a model. For these pits, typical waste consists of paper, cloth, wood, metal debris, concrete, asphalt, and various sludges delivered to the INEEL from the Department of Energy’s (DOE) Rocky Flats Plant. The wastes were originally containerized in metal drums and plywood boxes. Some of the waste has been buried in shallow pits for up to 40 years, such that the containers have been destroyed. For testing purposes in the simulated pits, cardboard boxes and drums are used to simulate long-term aging of the containers in an actual pit.

For the single-component materials, grout is forced through two nozzles located 180 degrees apart on the bottom of the drill stem. The nozzles are offset 5 cm to maximize waste coverage in creating a column. At the bottom of the drill stem is usually a conical drive point to facilitate driving the rotating

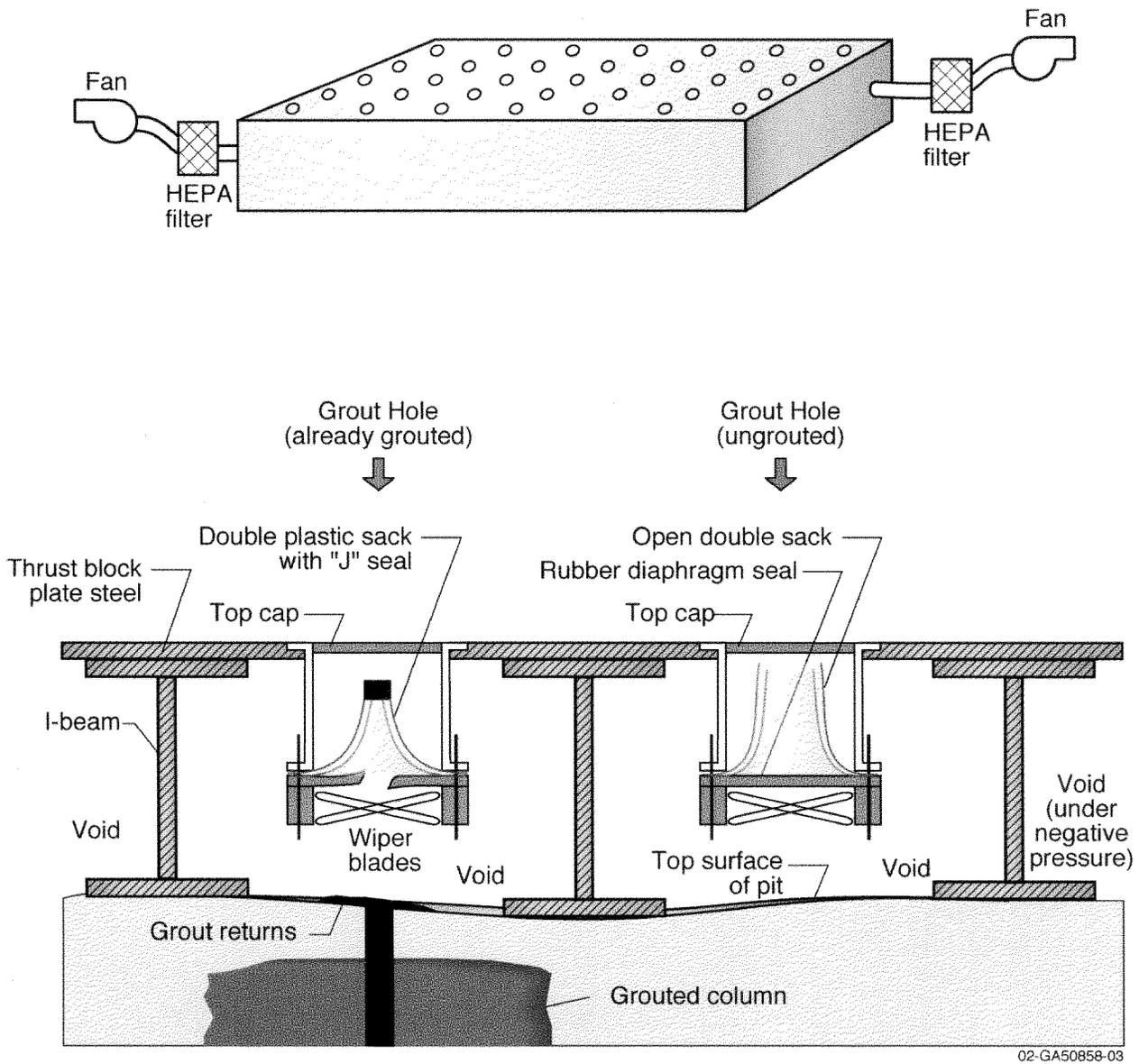
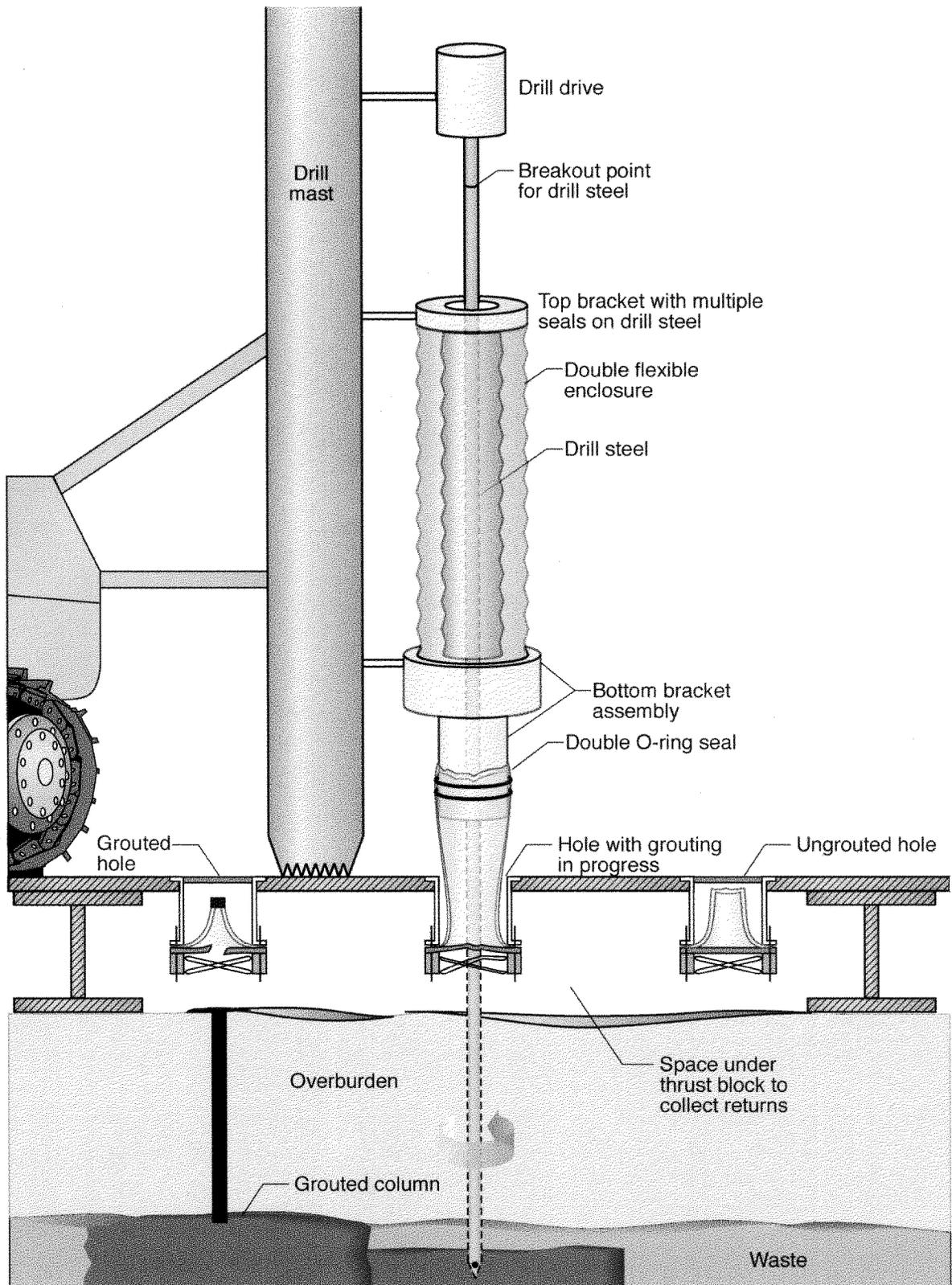
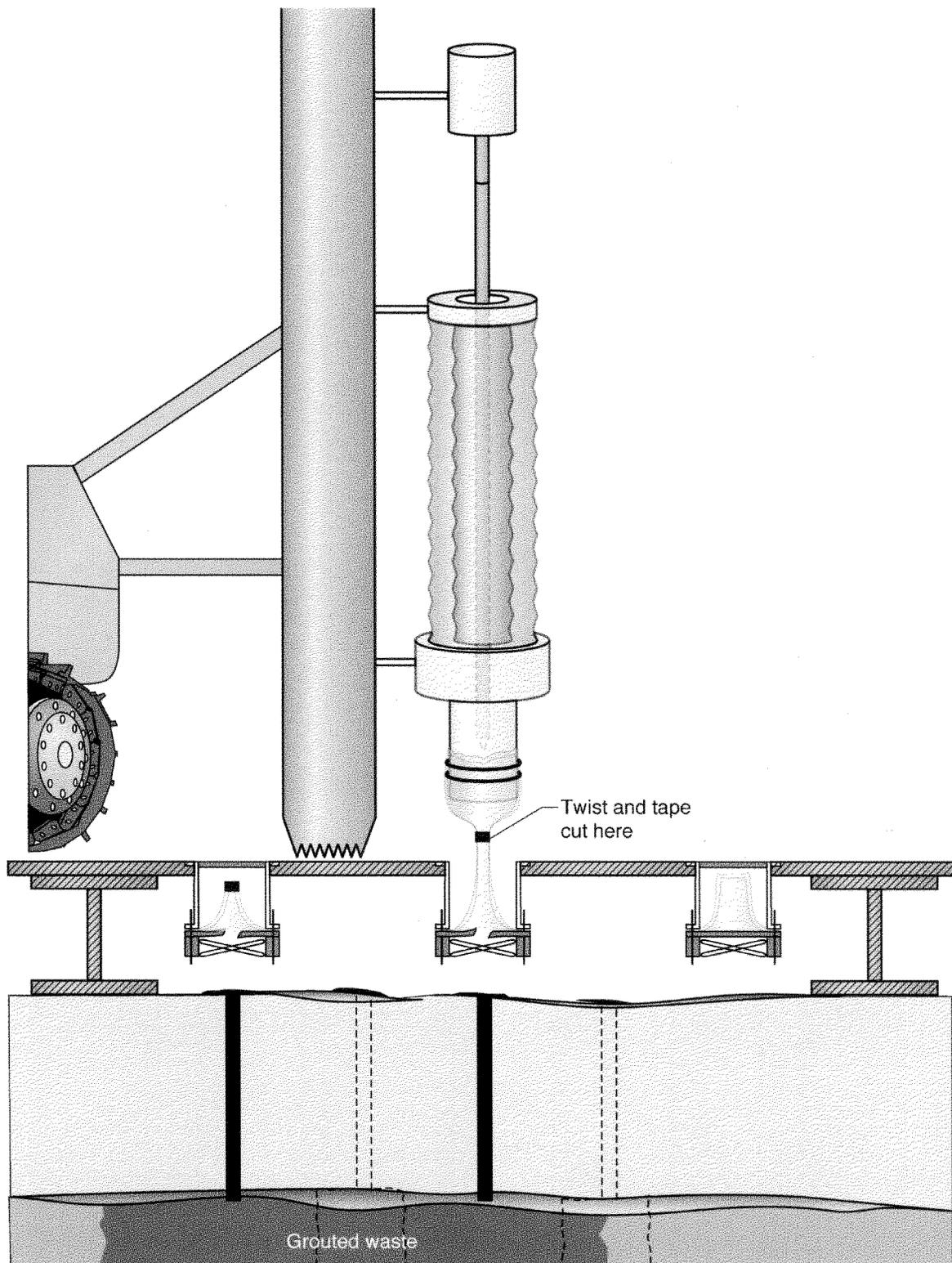


Figure 1. Thrust block design features.



02-GA50858-01

Figure 2. Grouting process with contamination control.



02-GA50858-02

Figure 3. Drill string withdrawn—preparing to move to a new hole.

drill stem into the waste. A typical set of parameters for grouting a variety of single-phase materials includes two revolutions of the drill stem per discrete step (a step is nominally 5 cm), with the step time usually between 2-6 seconds depending on grout returns. It was found that with a balance of these conditions along with specified grout physical characteristics 400 bar pressure created the best commingling of grout, soil, and waste and filling of voids within the waste.

Single-component materials that have been successfully grouted include simple Type-I Portland cement (Loomis and Thompson 1995; Loomis, Thompson, and Heiser 1995; Loomis, Zdinak, and Bishop 1997); Type-H Portland cement (Loomis, Zdinak, and Bishop 1997); TECT, a proprietary iron oxide cementitious grout from Carter Technologies of Houston, Texas (Loomis, Zdinak, and Bishop 1997); and Waxfix, a molten hydrocarbon product also from Carter Technologies (Loomis, Zdinak, and Bishop, 1997). Table 1 shows grout injection data compiled from various INEEL technical reports for these single-phase material studies.

Table 1. Single-component grout injection data.^a

Grout Type/Pit Type	# Holes	Total Injected Grout (L/gal)	Total Grout Returns (L/gal)	Injected Grout per 30 cm or 1 ft (L/gal)	Reference/Comments
Type-H cement/debris pit	27	11435/ 3,021	427/113	47/12.5	Loomis 1997—grout mixed 1:1 by volume = 18 sacks/m ³
TECT Carter Technologies/ debris pit	11	4417/ 1,167	189/50	66/17.6	Loomis 1997
Type-H cement/debris pit	19	5435/ 1,436	79/21	47/12.5	Loomis 1997—grout mixed 1:1 by mass (= 14 sacks/m ³)
Waxfix Carter Technologies/ debris pit	15	4644/ 1,227	1483/392	51/13.5	Loomis 1997—molten wax @ 60°C (140°F)
Type-I Portland/debris pit	36	18347/ 4,847	760/201	51/13.5	Loomis 1994—mixed 1:1 by mass; created a monolith for retrieval studies
Type-I Portland/debris pit	52	18347/ 4,847	435/115	39/10.3	Loomis 1995—grout mixed 1:1 by mass; created a wall barrier for retrieval studies
TECT HG Carter Technologies/ soil only	52	12472/ 3,295	2759/729	26/7.0	Loomis 1999—Acid Pit stabilization in soil only

a. Nominal injection pressure 400 bar. See first four references for other injection parameters such as drill rotation speed, withdrawal rate, step size, and time on a step.

In general, for pits containing debris, the average amount of injected grout per 30 cm (1 ft) that supported minimal grout returns while creating a solid overlapped series of columns was 42.4 L (11.2 gal). However, when injecting grout directly into soil, the amount of grout is reduced to nominally 2.2 L/cm (7 gal/ft), primarily due to fewer voids in the soil than in debris to absorb the injected grout. However, all of these waste materials were successfully grouted to form cohesive in situ monoliths.

The molten Waxfix material was easily grouted, although approximately 40% of what was injected came to the surface as grout returns. This mandates a required large plenum volume under the thrust block or some other berming technique to contain the returning material. While the cementitious pits would cure (hydrate) in the 24–36-hour timeframe, the pit injected with Waxfix took up to 1 week to cool to a solidified mass.

Once cured, the monoliths were both cored and destructively examined. In general, through examination of the cores and the excavated monolith, it was observed that the jet-grouting process created a monolith free of voids. It was also found that the cementitious grouts such as Portland cement and TECT could not saturate the tightly bound paper products but filled all interstitial voids in the waste containers, thus completely encapsulating these difficult-to-penetrate materials. The jet-grouting action created mixtures of grout and surrounding soil. These mixtures appear to be neat grout, grout intimately mixed with soil, and small inclusions of ungrouted soil.

For the pit grouted with the hydrocarbon Waxfix, all waste materials showed a complete penetration by the relatively low viscosity molten Waxfix—as if the grout had soaked into the material prior to curing (solidifying). For the Waxfix case study, soil inclusions commonly observed in the mixtures of soil and grout were completely soaked in the molten hydrocarbon-based grout in contrast to the inclusions found in the mixtures of soil and grout from the cementitious pits. In addition, waste material such as paper and wood likewise showed penetration by the Waxfix hydrocarbon. Even metallic objects showed a “coating” of hydrocarbon on outer surfaces. This is attributed to the relatively long time for the molten hydrocarbon pit to transfer heat to the surrounding soils. The permeation of the molten material into the waste material and soil continued long after a cementitious grout would cure.

For the INEEL soil conditions used in these studies, the general soil hydraulic conductivity is relatively low ($1e-5$ to $1e-6$ cm/s); therefore, most of the injected molten hydrocarbon remained in the pit and did not tend to migrate to the surrounding soils. In pits where the surrounding soils are more porous, molten material may tend to disperse to the surrounding soils, thus leaving voids in the soil/waste zone. Observations from the destructive examination of the debris pits filled with cementitious grouts indicated that they tended to be extremely difficult to remove. The best analogy is destroying a concrete building reinforced with rebar. The waste pit injected with molten hydrocarbon is an exception, in that the contents of the pit were removed with simple digging.

2.3 Grouting with Dual-Component Material

Three separate two-component materials were jet grouted in simulated waste pits with varying results (Loomis, Thompson, and Heiser 1995; Loomis, Zdinak, and Bishop 1997). The materials included (a) an acrylic polymer from Minnesota Mining and Manufacturing Company (3M) known as Hard 5750 and Soft 5751, (b) a DOE-developed natural analog grout with a natural analog of hematite, and (c) a Carter Technologies-supplied water-based two-part epoxy. Grouting data are shown in Table 2.

The 3M acrylic polymer was found to be fully field implementable; however, the hematite and epoxy products could not be jet grouted without further development. Grouting parameters for the 3M acrylic polymer are given in Table 2. The hard material (3M-5750) was developed to form a hard durable monolith suitable for in situ disposal. The soft material (3M-5751) was created to allow ease in the retrieval process, with the added benefit that contaminants would be agglomerated to a nonaerosolizable size, which would eliminate contaminant spread. Grouting was performed using the CASA GRANDE system as discussed above. However, a separate positive displacement pump was added for the second component; and the drill stem, nozzle, and swivel (coupling between the delivery hose and rotating drill stem) were modified.

Table 2. Two-component grout injection data.^a

Total Holes	Total Grout Returns (L/gal)	Total A Part (L/gal)	Total B Part (L/gal)
Hard Polymer Pit (3M 5750)			
18	476/126	2157/570	2187/578
Soft Polymer Pit (3M 5751)			
15	113/30	1934/511	1934/511
242l or 64 gal/hole of combined A and B Part each pit			
Grouting parameters: 3 cm/step; 3 s/step 2 revolutions/step for both pits (6 ft deep holes)			

a. Acrylic polymer from 3M hard (5750) and soft (5751).

The drill stem had a dual concentric annulus arrangement such that the two components were delivered into the waste through a dual concentric nozzle at the bottom of the drill stem.

Mixing of the two components occurred in the waste as the two streams of grout encountered the soil and waste.

The 3M polymers consisted of two co-monomers with select benzoyl peroxide and amine additives to start the polymerization process. When mixed with soil, the polymer formed a high molecular weight waste form that had excellent durability results. Laboratory testing on samples of polymer and soil (33% polymer and 67% soil by mass) included hydraulic conductivity measurements; resistance to immersion in water, trichloroethylene (TCE), and alkali; and resistance to wet-dry cycling. The laboratory hydraulic conductivity of the soil/polymer mixture was 2.8×10^{-12} cm/s. This is several orders of magnitude lower than the hydraulic conductivity of concretes. Ninety-day immersion testing and the wet-dry cycling testing indicated negligible change in compressive strength. Following grouting and curing, the pit was cored and then excavated. Cores showed that the polymer had indeed cured, suggesting that the process sufficiently mixed the two components downhole. The cores also exhibited little void space, indicating good waste penetration.

When the pit was excavated, the resultant monolith was freestanding. In fact, it could be moved as a complete unit (approximately $1.8 \times 1.8 \times 1.8$ m [$6 \times 6 \times 6$ ft]). Examination of the debris within the monolith showed similar results to those found in the pit injected with Waxfix in that there was considerable soaking of paper, cloth, etc., with the fluid prior to the polymerization process. One drawback to the process is that polymerization is exothermic. Temperatures approaching 140°C were encountered with visible smoke emanating from the grout holes. In addition, although not hazardous, the acrylic polymers emit an obnoxious odor. Mitigation of both the high exotherm and the obnoxious odor would require reformulation of the 3M product.

Grouting of the INEEL (hematite) and the Carter Technologies epoxy grout as formulated were not field deployable. For the hematite (a two-part mixture of simple slaked lime slurry and an aqueous solution of ferrous sulfate fertilizer), an attempt was made to inject the mixture into a simulated pit. The fact that the INEEL (hematite) material was not field deployable was most unfortunate in that geological media near the INEEL's Radioactive Waste Management Complex, it was noted that iron oxide-rich deposits tended to be stable in nature and not prone to the natural aging process; therefore, by injecting a slurry that cured to a hard form in the interstitial voids within the waste should promote the natural making of hematite out of the soil/waste matrix in geological times. Unfortunately, the slaked lime slurry caused filter caking, which is where particulate in the lime phase tended to separate out in the process of

delivery from the high-pressure pump to the drill stem nozzle. This led to system plugging at points where the slurry was at low velocity. Field attempts to alter the viscosity of the lime slurry by adding water failed to eliminate the filter caking, and additional jet grouting using this material was abandoned. The iron sulfate slurry that is the second component of the INEEL (hematite) grout, however, was found to be jet groutable. As a minimum, to make the hematite grout material jet groutable, a new formulation for the lime slurry would be required. Another possible solution is to reformulate the mixture and inject it as a single-phase mixture with a retarded cure.

For the Carter Technologies epoxy, there were two components—an A part and a B part. The B part was simply too viscous to be pumpable, and the entire load was abandoned. It should be noted that in the laboratory this epoxy mixture created excellent monoliths. The lesson learned from this unsuccessful experience is that strict quality control of the various parts of the material must be maintained when converting from laboratory formulations to thousands of liters of material. However, it is possible through more rigorous quality control that the A and B parts could both be jet groutable, since the A part was shown to be pumpable in the CASA GRANDE class system. The epoxy had the desirable property when mixed with soil that there was not an excessive exotherm nor was there an obnoxious odor.

2.4 Grouting as a Pretreatment Prior to Retrieval

Grouting followed by retrieval was performed using three different grouting materials including Type-I Portland cement, acrylic polymer, and the Waxfix product discussed previously.

2.4.1 Retrieval of a Monolith Grouted with Portland Cement

The original jet-grouting operations to form monoliths in simulated buried waste were performed as a pretreatment to the retrieval of buried transuranic waste [Loomis and Thompson, 1995]. It was thought that by grouting the waste, the fine soil particles would be agglomerated and the ultrafine plutonium particulate would be bound in larger pieces of debris and not easily aerosolized during removal operations. If bound sufficiently, it was speculated that retrieval operations could be easily serviced by manned entry into retrieval arenas using bubble suits. Studies involving retrieval with common mining techniques [Thompson et al., 1993] such as misting with water and surfactants on the dig face showed that, at best, during digging and dumping operations contamination control only achieved a 70% reduction in dust spread (this assumes the plutonium and dust move together, which has been suggested [Loomis et al., 1994]).

It was desired to achieve 90% or better reduction in dust spread to allow manned entry during retrieval operations to perform routine maintenance on remote retrieval equipment. The first effort involved creating a monolith in a full-scale $3 \times 3 \times 3$ -m ($10 \times 10 \times 10$ -ft) pit filled with typical 208-L (55-gal) drums and $1.2 \times 1.2 \times 2.4$ -m ($4 \times 4 \times 8$ -ft) boxes containing simulated waste such as cloth, paper, metal, sludge, concrete, and asphalt. In each container was dysprosium oxide tracer as a stand-in for plutonium. The simulated waste was randomly dumped into the pit and backfilled with soil in a manner similar to the actual burial practices in past INEEL disposal operations.

Type-I Portland cement (mixed 1:1 by mass) was injected into the pit on a 0.6 m (2-ft) triangular pitch matrix. Once a hole was grouted, 5-cm (2-in.) diameter thin-walled metal tubes were inserted into each of the just-grouted holes. These tubes were access holes for application of an expandable grout to help break up the monolith and generally facilitate retrieval. Once cured (in approximately 2 weeks), the expandable demolition grout (BRISTAR) was inserted. However, very little demolition of the monolith occurred. It was determined that the BRISTAR material only correctly operates in a fairly narrow temperature band. Due to the heat of hydration of the monolith when curing, temperatures as high as 60°C (140°F) were measured. In the 2 weeks of curing, the bottom contact temperature of each of the 5-cm (2-in.) tubes in the monolith was measured daily; and after 2 weeks, the temperatures equilibrated at about

21°C (70°F). From these data, it was assumed that the entire monolith was at this temperature. This assumption proved false, which led to an improper application of the expandable grout. When applying the BRISTAR, the bottom contact temperature of the holes was used; and even though the holes showed a relatively even temperature, it was not indicative of the temperature throughout the monolith. To correctly apply the BRISTAR would require waiting until internal temperatures in the monolith equilibrated (perhaps months). Use of a more extensive temperature measuring system would have allowed a correct application of the BRISTAR and most likely expansion and cracking of the monolith would have occurred.

Approximately 200 g of dysprosium oxide tracer material simulating plutonium was placed in each container. The spread of this tracer material was assessed for the grouting and retrieval phases of the innovative grout/retrieval operation. No tracer spread was measured in high-volume air samplers above background for the entire grouting operation. Once the pit was cured and the attempt was made to apply the BRISTAR, the pit was excavated with a standard backhoe using a thumb-lifting attachment.

Retrieving the monolith was extremely difficult and involved dropping the backhoe bucket onto the monolith. The resulting monolith resembled a reinforced concrete building demolition project. Especially difficult were the regions of the grouted 1.2 × 1.2 × 2.4-m (4 × 4 × 8-ft) boxed waste material. An evaluation of filters in air samplers situated around the dig face showed that during the retrieval process as much as a 90% reduction in dust spread over a base case of simply digging in surrounding soils was achieved as long as the clean overburden was removed first. If the overburden was not removed first, the top relatively dry material sloughed off into the pit and caused aerosolization of the soil, which was picked up on the high-volume samplers. The tracer material (dysprosium oxide powder) was measured on the high-volume air sampler filters at 1.35 times background for the retrieval activity.

2.4.2 Retrieval of a Monolith Grouted with Acrylic Polymer

Grouting of a two-part polymer was previously discussed. Two versions of this acrylic polymer were grouted, including a “soft-retrievable” version and a “hard-durable” version for disposal. The soft version of acrylic polymer was jet grouted into a simulated pit, allowed to cure, and then retrieved while taking air samples. For the pit grouted with the acrylic polymer, the simulated waste material had as a tracer dysprosium oxide powder at 200 g per container to act as a stand-in for plutonium in an actual transuranic pit or trench. The use of lanthanide oxides as valid stand-ins for transuranic materials has been discussed (Loomis et al., 1994).

During retrieval, evaluation of the air samplers showed a 91% reduction in dust spread; however, the tracer measurement on the air filters showed a two-order-of-magnitude increase over background levels. This was attributed to the fact that an ungrouted portion of the pit was inadvertently retrieved along with the grouted region, thus invalidating the data. The grouted portion of the pit was very easy to retrieve, and no voids were present in the monolith. The acrylic polymer permeated items such as cloth, wood, and paper prior to curing, such that it would be difficult for contaminants to become aerosolized during retrieval operations.

2.4.3 Retrieval of a Monolith Grouted with Waxfix

The monolith created by grouting with Waxfix showed very desirable properties for retrieval of buried waste. The molten material greatly penetrated all positions in the waste pit and agglomerated all fines into essentially nonaerosolizable particles. The retrieval was easily performed with a standard backhoe, and no visible dust was observed. No tracer material was used in the simulated waste containers, nor were dust data taken; however, on a qualitative basis, this material has the potential to greatly reduce dust spread—perhaps as much as 98%.

2.5 Stabilization of the Acid Pit Using Jet Grouting

Following development in cold test sites, the technology was applied to a mixed waste contaminated soil site called the Acid Pit located at the RWMC SDA. This pit contained both mercury at a maximum concentration of 5,200 ppm and minor amounts of fission products and pCi/g quantities of transuranics. Grouting this soil pit was extremely difficult to accomplish without excessive grout returns. While the debris pits could accommodate up to 2.2 L/cm (17.6 gal/ft) without excessive returns, the Acid Pit grouting averaged 0.86 L/cm (7 gal/ft). The operation was successfully completed in that the process was accomplished inside a radiation-controlled zone without the spread of either hazardous or radioactive materials (Loomis et al. 1999). It was estimated that the grouting process filled voids with grout equal to about 25% of the volume of the pit, which is consistent with the void volume in the soil. Based on experience during grouting, it was recommended that, when grouting contaminated soil zones, more grout volume per foot be delivered and more grout collection space under the thrust block be allowed.

2.6 Contamination Control During Grouting

For most grouting demonstrations, contamination control was assessed by evaluation of smears and high-volume air sampling for tracer materials. In all cases, tracer materials were placed in each debris container and generally were the “flour” form of a lanthanide oxide (tracers used included oxides of dysprosium, praseodymium, and cerium). Smears were obtained on the top of the thrust block and on the drill stem, and grab samples were collected under the thrust block. For the smears obtained on the drill stem (under the shroud) and for the grab samples, tracer materials were found to be above background values; however, smears on the thrust block showed no spread of tracer. In previous studies (Loomis and Thompson 1995; Loomis, Thompson, and Heiser 1995; Loomis, Zdinak, and Bishop 1997; Loomis et al. 1999) encompassing grouting with and without use of the thrust block, the high-volume air samplers showed no tracer above background. This was attributed to the simple fact that any contaminant brought to the surface was locked up in a slurry of grout and soil or actually in neat grout returns, and this slurry eliminated airborne release of contaminants.

3. BENCH STUDIES

A complex series of laboratory tests were performed on six promising 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. Although the in situ grouting technology was developed for in situ remediation buried transuranic debris such as what is found in the INEEL SDA it has potential application for supporting in situ treatment of low-level buried waste, as well as retrieval of buried transuranic waste (confinement during retrieval). The ultimate goal of the bench studies is to down-select from the six possible grouts to three grouts to carry into the field during “implementability” testing discussed later.

Desirable properties of the grout for application in buried transuranic waste sites include:

- Durability
- Low hydraulic conductivity
- Low temperature of set
- Chemical buffering
- Physical stability to support a cap
- Administrative feasible (grout availability, nonhazardous components)
- Field implementability
- Grout/interference compatibility
- Volatile organic compounds (VOCs) micro and macro encapsulation

Properties associated with using in situ grouting for supporting retrieval of buried waste relate to dust control, combustibility during handling, and the evaluation of using boron based grout additives to prevent criticality reactions. Desirable properties for the paraffin-based grout include:

- Neutron absorber compatibility
- Low combustion hazard

3.1 Background

The six grouts chosen for the bench study were selected from the grout types used during previous in situ grouting investigations at the INEEL (Loomis 1996, Loomis 1999). In these past investigations, grouts that exhibited good implementability tended to have relatively low viscosities, and high specific gravity. Grouts that exhibited initial gel times less than 2-hours caused problems in pumping equipment. Other grouts exhibited particulate separation causing “filter caking” on small jet grouting nozzles. Using these lessons learned, there was an initial screening for the six grouts followed by extensive physical and chemical testing on both the neat grouts and grouts mixed with expected interference materials from the buried waste. Grouts were also selected for compatibility with:

- Conventional jet-grouting techniques
- Environment and geotechnical characteristics of the SDA soil
- Buried waste contaminants and chemistry
- Cost of base materials.

Grouts selected for bench-testing are listed below:

3.1.1 TECT HG

TECT HG is a pozzolanic cementitious grout with proprietary additives from Carter Technologies. TECT exhibited good performance in previous INEEL studies and the HG version of TECT was used for the INEEL SDA Acid Pit project (Loomis et al. 1999).

3.1.2 Saltstone

Saltstone was developed at the Savannah River Site to stabilize aqueous nitrate salt waste streams and associated radioactive contaminants. The grout was specifically designed to stabilize technetium and plutonium. Saltstone is composed of blast furnace slag, fly ash and minor amounts of Portland cement. The grout exhibits acid-base properties (pH) of approximately 9 after set and cure and creates a reducing environment in waste site groundwaters.

3.1.3 Tank Closure Grout

Tank Closure Grout (reformulated as GMENT-12 by Technology Visions) was originally developed at the Savannah River site to stabilize waste remnants in storage tanks. Tank Closure Grout was specifically developed to immobilize uranium, plutonium, and other actinides. The formulation of the grout mix with a specific make up of ASTM Type-V Portland cement, blast furnace slag, and silica fume. The grout exhibits a pH of approximately 9 following set and cure and creates a reducing environment in waste site ground waters. Tank Closure Grout was reformulated by the University of Akron to INEEL jet-grouting specifications to allow jet grouting. Subsequent to the extensive reformulation effort, the grout was renamed GMENT-12.

3.1.4 Waxfix

Waxfix is a proprietary paraffin-based grout tested at the INEEL (Loomis et al. 1996). Waxfix from Carter Technologies exhibited excellent field performance. The molten material penetrated even the smallest void volumes in the pit and provided very low hydraulic conductivity.

3.1.5 U.S. Grout (Ultrafine Grout)

U.S. Grout premium grade is a pozzolanic cement from Hess Products of Malad, Idaho, that exhibits physical properties (low viscosity and delayed set parameters) indicating ease of grouting. It is a mixture of Type-H cement and local Idaho pumice.

3.1.6 Enviro-Blend—American Minerals (Phosphate)

This is a phosphate grout under development by American Minerals, Inc. The presence of phosphate in grout has been shown to result in good chemical fixation properties.

3.2 Test Objectives

The 9 CERCLA criteria provided the bases for all test objectives. Objectives for the bench part of the treatability study were given in detail in the test plan (Grant et al. 2000); however, a major programmatic objective was to down-select from the six grouts to three grouts to carry into the implementability testing discussed in the next section. The CERCLA related objectives included examining the grouts for implementability, overall protection of human health and the environment; compliance with applicable or relevant and appropriate requirements; long-term effectiveness; and reduction of toxicity, mobility, and volume.

3.2.1 Bench Testing

The critical objectives for bench testing outlined in the test plan (Grant et al. 2000) associated with the bench testing include (listed with the same numbering system as shown in the test plan):

Test Objective 1—Estimate the Durability of the Grouted Waste Monoliths

SrCO₃ and/or KNO₃ were added to the grout material at 0.1 percent by weight prior to mixing. The cured grouts were subjected to American Nuclear Society (ANS) 16.1 leach testing and the leachates were analyzed for either strontium tracer alone or for strontium tracer and nitrate tracer as well as aluminum, calcium, and silicon depending upon the test phase. Performance lifetime for the test grout mixture(s) in the waste site will be calculated from the ANS 16.1 protocol. This information will provide an estimate of the long-term physical and chemical durability of the grout material and an estimate of the rate of diffusion of contaminant materials from the grout matrix. The release rate of calcium, aluminum, and silicon provides a measure of the dissolution rate of the grout matrix. This information may be used to estimate the time the grout will provide physical stability to the waste-site and will affect the chemical behavior of the waste-site ground water. The release rate of the strontium and nitrate tracer materials will provide an estimate of the release rate of contaminant materials. Because it is not feasible to test all contaminants of potential concern, literature values of most contaminants will be used by the ER risk model. The values for strontium and nitrate measured in this study will be compared to the accepted literature values to provide a standard of comparison for the data obtained in the test program. Dissolution rates will be used to predict the long-term chemical durability of the grout monoliths. The durability estimates will establish the ability of the grout monolith to resist chemical degradation, thus maintaining contaminant encapsulation and chemical buffering.

Test Objective 2—Evaluate the Hydraulic Properties of the Grouted Waste Monoliths

Hydraulic conductivity, tensile strength, set temperature and shrinkage tests were performed on grout samples. The hydraulic conductivity measurement (ASTM D 5084-90) was carried out using the flexible wall permeameter which measured water saturated porous material. Tensile strength was measured by ASTM C-496-96 to determine the splitting tensile strength of cylindrical concrete specimens. Set temperature was measured using a simple thermocouple and data logging system. In the case of cementitious grouts, the set temperature is the temperature maximum during the cement hydration (setting) process. Shrinkage measurements (ASTM-C-827-95) determine the change in height of cylindrical test specimens from the time of casting until the time of set. The measurements include shrinkage or expansion due to hydration, settlement, evaporation, and other effects.

Test Objective 3—Identify Grout Material to Support Monolith Application, Safety Related Objective

Several ratios of soil/waste to grout mixtures were used to determine the maximum matrix/interference-loading ratio. The physical and chemical properties and temperature of set was

determined for the grout–soil monoliths. The chemical and physical properties data will be used to evaluate the grout formulations, and to select an appropriate mixture for implementability- and field-phase testing. The temperature of set data determines if the grout/interference mixtures set at a temperature greater than or equal to 100°C. A temperature of set in excess of 100°C may represent a safety hazard due to possible steam generation and expulsion of soil, grout, and waste materials (Loomis 1995).

Test Objective 4—Evaluate the Chemical Buffering Properties of the Grouted Waste Form

The solubility of hazardous waste constituents is affected by the chemical environment. For example, the dissolution of metals is influenced by the pH and oxidation-reduction potential (eH) of the surrounding medium. The pH and eH of the grout formulations was measured in the leachate for the ANS 16.1 leaching testing for both neat grouts and grouts with interference materials. pH is measured using a glass electrode (ASTM D 1293-95) and eH is measured using an inert metal electrode (ASTM D 1498-93). The eH and pH is indicative of the buffered chemical environment produced by the grout monolith and solubility of the encapsulated waste constituents. The solubility of each of the contaminants of potential concern will be computed as a function of eH and pH in an aqueous solution similar to the ground water at the SDA, namely saturated with calcite and in equilibrium with CO₂ in the air. The contaminant solubility data will be used by the ER risk model, in conjunction with other data from the in situ grouting treatability study and chemical literature, to estimate the mobility and release of the waste constituents. The data will be used to evaluate the grout formulations, and to select an appropriate mixture for implementability- and field- phase testing.

Test Objective 5—Evaluate the Physical Stabilization of the Waste Site to Control Subsidence

Samples of neat grout mixed with interference materials are tested for unconfined compressive strength. The bench data will be compared to the interference test data from actual field samples taken from the monolith during the field testing. The monolith must provide a stable foundation for material placed upon it, including impermeable caps and cover material. Undesirable collapse and subsidence of soils into subsurface voids occurs at the SDA during wet conditions. Soil subsidence affects the hydraulic properties of the SDA by causing ponding of surface water and may lead to an increase in the development of permeable pathways to the waste.

Test Objective 6—Evaluate the Effects of Soil, Organic Sludge and Nitrate Salt on Grout Properties

The interference of soil, nitrate salt, and organic sludge on the concentrations may adversely affect grout performance. This assessment was performed on specially prepared grout samples mixed at various interference loading concentrations of the simulated materials. During field-testing, samples will be collected and evaluated for comparison to bench results using specific interference loadings. This comparison will give confidence in using bench-derived data to evaluate future grout types for application of in situ grouting to buried waste. Test results and observations will be used to determine the waste loading tolerance for the grout materials and the waste mix compatibility of the chosen grouts with contaminants expected in the wastes buried at the SDA.

Noncritical objectives listed in the Test Plan include:

Test Objective B—Evaluate Effectiveness of Grout Encapsulation in Retaining VOCs—Micro and Macro encapsulation tests

Grouts when mixed with interstitial soils have the potential to encapsulate and reduce the release of VOCs from buried waste. These quantitative micro and macro encapsulation tests will measure the amount of VOCs remaining in the grout-stabilized simulated organic sludge samples, at various stages of the curing process as well as after cure. Both microencapsulation and macroencapsulation tests using an actual combination of VOCs and mixtures of soil and grout will evaluate in a specially prepared chamber the transport of VOCs from the monolith.

3.2.2 Special Testing

Objectives relating to special testing of grouting material appropriate for supporting special problems of using the paraffin based grouts

The critical objectives for these studies include:

Test Objective 1—Evaluate the Effects and Implementability of the Boron Additive on the Properties of the Paraffin-Based Grout (Waxfix), Safety Related Objective

Bench-testing was performed to determine the type and amount of boron compound that can be mixed with the paraffin-based grout. Addition of paraffin to waste containing fissionable material may increase neutron moderation and the potential for criticality thereby creating a safety hazard. Boron is commonly used at nuclear facilities to prevent criticality due to its capacity to adsorb neutron. A sample of the paraffin-based grout was heated until liquefied followed by addition of a solution of boron/borate and glycerin. The blended solution was then allowed to cool to ambient temperature. The solidified paraffin-boron matrix was then examined to determine the effects of the boron/borate additive on the physical characteristics of the grout, the maximum achievable boron concentration, and the suspension and distribution of the boron within the paraffin matrix. The data will be used to determine if the paraffin grout-boron mix may be safely emplaced during field operations. The data will also be used to determine the implementability of the paraffin-based grout with boron additive. The data will indicate if the distribution of the boron within the paraffin matrix is sufficient to be effective as a neutron absorber. The test results will also be used to determine the maximum concentration of boron that may be successfully added to the paraffin grout. The data will be used to determine if the introduction of boron to the paraffin grout will allow for the safe emplacement of the paraffin grout mix.

Test Objective 3—Evaluate the Combustion Hazard of the Paraffin-Based Grout (Waxfix), Safety Related Objective

The Department of Transportation oxidizer test will be carried out on prepared samples of paraffin and nitrate salt mixtures to determine the combustion hazard of potential waste material mixtures. Samples have nitrate salt loadings of 12, 25, 50, and 75 wt%. Testing will be performed according to 49 CFR 173.127. The data will be used to evaluate the combustion hazard of paraffin-based grout and nitrate mixtures.

Noncritical objectives for confinement during retrieval for bench testing include:

Test Objective A—Evaluation of the British thermal unit (Btu) Content of the Retrieved Grout Waste Form

A paper study evaluating the Btu content is presented. The study will show the Btu content of the waste form due to addition of the paraffin-based grout. The increase in Btu content due to the addition of paraffin-based grout will be used to evaluate potential ex situ waste treatment options.

3.3 Bench Testing Protocol

Testing was performed at the University of Akron under the direction of Dr. Al Sehn and Dr. Chris Miller (Miller). The bench testing followed a complex protocol involving first screening tests on neat grouts and grouts with interferences followed by specific physical and chemical testing on both neat grouts and grouts mixed with interferences. Other testing included micro and macro encapsulation testing to evaluate the transport of VOCs either intimately combined with a mixtures of soil and grout matrix or macroencapsulated by the same matrix. Finally, special testing was performed to examine technical issues with using a wax-based grout to support either the in-situ disposal option or the retrieval option. Tables 3 and 4 give a summary of testing protocols:

Table 3. Summary of testing of cementitious grouts for bench grout studies.

SCREENING-NEAT GROUTS	Viscosity (Marsh funnel API RB13B-1); Initial gelation/Final gelation (Shear Vane 100Pa/1,000Pa respectively); Pressure Filtration (API RP-10B); Maximum set temp (in situ thermocouple); minimum free water (volume measurement)
SCREENING-GROUT/ INTERFERENCE MIXTURE Soil-0,12,25,50,75 wt% Organic-0,3,5,7,9,12,25,50,75 wt% Nitrate-0,12,25,50,75 wt%	Compressive Strength (ASTM-C-3996)-triplicate measurements for all grout/ interferences that remain cohesive; Temperature of set-taken for one interference concentration for each grout-interference combination; Qualitative Observations: Cracking and fracturing, set retardation, incomplete mixing, swelling and disintegration.
PHYSICAL AND CHEMICAL TESTING-NEAT GROUTS(a)	Viscosity (API-RP-13B-1) triplicate; Density (ASTM D 4380-84) triplicate; Time to set (Shear Vane 100Pa/1,000Pa) triplicate; maximum temperature of cure (In situ thermocouple-based on neat grout cured in an insulated bottle. There was an environment matching a reference temperature of curing of a 50 wt% mixture of soil and the grout being tested for all samples used in physical testing) triplicate; tensile strength (ASTM C 496-96) 5 measurements; compressive strength (ASTM C 39-96) 5 measurements; Hydraulic Conductivity (ASTM-5084-90) duplicate; shrinkage (measured settlement) triplicate; Pressure Filtration (API-RP-10B) triplicate; Leach (ANSI/ANS 16.1 for Calcium, Strontium, Aluminum, Silicon, Nitrate) triplicate with eH and pH measured for each leach.
PHYSICAL AND CHEMICAL TESTING-INTERFERENCE/ GROUT MIXTURE(b) Soil-@50 wt% Organic-@9 wt% Nitrates-@12 wt%	Hydraulic conductivity (ASTM-D-5084-90) duplicate; Density (volume and mass) triplicate; Tensile strength (ASTM-C-496-96) triplicate; Compressive strength (ASTM-C-39-96) triplicate; Leach test (ANSI/ANS 16.1 Strontium only) triplicate
MICRO/MACRO ENCAPSULATION TESTING FOR VOLATILE ORGANICS	Microencapsulation testing for U.S. Grout, GMENT-12, and TECT HG a neat grout mixture and Rocky Flats Plant organic sludge containing 9 wt% Volatile organics are intimately mixed and the samples placed in a specially sealed chamber and the offgas measured at various times over a 90-day period. Macroencapsulation testing for U.S. Grout, GMENT-12, and TECT HG:A special hollow cylinder is created out of a 25 wt% mixture of soil and grout and the hollow portion is filled with the pure Rocky Flats Plant organic sludge and sealed in place. The system is placed in the special sealed chamber and the offgas is measured with time over a 90-day period.
SPECIAL LITERATURE STUDY (Activated Carbon as a Grout Additive)	Determine the efficacy of using finely divided activated carbon powder as an admixture to the grouts to adsorb and hold volatile organics present in the buried waste.
a) 0.1 wt% strontium carbonate and 0.1 wt% potassium nitrate added to the neat grout as a tracer	
b) 0.1 wt% strontium carbonate added to the neat grout as a tracer	

Table 4. Summary of testing for Waxfix.

<p>PHYSICAL AND CHEMICAL TESTING-NEAT GROUTS(a)</p>	<p>Viscosity (API-RP-13B-1) triplicate; Density (ASTM D 4380-84) triplicate ;Time to set (Shear Vane 100Pa/1,000Pa) triplicate; maximum temperature of cure (In situ thermocouple in an insulated bottle.-cured in an environment matching a reference temperature of curing of a 50 wt% mixture of soil and TECT HG grout triplicate; tensile strength (ASTM C 496-96) 5 measurements; compressive strength (ASTM C 39-96) 5 measurements; Hydraulic Conductivity (ASTM-5084-90) duplicate; shrinkage (ASTM-C 827-97) triplicate; Pressure Filtration (API-RP-10B) triplicate; Leach (ANSI/ANS 16.1 for strontium, Nitrate) triplicate with eH and pH measured for each leach.</p>
<p>PHYSICAL AND CHEMICAL TESTING-INTERFERENCE/ GROUT MIXTURE(b) Soil-@50 wt% Organic-@9 wt% Nitrates-@12 wt%</p>	<p>Hydraulic conductivity (ASTM-D-5084-90) duplicate; Density (displaced volume and mass) triplicate; Tensile strength (ASTM-C-496-96) triplicate; Compressive strength (ASTM-C-39-96) triplicate; Leach test (ANSI/ANS 16.1 Strontium only) triplicate</p>
<p>SPECIAL TESTING FOR Waxfix(c) (Neutron Absorber Additives)</p>	<p>Six samples of a mixture of Waxfix and a mixture of sodium tetraborate and glycerin that gives 1 g/L of B-10 in the mixture will be made with three samples gradually cooled to room temperature and three gradually cooled to 5F. For each of the six samples, 5 samples at 5 different axial locations will be taken for Inductively Coupled Plasma-Mass Spectroscopy for boron (the presence of B-10 will be inferred from this value); Department of Transportation Oxidizer Test for samples containing 0,12,25,50,and 75 wt% potassium nitrate (following 49 CFR 173.127);literature review for the Btu content of Waxfix will also be performed and reported.</p>
<p>a) 0.1 wt% strontium carbonate and 0.1 wt% potassium nitrate added to neat grout as a tracer b) 0.1 wt% strontium carbonate added to neat grout c) Physical testing as well as Department of Transportation oxidizer test and Btu content testing deferred based on negative results of B-10 concentration testing.</p>	

3.4 Screening Test Results

An initial screening of both neat grout samples and neat grout samples with interferences was performed for the cementitious grouts. The Waxfix grout was not part of this screening process. The screening tests were designed to eliminate those grouts not meeting the minimum criteria from the extensive testing protocol. Data gathered during past in situ grouting operations conducted at the INEEL established that small amounts of certain interferences have severe and adverse effects on the physical and containment characteristics of the grout monolith (Loomis et al. 1996, 1998). The presence of interferences such as volatile organic chemicals, nitrate salts, and soils in the waste material may slow or sometimes stop grout setting and curing reactions. In addition, past experience has shown that some grouts, while promising in the laboratory, are not jet-groutable in the field. All these screening tests support critical test objectives 3, 5, and 6.

Samples of neat grout were mixed according to the mix formulas supplied by the vendor. Grout formulations that required modification to meet these stated implementability criteria included the Tank Closure grout (renamed GMENT-12) and the Saltstone grout. It should be recognized that these nonvendor grouts were not developed specifically for jet grouting operations, thus the required modifications. The changes to the Saltstone grout and the Tank Closure grout were changes in the formulation to provide improved jet grouting capability. Such changes resulted in a better score in the evaluation ranking. The changes mainly altered the set time, maximum temperature during curing, Marsh funnel time, filtration performance, and amount of settlement/bleed water. The objective was to alter these characteristics of the grouts while either maintaining or improving the strength, permeability, and leaching characteristics of the grouts.

Once mixed, samples of neat grout were poured into 3-in. diameter by 6-in. high plastic molds and allowed to cure for 14 days in a special curing environment. The neat grout was cured in a temperature controlled water bath. The water bath temperature was controlled by following the curing temperature of a reference mixture of 50 wt% soil and TECT HG grout. The grouts were evaluated for specific gravity, initial and final gel time, pressure filtration, maximum set temperature, and free water/shrinkage. Table 5 summarizes the data for this initial screening for the neat grouts with the minimum required criteria for each parameter.

Table 5. Screening test results and criteria.

Grout Property	Grout Product					Screening Criteria
	GMEN 12	Enviro- Blend	Salt Stone	TECT HG	U.S. Grout	
Specific Gravity	1.84	1.78	1.60	2.16	1.65	
Viscosity (Marsh Funnel Time) (sec.)	56	165	110	113	58	< 420
Initial Gelation Time (hours)	4.9	9.4	1.8	6.0	4.7	> 2
Final Gelation Time (hours)	10.7	27.5	8.3	17.9	7.6	> 2
Pressure Filtration Coefficient (min ^{-0.5})	0.072	0.077	0.023	0.008	0.033	0.1 to 0.6
Maximum Set Temperature (deg. C)	59	32	28	62	46	< 100
Settlement/Shrinkage (%)	1.82	3.16	0.25	0.44	0.84	minimized

Examination of Table 5 shows that for every screening performance criteria the grouts GMENT-12, Enviro-Blend, TECT HG, and U.S. Grout, passed. The only grout that did not meet the minimum requirements was the Saltstone grout in that the initial gel was below 2 hours. This property eliminates Saltstone from further consideration for the treatability study in that a too early set is incompatible with expensive pumping equipment which could “freeze” with early setting grout. With some further laboratory manipulations, it is thought that Saltstone grout could achieve an initial gel time above 2 hours by applying common set “retarders” such as lignosulfonates. However, as formulated it was removed from further consideration for the present application (it is noted here that certain long term testing was performed on Saltstone and is reported in this document; however, as formulated it cannot be considered for jet grouting applications).

3.5 Screening of Grout/Interference Mixtures

In general, the jet grouting process creates a solid monolith. However due to certain interferences there may be regions in the solid monolith that are pockets of mixed neat grout and loose buried waste material. Examples of loose material include interstitial soil, inorganic sludges (that for all practical purposes look like soil both physically and chemically), organic sludges, and nitrate salts. All of these loose materials or interferences can degrade the structural integrity locally within the monolith. As part of the Bench study then, mixtures of grout and interferences were created to further screen the grouts in that if a grout had virtually no tolerance for maintaining its integrity represented by compressive strength at any loading of interference, that grout could be eliminated from further consideration. What follows are experimental results of the effect on compressive strength for three common interferences. The results are tabularized in Table 6. Appendix B has the detailed data sets for the averages shown in Table 6. The data was taken as a set of five measurements for each interference wt%. Five data points provide a reasonable statistical average for compressive strength.

Table 6. Average compressive strength in psi for the interference tolerance testing specimen groups.

Interference Type	Interference Percentage	Grout Product				
		GMENT 12	Enviro-Blend	Salt Stone	TECT HG	U.S. Grout
None		7,639	150	1,306	6,320	2,582
INEEL Soil	12	5,884	62	1,259	4,150	3,896
INEEL Soil	25	6,048	26	910	3,654	3,098
INEEL Soil	50	2,529	43	1,318	1,924	1,278
INEEL Soil	75	NA	NA	403	NA	805
Nitrate Salts	12	3,171	39	700	3,239	4,801
Nitrate Salts	25	2,885	4	403	1,193	1,383
Nitrate Salts	50	3	NA	1	NA	1,813
Nitrate Salts	75	104	11	3	NA	869
Organic Sludge	3	7,349	133	1,275	4,296	3,276
Organic Sludge	5	6,100	132	1,075	3,706	2,878
Organic Sludge	7	6,215	102	985	2,820	2,644
Organic Sludge	9	6,083	105	1,021	2,618	3,136
Organic Sludge	12	NA	116	924	2,347	NA
Organic Sludge	25	NA	NA	507	204	NA
Organic Sludge	50	NA	52	NA	7	NA

NA – Generally could not form a “stand-alone” monolith.

3.5.1 Soil as an Interference

Mixtures of neat grout and INEEL soil (sieved to 50 Mesh) were mixed at 12, 25, 50, and 75 wt% soil and allowed to cure in 100% humidity environment. It was thought that in a “free standing” monolith in a field application this range of soil/grout mixtures would cover the expected range in the actual jet grouting of buried waste. Considerable tolerance to soil loading was observed; however, for the Enviro-Blend grout, the values all were below 100 psi. Examining Table 6, shows that the individual triplicate test results for neat grout and soil at 12,25,50 and 75 wt%, the GMENT-12 had the highest neat grout compressive strength values which generally continued when adding interferences. Even with 50 wt% soil loadings, the GMENT-12 had compressive strength for the triplicate measurement higher than 2,500 psi, which was higher than the neat Saltstone grout. There was an interesting aggregate effect for the GMNET-12 grout in that the average compressive strength for 50 wt% is higher than for 25 wt% much like adding aggregate to concrete in the building industry. Enviro-Blend had such low initial neat grout compressive strength that any addition of interferences degraded the grout to a condition of not being able to “stand alone.” Since soil is pervasive throughout a waste pit and further that during jet grouting one of the main binders for the monolith will be the resultant mixtures of soil and grout, the Enviro-Blend grout as formulated does not pass the screen for tolerance testing. However, GMENT-12, Saltstone (note: Saltstone was eliminated during the neat grout screening in section 2.3 for short set time, however, considerable simultaneous data was obtained and thus will be reported herein), TECT HG and U.S. Grout all met competency soil requirements for 50 wt% tolerance testing. From these data it was recommended that 50 wt% soil be used during the physical and chemical testing for grouts with interferences described in a following section. In addition, 50 wt% soil represents a typical condition found throughout a monolith created by jet grouting a buried waste site.

3.5.2 Organic Sludge as an Interference

Organic sludge when mixed with neat grout during the jet grouting process has the potential to produce zones of considerably degraded grout (higher hydraulic conductivity, loss of compressive strength). On an average in the INEEL SDA transuranic pits and trenches organic sludge makes up about 5vol% of the waste pit volume; however, zones of almost total organic sludge drums are possible. Past studies (Loomis et. al. 1996) have shown that jet grouting grease-like materials can degrade grout curing and monolith stability; however, with certain grouts, when isolated drums of organic material are jet grouted cohesive monoliths can be formed. Grout was mixed with an organic sludge formulation based on Rocky Flats waste (see Table 7) using trichloroethylene, tetrachloroethylene (PCE), carbon tetrachloride (CCl₄), and trichloroethane (TCA) as volatile organics mixed with absorbers and TEXACO REGAL MOTOR OIL. The resultant mixture of volatile organics, oil, and absorbers exhibit a grease like consistency. Once mixed with neat grout and allowed to cure in a 100% relative humidity curing room, the resultant monolith was tested for compressive strength in triplicate at 0, 3, 5, 7, 9, 12, 25, 50, 75 wt% sludge.

Table.7. Material proportions for the organic sludge interference mixture.

Ingredient	Quantity
Calcium Silicate	4120 grams
Oil Dri	620 grams
Carbon Tetrachloride (CCl ₄)	2680 milliliters
Tetrachloroethylene (PCE)	740 milliliters
Trichloroethylene (TCE)	740 milliliters
Trichloroethane (TCA)	1030 milliliters
Texaco Regal Oil, R&O 68	5130 milliliters

For GMENT-12, Saltstone, TECT HG, and U.S. Grout, there was good tolerance to organic interferences for lower wt% of the organic sludge (up to 9-12 wt%) as shown in Table 6. However, for higher than 9-12 wt% organic loading, the resultant monolith exhibited low compressive strength. As with the soil interference, the Enviro-Blend grout showed low tolerance for organic sludge at all sludge loadings. Table 6 summarizes the individual test results showing that GMENT-12 had very little degradation and in fact maintained a relatively high compressive strength (nominally 6,000 psi) for all triplicate samples through 9 wt% organic sludge. The TECT HG grout also had reasonably high compressive strength (3,000-4,000 psi) for up to 12 wt% and even tolerated 25 wt% sludge at an average of 2,347 psi, which is consistent with samples obtained during past in situ grouting experiments (Loomis 1996). Saltstone showed an average compressive strength of over 500 psi at 50 wt% sludge. Based on the results shown in Table 6, it was concluded that physical and chemical testing for grouted organic interferences (discussed in a following section) should be performed at 9 wt%.

3.5.3 Nitrate Salt as an Interference

Neat grouts were mixed with granular nitrate salts (roughly 33% potassium nitrate and 67% sodium nitrate representing Rocky Flats evaporation pond salts found in the transuranic pits and trenches at the INEEL SDA.) at various nitrate loadings (12, 25, 50 and 75 wt%). Salts in general have been shown to cause degradation of concretes and knowing the tolerance to these nitrate salts is important for determining localized long term monolith integrity. Within local regions around a nitrate drum in a grouted solid monolith, there may be some local degradation due to the presence of nitrates.

Following curing, the compressive strength was performed on the monoliths in triplicate and the average results are presented in Table 6. U.S. Grout showed the best tolerance to the nitrate salts loadings with compressive strength in excess of 800 psi even at 50 wt% loading. Of the grouts that formed cohesive monoliths, the Saltstone grout showed the poorest tolerance to the nitrate salts with virtually no tolerance after 25 wt% loading. Again, as with the other tolerance testing, the Enviro-Blend grout showed virtually no tolerance to interference loadings. Based on the results shown in Table 6, a nitrate loading of 12 wt% were selected to perform physical and chemical testing on the nitrate interference testing. 12 wt% was chosen because it represents the highest nitrate loading that still has structural integrity such as might be found in a monolith near a grouted drum.

3.6 Testing of Neat Grouts

3.6.1 Physical and Chemical Testing of Neat Grouts

Physical testing performed on cured neat grout samples include determining the grout density, viscosity, splitting tensile strength, compressive strength, and hydraulic conductivity as well as the leaching characteristics in water. Chemical testing includes determining the buffering qualities of the grout by measuring pH and eH of leach waters from leaching procedures. The neat grout samples used for physical and chemical testing were cured in a unique temperature controlled bath of fluid rather than exposing the curing samples to supply constant air temperature. This was done to simulate neat grouts curing in an actual buried waste pit in which much of the pit is a mixture of soil and grout. The bath temperature was controlled by using a feedback system in which heat was added to the bath as the reference mixtures temperature of soil and grout increased during hydration or curing. The reference material in this case was 50 wt% soil and 50 wt% grout which is typical of mixtures of soil and grout. The thermocouple in the reference mixtures of soil and grout showed an increase during curing; however, the bath temperature was kept 1–2°F cooler than the curing mixtures of soil and grout. Within this bath, the various neat grout samples of physical cured chemical testing were allowed to hydrate or cure as their nature allowed. This action prevented unwanted physical cracking due to differential heat stresses during the curing process associated with curing in open air.

Physical Testing of Neat Grouts

Table 8 summarizes neat grout properties including specific gravity, viscosity (as measured in a Marsh Funnel), pressure filtration and hydraulic conductivity. There was a considerable range for specific gravity of the various grouts (range 2.16 for TECT HG and 1.60 for Saltstone). Past grouting studies have indicated a tendency for larger column formations for the denser grouts such as the TECT HG. GMENT-12 at 1.85 specific gravity is an intermediate density grout. The Marsh-Funnel test for viscosity showed an average range of 61s for GMENT-12 to 165s for Enviro-Blend. Basically, all of the grouts tested with low enough viscosity to be considered jet groutable.

In past studies, it was found that grouts with as high as 7min in the Marsh Funnel test could be jet grouted; therefore, all of the grouts are acceptable on the viscosity test. The pressure filtration test suggest that all of the grouts are to be considered stable for jet grouting applications in that the grout does not exhibit a tendency to lose water under pressure when pressed through a filter material. Basically, this means that pressure filtration numbers above 0.4 min (-1/2) are considered unstable mixtures and numbers in the range of .008 to .08 min (-1/2) (which is the range of those tested in this study) are stable and do not bleed excess water under pressure. The hydraulic conductivity values shown in Table 8 are excellent for all 5 grouts tested. GMENT-12 and TECT HG had hydraulic conductivities on the order of 10^{-9} cm/s, which is nearing measurement limitations for the time allowed to perform these studies.

The porosity of the GMENT-12 cured neat grout is estimated by Dr. Al Sehn of the University of Akron at 25%. The porosity of other grouts considered in this study were not measured, in that the technique involves baking the sample thus, introducing cracks in the system (ASTM C 642-97 was called for in the test plan [Grant et al. 2000]).

Table 8. Specific gravity values, Marsh funnel times, filtration test results, and hydraulic conductivity values for the neat grouts.

Test	Grout Product				
	GMEN-12	Enviro-Blend	Salt Stone	TECT HG	U.S. Grout
Specific Gravity, Test 1	1.85	1.77	1.60	2.16	1.65
Specific Gravity, Test 2	1.85	1.78	1.60	2.16	1.65
Specific Gravity, Test 3	1.84	1.78	1.60	2.16	1.65
Average Specific Gravity	1.85	1.78	1.60	2.16	1.65
Marsh Funnel, Test 1 (sec)	62	164	87	129	49
Marsh Funnel, Test 2 (sec)	63	165	97	141	50
Marsh Funnel, Test 3 (sec)	57	166	103	148	53
Average Marsh Funnel (sec)	61	165	96	139	51
Filtration Test, Test 1 (min ^{-0.5})	0.087	0.084	0.024	0.008	0.026
Filtration Test, Test 2 (min ^{-0.5})	0.080	0.082	0.023	0.008	0.026
Filtration Test, Test 3 (min ^{-0.5})	0.084	0.082	0.024	0.008	0.024
Average Filtration Test (min ^{-0.5})	0.083	0.083	0.024	0.008	0.025
Hydraulic Conductivity, Test 1 (cm/s)	8.5E-09	1.6E-07	1.2E-08	9.8E-09	1.7E-08
Hydraulic Conductivity, Test 2 (cm/s)	6.1E-09	1.3E-07		1.7E-09	1.9E-08
Average Hydraulic Conductivity (cm/s)	7.3E-09	1.5E-07	1.2E-08	5.8E-09	1.8E-08

Table 9 presents the compressive and splitting tensile strength values for the neat grouts. For GMENT-12, TECT HG, and U.S. Grout both compressive and splitting tensile strength were relatively high with a maximum compressive strength for U.S. Grout as high as 9,000 psi and for all grouts the splitting tensile strength was in the range of 500 to 700 psi. In sharp contrast, Enviro-Blend had low compressive strength and splitting tensile strength and Saltstone had relatively low splitting tensile strength.

In summary, from a physical testing standpoint, many of the grouts showed excellent properties for application in buried waste. GMENT-12, TECT HG, and U.S. Grout showed good jet grouting properties while exhibiting excellent strength of grout and low hydraulic conductivities.

Table 9. Compressive strength and splitting tensile strength values for the neat grouts.

Test	Grout Product				
	GMEN-12	Enviro-Blend	Salt Stone	TECT HG	U.S. Grout
Compressive Strength, Specimen A	3040	103	1407	7443	8230
Compressive Strength, Specimen B	2213	85	1457	6566	8442
Compressive Strength, Specimen C	3154	104	1230	7815	9431
Compressive Strength, Specimen D	6463	100	1421	7947	9432
Compressive Strength, Specimen E	7106	104	1400	6922	8564
Average Compressive Strength (psi)	4395	99	1383	7339	8820
Tensile Strength, Specimen A	668	13	126	757	332
Tensile Strength, Specimen B	836	11	156	758	453
Tensile Strength, Specimen C	781	13	86	780	613
Tensile Strength, Specimen D	643	14	166	692	661
Tensile Strength, Specimen E	605	14	138		481
Average Tensile Strength (psi)	707	13	134	747	508

Leaching Data for Neat Grouts

To determine leaching characteristics, the testing protocol suggested in “Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure, American National Standard ANSI/ANS-16.1 –1986” was followed. This procedure involves immersing the solid grout samples in a series of baths of demineralized water for various specified times over an interval of 90 days. For each of these baths, the leachate waters were tested for specific leached elements, specifically in this case, aluminum, calcium, silicon, and strontium tracer. If there are materials of interest on the surface, they are theoretically washed off in the early baths such that in an evaluation of later baths for the materials of interest, any that show up in the leachate water are there from deterioration or diffusion within the solid samples. For instance if the Diffusion coefficient changes to higher numbers in the later baths, this is an indication of relatively rapid break-up within the water immersion. If the numbers remain relatively constant or only change slightly, this is suggesting a diffusion controlled release of material and the sample is fairly stable. The volume of leachant employed was 2,200 mL, as specified by the ratio of 10 ± 0.2 of leachant volume to external geometric surface area of the specimen. After rinsing the specimens for an initial period of 30 seconds, the leachant was replenished at specified time intervals: for a total of 10 leachate samples. Aliquots of the leachates were analyzed for Sr, Al, Si, Ca, and NO_3^{-2} using inductively coupled plasma (ICP). Recall that the concept is to measure the dissolution of the building block of the grout (Ca, Si, Al) and mobile contaminants represented by Sr and nitrates. Comparison of the rates of dissolution can be used to support modelings of long term durability of a monolith and the rates of contaminant release. The leaching data are presented in terms of diffusivity coefficient and leachability index. Average leachability indices and diffusivity coefficients were

calculated for each of the replicate sets. In rough terms, the negative exponent of the diffusivity coefficient is the same as the leachability index. The detailed data are included in Appendix C.

The results of the leaching test were fitted to a semi-empirical mathematical model based on simple leaching rate mechanisms, which permitted the evaluation of an apparent diffusion coefficient and a leachability index, thus providing a measure of the contaminants' mobility in the solidified waste. In the case of Sr, Al, Ca and Si, the rate of leaching was controlled by an initial wash off, followed by diffusion.

The leach test is a semi-dynamic test; that is, the leachant is sampled and replaced periodically. The test method is applicable to any material that does not degrade, deform, or change its leaching mechanism at the temperatures used in the test. In Appendix C of this report, detailed results of the calculations are presented in several ways. The most basic value determined from a leach test is the incremental fraction leached, from which the cumulative fraction leached is calculated. If less than 20% of a leachable species is leached from a uniform, regularly shaped solid, its leaching behavior (if diffusion controlled) approximates that of a semi-infinite medium. Under these conditions the mass-transport equations permit the calculation of an "effective diffusion coefficient" by the expression:

$$De = \pi \left[\frac{an / A_0}{(\Delta t)_n} \right]^2 \left[\frac{V}{S} \right]^2 T \quad (1)$$

Where

De = effective diffusivity, cm²/s,

V = volume of specimen, cm³,

S = geometric surface area of the specimen as calculated from measured dimensions, cm², and

$$T = \left[\frac{1}{2} (t_n^{1/2} + t_{n-1}^{1/2}) \right]^2 \quad (2)$$

Leaching time represents the "mean time" of the leaching interval.

To measure the base amount of Al, Si, Ca, Sn, and nitrates in the solid grout samples, the following analytical technique was followed:

5 mL (12 M) hydrochloric acid was added to the 1 g solid sample in fluon crucibles and mixed thoroughly. A sequential heating process was then carried out for 2 hours at 150°C. They were removed from the heat, when the solution in the crucible was evaporated. After a cooling period, concentrated nitric acid (2.5 mL) was added and the crucibles were then heated at 150°C for another 3 hours. Once removed from heat, 6-7 mL hydrofluoric acid and 0.25 mL HClO₄ were added to each crucible and heated for 5 hours until the solution evaporated to near dryness. 2 mL hydrochloric acid were added to each crucible and then leached for 1 hour. The residues were finally dissolved in 0.2 M HCl. The resultant solutions were subsequently used for analysis by ICP and are represented in Table 10 as mg/g of material for the leachate materials of interest, i.e., calcium, silicon, aluminum, and the tracer material strontium, which was added to a concentration of 0.593 mg/g.

Table 10. Element concentration determination for each grout.

Grout	Element (mg/g)		
	Al	Si	Ca
U.S. Grout	7.79	10.69	37.01
TECT HG	7.28	14.87	107.56
Enviro-Blend	4.88	19.08	4.31
GMENT-12	6.91	8.04	91.64
Saltstone	16.48	5.25	46.48

Note: Spiked with Strontium (Sr = 0.593 mg/g) and nitrate (NO_3^{-2} = 0.614 mg/g).

The individual leaching data for each grout are shown in total in Appendix C and Table 11 summarizes the average leach index for the various grouts (note, the Leach Index is approximately the absolute value of the negative exponent of the diffusivity coefficient, therefore, the higher the leach index, the more resistive a material is to leaching).

Table 11 shows the evaluation of leach index for grout specific elements (aluminum, silica, calcium) as well as for a nonradioactive tracers strontium and nitrate salt placed in the grout as a 0.1 wt% of grout mixture strontium carbonate and sodium nitrate. A higher leach index (or smaller diffusion coefficient, which is basically the negative exponent of the leach index-see the Appendix C for a complete listing of diffusion coefficients as well as other data) is an indicator of durability. As shown in Table 11, all grouts exhibited relatively high leach indexes (10-14.5) for all constituents in the grout (aluminum, strontium, calcium, and silicon) with the phosphate containing American Minerals, Inc.'s Enviro-Blend having the highest leach index.

Table 11. Neat grout average leach index (n = 3) results for Sr, Al, Ca, Si, and NO_3^- .

Grout	Sr	Al	Ca	Si	NO_3^-
U.S. Grout	10.6 ± 0.9	11.1 ± 0.4	9.8 ± 0.9	10.2 ± 0.7	9.2 ± 0.3
TECT HG	10.1 ± 0.3	12.3 ± 0.6	10.1 ± 0.5	11.1 ± 0.5	11.0 ± 0.7
Enviro-Blend	12.8 ± 1.2	14.5 ± 1.6	9.8 ± 0.3	14.2 ± 1.5	8.8 ± 0.2
GMENT-12	10.0 ± 0.5	12.2 ± 0.8	10.5 ± 0.5	10.7 ± 1.1	10.4 ± 0.6
Saltstone	10.2 ± 0.6	12.6 ± 0.9	10.5 ± 1.0	10.2 ± 0.9	10.8 ± 0.8

Results reported ± one standard deviation.

As expected, the nitrate material showed lower leach indexes with a range of 8.8 to 11.0 which are impressive considering the solubility of nitrate materials. The Enviro-Blend grout had higher leach indexes than the other cementitious grouts because of the presence of phosphates which form insoluble compounds with leachable material. As an example of a complete data set (the leaching was performed in triplicate for each grout), Table 12 shows the complete data for the TECT HG grout for one replicate sample for the entire 90-day testing (using diffusion coefficient rather than Leach index). Notice in Table 12 that the diffusion coefficient is relatively stable in that there is not a tendency to decrease with further immersion in the leachate with time for all elements except for the nitrate salt as expected. As a further example, during the time period between 47 days and 90 days (a total of 43 days leaching), there was only .664 mg/L of Sr leached (average .015 mg/L per day) compared to the surface wash-off seen in the first few days which is on the order of 0.2 mg/L leached. This suggests that following the surface wash-off effects, the process of elements entering the leachate water is diffusion controlled.

Table 12. TECT HG grout replicate neat grout ANS 16.1 data.

Time (d)	Sr (mg/L)	Al (mg/L)	Ca (mg/L)	Si (mg/L)	NO ₃ ⁻ (mg/L)
0.083	0.073	0.028	14.097	0.625	0.019
0.292	0.086	0.051	15.697	0.489	0.019
1.000	0.182	0.174	36.784	1.421	0.028
2.000	0.187	0.220	47.850	1.782	0.038
3.000	0.147	0.186	40.863	1.722	0.038
4.000	0.100	0.184	16.329	1.676	0.029
5.000	0.118	0.209	30.554	1.925	0.019
19.000	0.975	0.611	208.154	3.825	1.010
47.000	0.564	0.639	95.820	4.554	1.010
90.000	0.664	0.757	82.308	4.463	0.820

Time (d)	De (cm ² /s)				
	Sr	Al	Ca	Si	NO ₃ ⁻
0.083	4.33E-11	4.22E-14	4.89E-11	5.04E-12	2.75E-12
0.292	7.82E-11	1.83E-13	7.93E-11	4.01E-12	3.58E-12
1.000	1.04E-10	6.29E-13	1.30E-10	1.01E-11	2.31E-12
2.000	1.35E-10	1.25E-12	2.69E-10	1.96E-11	5.22E-12
3.000	1.43E-10	1.52E-12	3.35E-10	3.10E-11	8.87E-12
4.000	9.26E-11	2.07E-12	7.51E-11	4.14E-11	7.28E-12
5.000	1.66E-10	3.44E-12	3.39E-10	7.02E-11	4.04E-12
19.000	1.40E-10	3.65E-13	1.94E-10	3.41E-12	1.40E-10
47.000	3.40E-11	2.89E-13	2.98E-11	3.52E-12	1.01E-10
90.000	4.23E-11	3.65E-13	1.98E-11	3.05E-12	6.03E-11

Chemical Testing of the Neat Grouts

The in situ grouting materials performance goals include (a) provide physical stability to the waste site (b) inhibit mobilization of contaminants of potential concern by limiting waste site hydraulic conductivity and (c) provide a constant chemical environment so that the solubility of the contaminants of potential concern can be predicted. The durability estimate is based on the dissolution rate of the chemical elements, which constitute the waste stabilization materials, namely the chemical components aluminum, silicon, and calcium. Such an estimate assumes that factors such as the recrystallization of mineral structures within the grout material are negligible in comparison to the rate of dissolution of the waste form and that the SDA climate remains virtually unchanged.

To determine the buffering capabilities of the grout and to determine the chemical compatibility of the grout with the surrounding soils (whether these soils are INEEL silty clay soils or elsewhere), the pH and eH of the leachate water for each bath of the ANS 16.1 testing described above was analyzed. A detailed discussion of how eH and pH relate to chemical buffering of the waste contaminants and long term durability is included in Appendix E. Table 13 summarizes the neat grout pH and eH data from the ANS 16.1 testing.

The range of pH range was measured at 9.6 to 11.2 and the eH ranged less than 390 mV during the 90-day testing. This pH and eH data can be used by computer models that calculate the long-term response of the grout in a flowing water situation.

The chemical properties of the grout material may effect, and be affected, by the chemical properties of the waste site ground water and waste materials. The pH and eH are two chemical properties, which are particularly important. for estimating the behavior of grout materials in the waste site chemical environment. Changes in pH and/or eH can affect the dissolution/precipitation of mineral material and the dissolution/evolution of gasses and also the adsorption/desorption of aqueous species. The pH can affect the solubility of the grout and waste materials by altering the chemical speciation in aqueous solution. PH is defined as the negative logarithm of the hydrogen ion activity. The eH is the electrical potential for moving electrons between oxidized and reduced species in an aqueous solution and is measured in millivolts. eH is important for estimating the behavior of elements, which can exist in more than one oxidation state, such as technetium, chromium, plutonium, neptunium, and americium. Elements such as technetium and chromium are very insoluble in reducing conditions, but become very soluble in a more oxidized environment. Some elements can exists in as many as four oxidation states. Each oxidation state has a different solubility because the oxidation state (and pH) affects the speciation of the element.

The pH and eH of the leachates were measured during the leach tests described above. All grouts produced alkaline, moderately oxidizing solutions having a pH in the range 10.9 (GMMENT-12) to 11.4 (TECT HG) and eH of about 225 mV (Saltstone) to 390 mV (U.S. Grout). For comparison the ground water at the SDA is slightly alkaline, at about 7.16 pH, moderately oxidizing, and is in equilibrium with calcite and variable CO₂ soil gas concentration (Pace and Hull 2000).

Appendix C gives a complete listing of pH and eH during the ANS 16.1 testing for use in modeling the buffering properties of the grout.

Table 13. Summary of pH and eH measurements of the leachate during ANS 16.1 testing.

Grout Name	Range pH	Range eH mV
U.S. Grout	9.7 to 11.2	Less than 390
TECT HG	9.6-11.4	Less than 384
Enviro-Blend	9.6 to 11.1	Less than 375
GMMENT-12	10.6 to 11.2	Less than 313

Durability Estimate Based on Leach/eH-pH Data

The “durability” of a waste stabilization material is defined as the length of time through which it will function as designed. For the Subsurface Disposal Area, these results indicate that the properties of the in situ grouting materials will remain virtually unchanged for many thousands of years.

The application of in situ grouting at the SDA will produce tabular bodies of grouted buried waste material two to three meters thick and several meters in length and breadth (this is caused by the sequence of grouting). The monolith will be resting upon basalt bed rock and will be covered with about 2 m of soil and an engineered cap (Armstrong et al. 2002) and will be below the frost line. Typically, soils at the SDA are virtually water-saturated at the basalt soil interface and contain less pore water near ground surface (J. Weidner, personal observation, 1991) with about 25% average pore filling (estimated by Dr. Al Sehn of the University of Akron). The grout monolith will be subjected to virtually no wet-dry or freeze-thaw cycles. The compressive strength and tensile strength of both pure grout and grout with waste

materials indicate that the grout monolith will not be affected by seismic events. The remaining grout degradation mechanism is interaction with SDA ground water.

An estimate of the rate of grout erosion by dissolution is computed from leach rate data measured by the ANS/ANSI 16.1 leach procedure. ANS/ANSI 16.1 is a standard test method designed to determine the release rate of contaminants from porous-media waste forms such as cement-based grout used to stabilize waste materials. The AN/ANSI 16.1 procedure measures the dissolution rate of the elements of interest into a specified amount of demineralized water, i.e., pure water, at STP, over specified periods for a total of 90 days.

Under the above assumptions, the time required for 1% dissolution of the known grout components (aluminum, silicon, and calcium) was estimated. This calculation assumed a 2-m thickness for a pure grout monolith, as it would be applied in the field, and using data from the ANSI 16.1 43 day test interval (presented in this report) and the 8.5 cm/year average water infiltration rate at the SDA. Results of the computations indicate that “tens of thousands of years” will be required for loss of 1% of the chemical constituents composing the waste form materials. For example, GMENT-12 would require 15e4 years for one percent aluminum loss, 16.3e3 for one percent silicon loss, and 39e3 years for one percent calcium loss, and SALT STONE grout data indicated 32e4 years for one percent aluminum loss, 13e3 years for one percent silicon loss, and 15e3 years for one percent calcium loss. All the tested grout materials had comparable material loss rates.

3.6.2 Physical and Chemical Testing of Interference/Grout Mixtures

Both physical and chemical testing protocols were performed on cured grout/interference samples consisting of mixtures of neat grout and determined maximum tolerance conciliations of either soil, organic, sludge, nitrate soil. Physical testing including porosity, leach testing, hydraulic conductivity testing, compressive and spitting tensile strength testing. Chemical testing included ensuring eH and pH of the leachate water during ANSI 6.1 leach testing. All samples for physical and chemical testing for the neat grouts mixed with interferences were cured in a special curing room in which the temperature of the room was kept constant at $73.3^{\circ}\text{F} \pm 3^{\circ}\text{F}$ and 100%, relative humidity. This eliminated unwanted differential temperature at the surface of the samples during curing which could affect the results. In an actual in situ case, there would not be surfaces exposed to surface environmental fluctuations during curing.

Physical Testing Results

Porosity. Measuring the porosity of the neat grout interference mixtures (another physical property) was planned. However, the procedure (ASTM C 642-97) called for baking the cured samples which has historically produced large cracks in the samples. Dr. Al Sehn of the University of Akron estimated the porosity of a cured mixture of 50 wt% soil and grout to be 28% for the GMENT-12.

Leach Testing. Table 14 summarizes leach results for interference samples. These leach indexes were not degraded more than one or two orders of magnitude from those shown for the neat grout in Table 11. Even though there was roughly a two-order-of-magnitude change, leach indices on the order of 10, indicate a very durable material.

Table 14. Leach results for interference samples (Leach Index ANS 16.1).

Grout	9 wt% Organic Sludge	12 wt% Nitrate Salt	50 wt% INEEL Soil
U.S. Grout	10.8 ± 0.7	11.6 ± 0.5	11.4 ± 0.8
TECT HG	10.4 ± 0.6	10.6 ± 0.7	10.5 ± 0.9
Enviro-Blend	12.1 ± 0.7	12.2 ± 0.9	12.6 ± 0.9
GMENT-12	10.3 ± 0.6	10.9 ± 0.6	10.6 ± 0.5
Saltstone	10.4 ± 0.4	10.4 ± 0.4	10.5 ± 0.5

Results reported ± one standard deviation.

Hydraulic Conductivity Testing. Table 15 shows the average hydraulic conductivity as measured in monoliths formed by the neat grouts and neat grout mixed with interferences. The testing protocol followed the essence of ASTM D 5084-90. Although there was a marked degradation for samples containing 12 wt% nitrate salts (as much as a two order degradation), there was little degradation in hydraulic conductivity for up to 9 wt% organic interference and 50 wt% soil. It is noted here that a mixture of grout and soil at 50 wt% soil is similar to what is expected in a jet-grouted monolith for the INEEL transuranic pits and trenches. In all cases shown in Table 15, the hydraulic conductivities are extremely low and definitely show an improvement over the ungrouted pits and trenches of around 10^{-5} cm/s (Loomis 97).

Compressive and Splitting Tensile Strength Testing. Table 16 provides splitting tensile strength values for mixtures of each grout with the various interferences. Table 17 provides compressive strength for grout with interferences. Using neat grout as a baseline (see Table 9), the grouts showed a marked reduction in physical strength from the introduction of interferences. For instance, the Enviro-Blend grout had a very poor neat grout compressive strength (150 psi) and basically low tolerance to any interferences. On the other extreme, TECT HG and GMENT-12 had an excellent neat grout compressive strength (6,320 and 7,639 psi, respectively) and high tolerance to all three interference types. For instance, both GMENT-12 and TECT HG monoliths had robust compressive strength (greater than 1,500 psi) even with 50 wt% soil and 25 wt% nitrate salts. However, both grouts exhibited less by mass tolerance to the simulated organic sludge material (tolerance for GMENT-12 was 9 wt% organic sludge and for TECT HG 12 wt%). The U.S. Grout across the board showed higher tolerance to the interferences. For instance, U.S. Grout could still produce stand-alone monoliths with 75 wt% soil and 75 wt% nitrate salts (refer each to Table 6 which shows the interference tolerance screening test results). GMENT-12 maintained the highest compressive strength in the presence of organic sludge (at 9 wt% organic sludge the compressive strength remained above 5,000 psi as shown in Table 17).

During ANS 16.1 testing for the neat grout samples with interferences (organic sludge, soil, and nitrate salts) each leachate was tested for pH and eH. The results of measurements on interference-material containing samples are shown in Appendix D. The results indicate that none of the interference materials have a significant affect on the eH and pH values. The pH measurements of leachates from grout with interferences materials versus leachate from grout without interferences materials were virtually identical within experimental error. The eH values of the two groups are nearly identical with the leachate from interference material containing grouts having higher values. For example, U.S. Grout leachate has average eH of 245 mV, whereas the leachate from U.S. Grout containing interference materials has average eH of about 405 mV. Both sets of grout leachates are oxidizing.

Table 15. Hydraulic conductivity values in cm/s for mixtures of each grout with the various interferences (cm/s).

Test Specimen	Interference Amount and Type	Grout Product				
		GMMENT-12	Enviro-Blend	Salt Stone	TECT HG	U.S. Grout
A	12% Nitrate Salts	5E-07	9E-06	2E-08	6E-09	7E-09
B	12% Nitrate Salts	7E-08	6E-06	2E-08	2E-08	2E-08
A	9% Organic Sludge	2E-09	7E-08	4E-08	5E-09	1E-08
B	9% Organic Sludge	4E-09	5E-08	2E-08	1E-09	2E-08
A	50% INEEL Soil	6E-09	7E-07	8E-08	2E-08	3E-09
B	50% INEEL Soil	1E-08	1E-06	8E-08	8E-09	2E-08

Table 16. Splitting tensile strength values for mixtures of each grout with the various interferences (psi).

Test Specimen	Interference Amount and Type	Grout Product				
		GMMENT-12	Enviro-Blend	Salt Stone	TECT HG	U.S. Grout
A	12% Nitrate Salts	373	5	84	313	256
B	12% Nitrate Salts	258	5	69	296	254
C	12% Nitrate Salts	246	5	95	176	183
D	12% Nitrate Salts	416	4	104	315	190
C	12% Nitrate Salts	413	4	80	376	262
A	9% Organic Sludge	515	19	97	347	187
B	9% Organic Sludge	488	17	93	330	197
C	9% Organic Sludge	513	18	106	241	173
D	9% Organic Sludge	516	19	100	312	152
C	9% Organic Sludge	476	18	98	320	166
A	50% INEEL Soil	308	4	134	313	231
B	50% INEEL Soil	417	3	92	319	257
C	50% INEEL Soil	352	3	161	283	193
D	50% INEEL Soil	359	2	143	328	225
C	50% INEEL Soil	334	3	135	303	201

Table 17. Compressive strength values for mixtures of each grout with the various interferences (psi).

Test Specimen	Interference Amount and Type	Grout Product				
		GMMENT-12	Enviro-Blend	Salt Stone	TECT HG	U.S. Grout
A	12% Nitrate Salts	5057	28	662	3034	4364
B	12% Nitrate Salts	4236	28	621	2256	4378
C	12% Nitrate Salts	4201	25	611	2518	4781
D	12% Nitrate Salts	6273	27	646	1556	3522
C	12% Nitrate Salts	5149	28	653	2553	2914
A	9% Organic Sludge	5502	114	973	1987	3147
B	9% Organic Sludge	5375	114	1014	2030	3388
C	9% Organic Sludge	4958	103	1020	1945	3204
D	9% Organic Sludge	5332	123	1040	1994	2843
C	9% Organic Sludge	5842	128	1041	1952	2539
A	50% INEEL Soil	2348	41	1117	1832	2553
B	50% INEEL Soil	3303	45	1030	1895	2405
C	50% INEEL Soil	2376	34	1092	2107	2397
D	50% INEEL Soil	2440	31	1062	1874	2702
C	50% INEEL Soil	2716	28	1050	2178	2617

3.7 VOC Encapsulation Testing

To study the potential VOC migration retardation in a grouted matrix created by jet grouting a buried waste site, microencapsulation and macroencapsulation tests were performed. The microencapsulation simulates the case in which the neat grout is intimately mixed with the waste matrix during the violent jet grouting operation. In this case, the organic sludge described in Table 7 was mixed with the neat grout and allowed to cure. The macroencapsulation case is where a region of VOCs is completely surrounded by a neat grout layer. For this case, a cylinder of neat grout was used as a “macro” and the pure organic sludge was placed inside the cylinder, the end sealed, and the VOC migration was due to diffusion of the VOCs through the surface area of the matrix.

3.7.1 Microencapsulation Testing

Each sample was prepared at 9 wt% sludge and 91 wt% neat grout with the sludge composition given in section 3.5.2 for each of the grouts. Enough grout-interference mix was prepared to allow for the creation of two samples of each of the three candidate grouts at the maximum identified organic sludge loadings. The organic sludge mixture recipe is the same as given in Table 7. The neat grout and VOC mixture were blended and poured into 7.62 cm diameter by 6.35 cm high cylinder molds.

After the cylindrical monolith cured, the monoliths were placed in a specially prepared airtight 305-mL chamber. Within the chamber, the sample was placed in the middle of moist soil to simulate field conditions inside a monolith. This chamber was of sufficient volume to allow removal of small syringes (nominally 5 cc) of air mixed with volatile organic off-gas without compromising the overall gas volume of the chamber. The testing followed a 90-day testing cycle in which the air sample is withdrawn and tested for the four volatile organics every 10 days using gas chromatography for each of the chambers. In addition, in a separate chamber, pure sludge material control was allowed to off gas and similarly tested for the VOCs.

The results of the microencapsulation testing are shown in Table 18 for the three grouts

When evaluating the offgas of the pure sludge sample in the chamber, there was an essentially instantaneous release (within minutes) for the all of the volatile organics sludges due primarily to a relatively low vapor pressure. This compares to the extremely low offgas rates observed for all of the grouts shown in Table 18.

Examining the data in Table 18, the release rate of the volatile material is extremely low (with the exception of day 10 results) compared to the release rate of just the organic material which is essentially 100% released in a matter of minutes. Day 10 is considered bad data in the evaluation of the air sample across all the grouts and can be thrown out of the data base. TECT HG and GMENT-12 show very consistent results with U.S. Grout showing a slightly better retardation of VOC offgas. For each 10-day testing interval the amount of material released was between $4e-5$ to $6e-4$ times the source term. To work with an order of magnitude, the amount released is approximately $e-5$ to $e-6$ times the source term per day (meaning “hundreds of thousands of days” for complete release). This means that in rough terms, the complete release of the volatile organics in the intimately mixed organic sludge could be retarded for on the order of thousands of years (1,000 years = 365,000 days), which is within the chemical half-life of these materials in surrounding INEEL soils.

Table 18. Gas phase concentration and mass percentage data for microencapsulation test.

(a) GMENT-12

Day	CTET (mg/L)	PCE (mg/L)	TCE (mg/L)	TCA (mg/L)	CTET (%)	PCE (%)	TCE (%)	TCA (%)
0	9.55	2.97	7.39	0.04	0.021	0.023	0.064	BDL
10	135.97	26.36	49.78	0.68	0.299	0.205	0.431	0.005
20	9.16	5.30	7.15	BDL	0.020	0.041	0.062	BDL
30	11.32	10.86	9.13	BDL	0.025	0.084	0.079	BDL
40	10.37	5.10	7.43	BDL	0.023	0.040	0.064	BDL
50	9.10	7.82	7.94	BDL	0.020	0.061	0.069	BDL
60	7.63	3.95	5.83	BDL	0.017	0.031	0.050	BDL
70	6.34	4.92	6.70	BDL	0.014	0.038	0.058	BDL
80	8.08	5.29	6.44	BDL	0.018	0.041	0.056	BDL
90	7.82	5.30	6.72	BDL	0.017	0.041	0.058	BDL

(b) TECT HG

Day	CTET (mg/L)	PCE (mg/L)	TCE (mg/L)	TCA (mg/L)	CTET (%)	PCE (%)	TCE (%)	TCA (%)
0	6.01	2.01	6.38	0.23	0.012	0.014	0.049	BDL
10	25.01	7.67	22.24	0.30	0.048	0.053	0.170	BDL
20	14.17	6.65	11.97	0.13	0.027	0.046	0.091	0.001
30	10.20	6.21	10.39	BDL	0.020	0.043	0.079	BDL
40	12.95	5.40	10.87	BDL	0.025	0.037	0.083	BDL
50	11.14	7.90	11.36	BDL	0.022	0.054	0.087	BDL
60	9.91	4.55	9.10	BDL	0.019	0.031	0.070	BDL
70	6.72	4.85	9.12	BDL	0.013	0.033	0.070	BDL
80	6.25	4.28	7.56	BDL	0.012	0.029	0.058	BDL
90	6.58	4.57	7.80	BDL	0.013	0.031	0.060	BDL

(c) U.S. Grout

Day	CTET (mg/L)	PCE (mg/L)	TCE (mg/L)	TCA (mg/L)	CTET (%)	PCE (%)	TCE (%)	TCA (%)
0	5.90	6.33	9.59	BDL	0.014	0.054	0.092	BDL
10	9.21	6.07	6.67	0.19	0.022	0.052	0.063	0.001
20	4.68	5.09	4.34	BDL	0.011	0.044	0.041	BDL
30	6.30	13.14	6.37	BDL	0.015	0.113	0.061	BDL
40	1.94	2.28	3.98	BDL	0.005	0.020	0.038	BDL
50	2.24	4.18	2.98	BDL	0.005	0.036	0.028	BDL
60	1.92	2.26	2.05	BDL	0.005	0.019	0.020	BDL
70	1.45	2.55	2.43	BDL	0.004	0.022	0.023	BDL
80	1.66	2.54	2.25	BDL	0.004	0.022	0.021	BDL
90	1.52	2.53	2.27	BDL	0.004	0.022	0.022	BDL

Notes:

All values reported are average of three (3) separate samples/bottles.

BDL = Below Detection Limit

Sample size of 7.62 cm diameter by 6.35 cm height and air volume of 15.42 mL.

3.7.2 Macroencapsulation Testing

Macroencapsulation testing was performed identically to microencapsulation testing only the sample preparation was completely different. For the three grouts used in implementability testing, triplicate monoliths were prepared. The monoliths were prepared by creating a cylindrical sample of neat grout in 7.62 cm diameter by 6.35 cm high cylinders. Immediately after mixing and pouring the soil/grout mixture into the cylindrical sample holders, a 1-in. outside diameter rod was inserted exactly to within 1 in. of the bottom of the sample holder such to allow the rod to remain vertical during the curing process. The samples were allowed to cure 14 days similar to curing techniques used for physical testing samples (i.e., using moisture controls). Once cured, the monolith was carefully removed from the case and the rod withdrawn and the sample inspected for visible cracking due to withdrawal of the rod. The interior of the cavity was quickly hand filled and tamped to within 1 in. \pm 1/8 in. of the top with a measured mass of the organic sludge material. The samples are evaluated for TCE, TCA, PCE, and CCl₄. Once filled with sludge, a prepared mixture of quick setting sealant with a special top made of grout was placed in the top 1 in. of the cavity thus sealing the sludge in place. After the top was cured, the monolith was cleaned with a damp rag and placed in a specially prepared airtight chamber similar to that used in the microencapsulation testing. The same testing protocol of withdrawing small amount of gas from the chamber at regular intervals that was used in the microencapsulation testing was used for the macroencapsulation testing.

Data from the macroencapsulation tests are shown in Table 19 with the unexpected result that there is not lower release of VOCs for the macroencapsulation compared to the microencapsulation results shown in Table 18. This was primarily expected because there was certainly a higher concentration of VOCs near the surface of the monolith for the microencapsulation case compared to the macroencapsulation case. In fact, for the GMENT-12 grout there was a general increase in release. Comparing the data between micro and macro tests show that for all cases the TCE tested with the highest release for both macro and micro testing. For the TECT HG grout the macro %age released results are generally across the spectrum of VOCs lower than the micro as expected (macro is generally lower than 0.05% and the micro is generally lower than 0.1%). For the U.S. Grout, there is less of an effect but generally, the macro is slightly lower than the micro tests (macro generally lower than 0.08% and the micro generally lower than 0.1%). However, for the GMENT-12 there is a larger difference than for the other grouts in that the macro test showed a higher release (macro generally lower than 0.175% and the micro generally lower than 0.1%). This increase was certainly not expected in that it was thought that the macroencapsulation would simulate a pure diffusion of the VOCs through the neat grout matrix and thus show a marked decrease in VOCs showing up in the gas volume of the chamber when compared to the microencapsulation results. The explanation for the higher release of VOCs for the GMENT-12 grout for the macroencapsulation tests compared to the microencapsulation tests is due to an obvious crack in the end plug of the samples for this grout as shown in Figure 4. This crack formation was most likely caused by differential curing between the seal material, the top cap, and the basic cylinder itself. Figures 5–6 show less obvious cracking in the base plugs for the U.S. Grout and TECT HG grout, respectively.

Even with the crack in the base plug of the GMENT-12 grout, the release values generally are below 0.175% per 10-day period which equates to a general release rate of e-4 times the source term per day which is still much lower than the instantaneous release from an ungrouted piece of organic sludge material. At e-4 times the source term released per day would equate to a release of 3% released per year or in general, there would be a retardation of VOC flow on the order of 100 years. Of course, for the TECT HG grout and the U.S. Grout, the expected retardation is less than that discussed for the micro tests (i.e., retardation for the macroencapsulation of these materials would be expected to last for thousands of years).

Table 19. Gas phase concentration and mass percentage data for macroencapsulation test.

(a) GMENT-12

Day	CTET (mg/L)	PCE (mg/L)	TCE (mg/L)	TCA (mg/L)	CTET (%)	PCE (%)	TCE (%)	TCA (%)
10	61.88	13.12	26.95	BDL	0.151	0.111	0.281	BDL
20	53.38	21.42	25.63	0.05	0.130	0.181	0.268	0.001
30	33.08	10.73	16.54	6.45	0.081	0.091	0.173	0.058
40	23.28	13.40	13.52	8.31	0.057	0.113	0.141	0.074
50	14.52	19.33	11.38	8.14	0.035	0.163	0.119	0.073
60	5.76	14.02	7.98	6.80	0.014	0.118	0.083	0.061
70	3.33	9.67	4.64	4.85	0.008	0.082	0.048	0.043
80	2.43	16.74	4.16	5.15	0.006	0.141	0.043	0.046
90	0.83	18.74	3.60	4.45	0.002	0.158	0.038	0.040

(b) TECT HG

Day	CTET (mg/L)	PCE (mg/L)	TCE (mg/L)	TCA (mg/L)	CTET (%)	PCE (%)	TCE (%)	TCA (%)
10	2.06	2.71	22.24	BDL	0.005	0.023	0.232	BDL
20	1.24	0.94	2.17	BDL	0.003	0.008	0.023	BDL
30	7.97	2.69	5.62	1.00	0.019	0.023	0.059	0.009
40	1.19	0.75	1.44	0.33	0.003	0.006	0.015	0.003
50	0.93	0.92	1.38	0.39	0.002	0.008	0.014	0.003
60	0.76	0.62	1.29	0.28	0.002	0.005	0.013	0.002
70	0.19	0.37	1.54	BDL	0.001	0.003	0.016	BDL
80	1.03	0.85	1.65	0.40	0.003	0.007	0.017	0.004
90	0.94	0.91	1.98	0.44	0.002	0.008	0.021	0.004

(c) U.S. Grout

Day	CTET (mg/L)	PCE (mg/L)	TCE (mg/L)	TCA (mg/L)	CTET (%)	PCE (%)	TCE (%)	TCA (%)
10	15.48	4.57	8.62	BDL	0.038	0.039	0.090	BDL
20	11.06	2.31	5.66	0.22	0.027	0.020	0.059	0.002
30	13.14	2.94	6.35	0.11	0.032	0.025	0.066	0.001
40	11.04	2.43	6.01	1.11	0.027	0.021	0.063	0.010
50	13.52	4.45	7.38	1.51	0.033	0.038	0.077	0.014
60	9.37	5.26	7.08	7.32	0.023	0.044	0.074	0.065
70	10.12	2.31	6.29	1.28	0.025	0.020	0.066	0.011
80	20.59	5.99	11.53	2.96	0.050	0.051	0.120	0.027
90	15.67	5.63	11.90	2.56	0.038	0.048	0.124	0.023

Notes:

All values reported are average of three (3) separate samples/bottles. BDL = Below Detection Limit. Sample size of 7.62 cm diameter by 6.35 cm height and air volume of 15.42 mL.

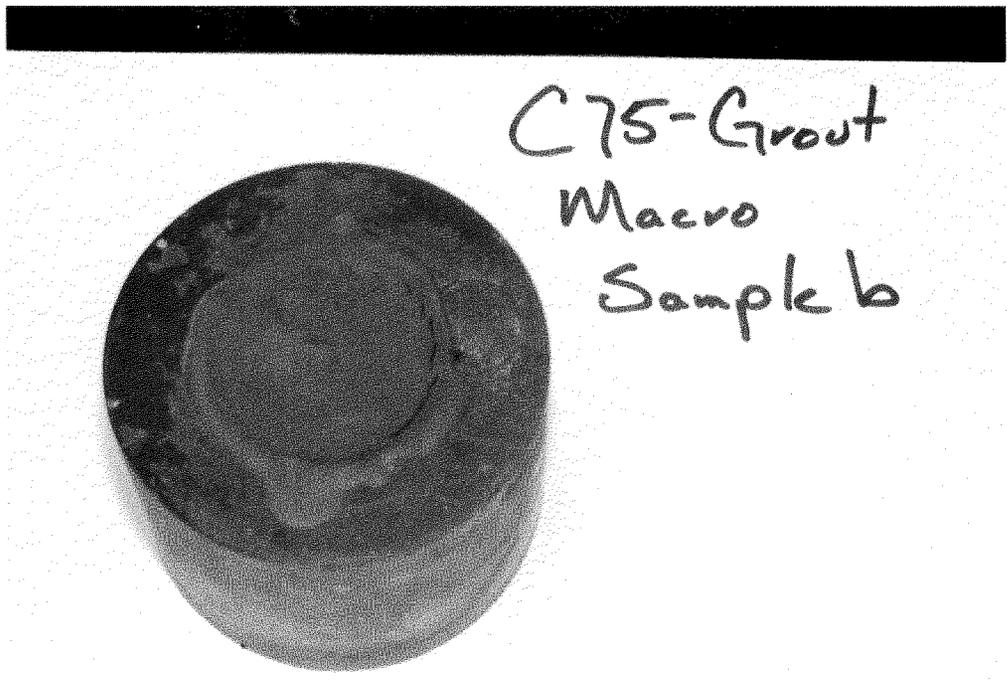


Figure 4. Macroencapsulation cylinder for GMENT-12 (C-75, Tank Closure Grout).

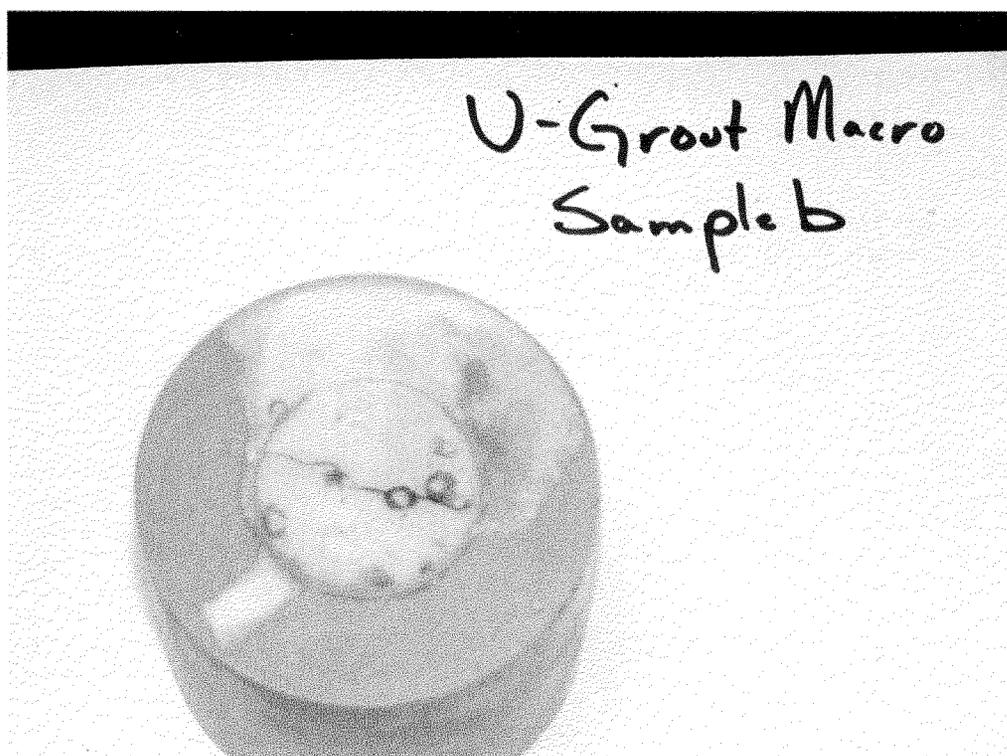


Figure 5. Macroencapsulation cylinder for U.S. Grout.

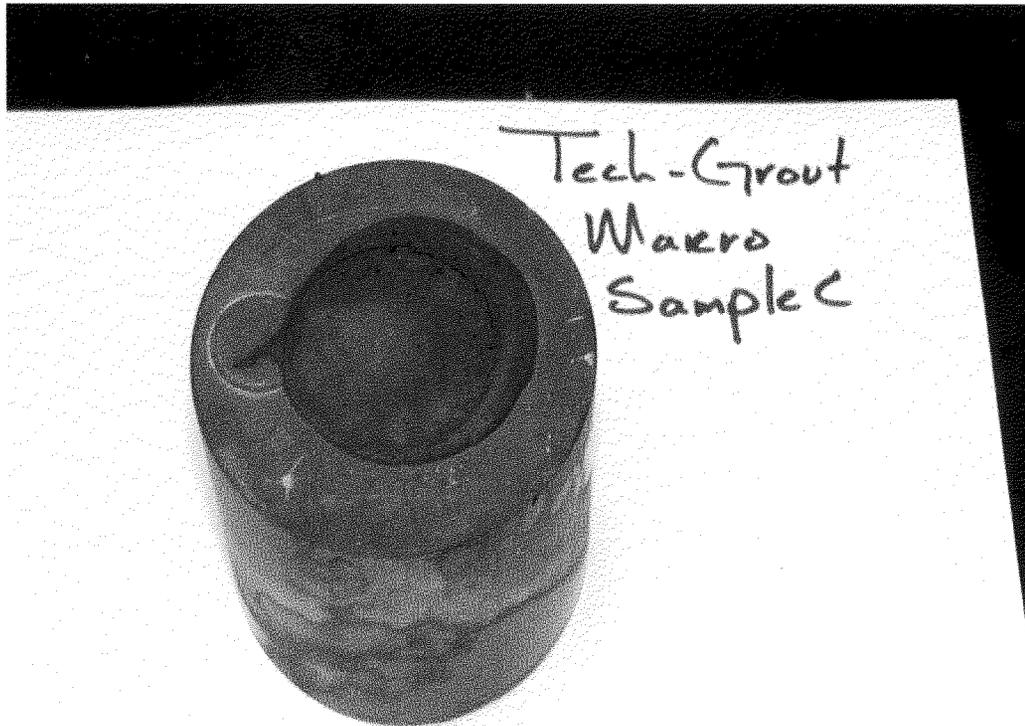


Figure 6. Macroencapsulation cylinder for TECT HG.

3.8 Special Testing for Wax-Based Grouts

A special testing protocol was performed to examine settling properties of introduced boron compounds in Waxfix. Boron-10 is commonly used in neutron absorber in reactor applications to control reactor criticality. The settling of the boron in the Waxfix is a very undesirable property in that introduction of the Waxfix in a pit containing Pu-239 and U-233 and U-235 raises the possibility of an uncontrolled criticality because of the effective increase in moderation afforded by the hydrocarbon wax increases the potential for a criticality. As an example, the neat Waxfix may fill a box containing a 800g piece of pure plutonium metal and criticality calculations suggest that this is a potential for a criticality. Therefore, the test plan called for a screening test in which a nearly saturated solution of sodium tetraborate in glycerin was mixed with molten Waxfix (140–160°F) such that there was a net 1 g/L of B-10 (the effective boron speciation that has excellent neutron absorption properties). At 1 g/L there was a large safety factor in criticality calculations such that the conservative hypothetical plutonium-239 concentration of particles in a pit would not go critical.

Basically, when correctly mixed and cooled there was a large separation in the boron compounds as shown in Figure 7 during the cooling process. The mixture was allowed to cool down over a multiple day period (5-days), thus simulating the “cooldown” in an injected pit and then examined for settlement by performing ICP-mass spectroscopy (ICP-MS) on samples for boron. Results showed both a strong visual separation of the mixed boron compounds which was in agreement with the ICP-MS results. As an

example, the sample was mixed with 56 g of sodium tetraborate per liter of wax and the post cooling separation from samples analyzed with ICP-MS was top of the sample 18mg/L, middle of the sample 43mg/L and the bottom was 316 mg/L. These results suggest that a completely different introduction scheme be devised to first introduce the boron and then have it stay distributed during cooling.

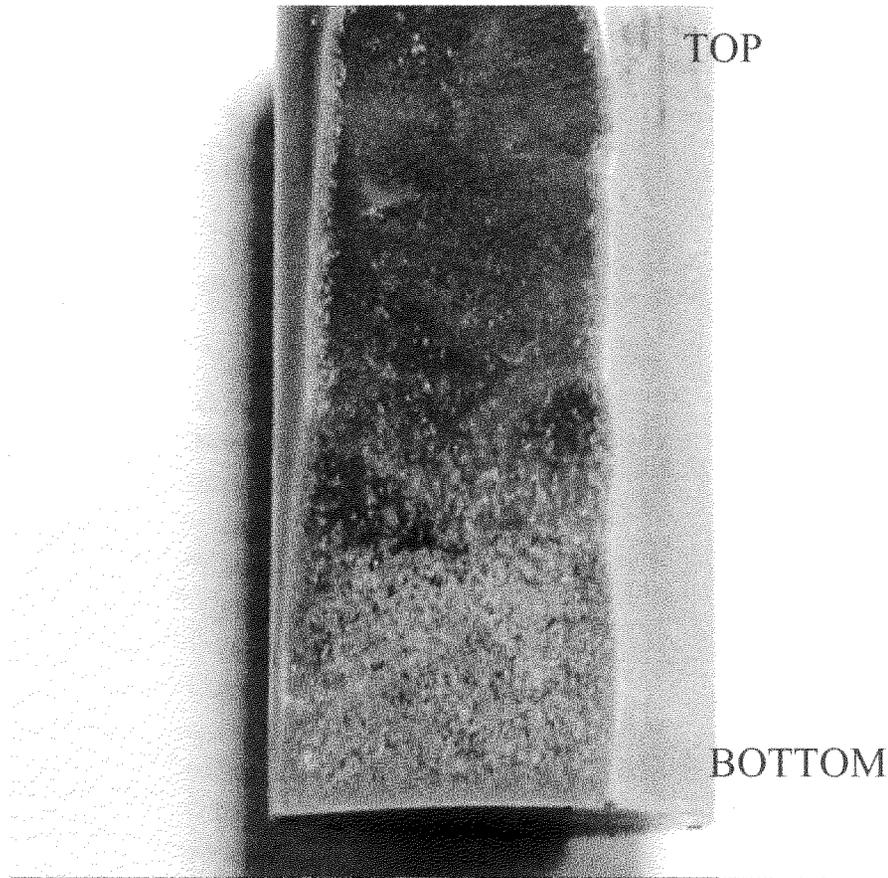


Figure 7. Separation of sodium tetraborate/glycerin during cooling in Waxfix.

In summary, it is still possible that the B-10 can be introduced by other means into the cooling Waxfix and still maintain a 1g/L concentration throughout the cooling process. These processes have not been identified in this document; however, are recommended for future work. Because of the negative results for the boron distribution testing, objectives relating to Btu content and combustibility of the Waxfix grout were not performed.

3.9 Use of Powdered Activated Carbon in Grouts

Past studies (Hebatpuria, Vikram et al., "Leaching Behavior of Selected Aromatics in Cement-Based Solidification/Stabilization under Different Leaching Tests," *Environmental Engineering Science*, Vol. 16, Number 6, 1999) suggested that by adding inexpensive reactivated carbon to cement that there was a significant lowering of the diffusion coefficient for aromatic hydrocarbons under ANS 16.1 leaching protocol. Adding inexpensive reactivated carbon or alternatively activated carbon, to a grout matrix during jet grouting could also increase the leach index and effectively lower the diffusion of volatile organics in the matrix. An analysis of the potential use of powdered activated carbon (PAC) as an

absorber for volatile organic hydrocarbons was performed and a complete report on that work is in Appendix B. The basic findings of that work are as follows.

Addition of PAC to the exterior barrier confining the bulk of the waste could reduce the target VOC concentrations at very low concentrations. Using several conservative assumptions, this barrier is expected to be effective for approximately 30 years. After that time, the weakest adsorbed VOC could be displaced by a more strongly adsorbed VOC and the displaced VOC would enter the vapor phase outside the cell. While the 30-year life may not appear good, there are two main reasons to think this may be underestimated by a factor of 10 to 100:

The equilibrium vapor phase concentrations are very high. Including water in the estimates would reduce these values by at least an order of magnitude. Sorption of the VOCs to the solid matrix within the cell could also reduce these concentrations by an order of magnitude. Both these effects would drastically reduce the amount of VOCs being transported to the barrier and the PAC. Vapor phase VOC concentrations need to be determined for the main cell design.

Effective diffusivities of the VOCs in the main cell and within the barrier may greatly reduce the transport of VOCs. The values used were deduced from gas phase values and correspond to transport in soils with 29% porosity. If the main cell and the barrier porosities are smaller and if the materials are retarded in their movement by constant sorption/desorption on the solid matrix, the amount of VOCs transported to the barrier and the PAC would be substantially reduced. Effective VOC diffusivities need to be experimentally determined for the main cell and barrier wall materials.

There is one main reason why the estimate could be optimistic: The matrix surrounding the PAC could block access to the activated carbon microspore surface area and prevent sorption from occurring. This would drastically reduce the sorptive capacity of the PAC and prevent VOC sorption. PAC needs to be imbedded into a barrier matrix and adsorption equilibrium studies determined. In addition, analytical studies should be performed to study using PAC in monolith formation in the waste zone.

3.10 Down-Selection of Grouts for Implementability Testing

The down-selection from five grouts to three for use in the implementability tests involved a unique grading process considering parameters related to the field implementability (jet-groutability), chemical compatibility with the surrounding soils, and durability of the grout once grouted. The list of candidate grouts included Saltstone, Tank Closure Grout, TECT HG, Enviro-Blend, and U.S. Grout. The Waxfix paraffin-based grout and Saltstone were excluded from this selection process during screening testing as described before. The three highest scoring grouts were included in the implementability test program.

Grout Performance Scoring System. The overall performance of an in situ grout material is the sum of the contributions from five performance goals. Because the performance goals do not provide equal contributions to the overall performance of the grout, they are assigned a weighing factor according to their importance. From greatest importance to least importance these include:

- Monolith implementability variables (weighting factor = 5.0)
- Waste site physical stability variables (weighting factor = 4.5)
- Waste site permeability variables (weighting factor = 4.0)
- Grout monolith long-term durability (weighting factor = 4.0)
- Chemical effect of grout material on contaminant mobility (weighting factor = 3.0)

The ranking presented here is based on the assumption that the performance goal is one thousand years and the recognition that these properties are not independent variables.

One or more grout properties affect each performance goal. These include such properties as density, viscosity, cure temperature, hydraulic conductivity, and many others. Each grout property normally has a range of values that may vary from very good to poor. A numerical score is assigned to each acceptable value of each grout property. These are shown in Table 20.

The total score for a candidate grout is obtained by multiplying the performance objective weighting factor times the individual property score, then summing the total number of weighted scores as follows.

$$\text{Total Score for a Grout} = \sum_{I=1}^n \text{Property Score (I)} \times \text{Performance Objective Weighting Factor (I)}$$

The individual property scores assigned to the variables within each performance objective category are based on experience gained from past grouting operations at the INEEL. For example, experience has shown that grout column diameter depends on grout density. In general, higher density grouts produce larger diameter columns of stabilized buried waste. Therefore the denser grout materials are more desirable than less dense grouts and are assigned a higher individual property score. A second example is the set temperature. Low set temperature is more desirable than a higher set temperature because less shrinkage and less cracking are produced and therefore results in lower waste site permeability. In general, an individual property score of 25 was assigned to the least desirable, but acceptable, value of a particular property. An individual property score of 100 was assigned to the best value of a particular property.

3.10.1 Monolith Implementability Variables (Weighting Factor = 5)

Proper monolith development requires high performance from several variables to be successful. Those parameters that affect the implementability of the process include density of the grout, viscosity of the grout, the grout set time and the pressure filtration values. The density of the grout is directly related to column size and thus the ability of the system to overlap columns and produce a continuous monolith without significant untreated zones. Therefore, grout density has relatively high importance. The grout viscosity must be within the appropriate range to be properly injected. If the grout setting time is too fast, or marginally so, the grout could not be injected before set or a coherent monolith could not be produced. Pressure filtration is a measure of the tendency of a particle to stay in suspension and is used to estimate the pumpability of a material. Implementability is the highest priority because it is necessary for the formation of a monolith which stabilizes and encapsulates buried waste in situ.

3.10.2 Physical Stabilization of the Waste Site (Weighting Factor = 4.5)

Physical stabilization is required to prevent waste site subsidence and the resulting ponding and increased infiltration of surface water. Physical stability depends on several variables including low grout viscosity to promote void filling, tolerance of the grout to interference from waste materials and soil, and unsupported compressive strength. The unsupported compressive strength needs to be at least 50 psi or higher (NRC guideline for low-level waste landfills) to support the weight of the over-burden if void filling is not complete. The tolerance of the grout to interference from material such as organic materials, nitrate salts, and soil should be as high as possible to ensure physical stabilization. The viscosity should be as low as possible to promote virtually complete void filling. Note, however, that low grout viscosity is

Table 20. Weighting factors and scores.

Performance Objective Grout Property	Weighting Factor	Property Ranges	Property Score
Monolith Implementability Variables	5		
Initial Set Time		2 hr	50
		4 hr	75
		6 hr	100
Density		10 to 13 lb/gal	50
		13 to 15 lb/gal	75
		15 to 20 lb/gal	100
Pressure Filtration		0.5 to 0.6 min	50
		0.3 to 0.5 min	75
		0.1 to 0.3 min	100
Viscosity		7 min	50
		6 min	75
		<5 min	100
Physical Stabilization of the Waste Site Interference Tolerance	4.5		
		Organic at 3%	25
		Organic at 5%	30
		Organic at 7%	40
		Organic at 9%	50
		Organic at 12%	70
		Organic at 25%	80
		Soil at 50%	75
		Soil at 75%	100
		Nitrate at 12%	50
		Nitrate at 25%	75
		Nitrate at 50%	100
Long-term Durability Accelerated Leach Dissolution	4		
		<500 yr	50
		500 to 1,000 yr	75
		>1,000 yr	100
Waste Site Permeability Hydraulic conductivity	4		
		e-6 cm/s	50
		e-7 cm/s	75
		e-8 cm/s	100
Shrinkage		<0.1%	100
		0.1 to 0.5%	50
		0.5 to 1%	25
Porosity		0 to 5%	100
		5 to 25%	75
		25 to 50%	5
Temperature of Set		<100°F	100
		<120°F	75
		<140°F	60
		<150°F	50
		<160°F	40
		<170°F	25
Chemical Stabilization Chemical properties	3		
		pH = 8 to 10; eH < 0 mV	100
		pH = 8 to 10; eH > 0 mV	75
		pH > 10; eH < 0 mV	50
		pH > 10; eH > 0 mV	25

also an important property in the Monolith Implementability category and is not tabulated in the Physical Stabilization category. Physical Stabilization of the waste site is ranked only slightly lower than Implementability because Implementability is mandatory. Physical stability is ranked as the next most priority performance objective. Long-term Durability and Waste Site Permeability are also very important and are ranked nearly as high as Physical stabilization. Because Physical Stability of the site is required before Waste Site Permeability as a function of time or Long-term Durability can be considered, Physical Stability was assigned a higher value than these two categories.

3.10.3 Long-Term Durability (Weighting Factor = 4)

The long-term durability of the treated waste site is required to be 1,000 years or more. The long-term durability is the length of time that the grout will provide physical stability to the waste site, i.e., prevent subsidence or change of ground surface contour and/or control/buffer the site chemistry. Because the monolith is below the affect of frost, the grout degradation mechanism is dissolution to cause eventual collapse of the monolith. An absolute value for the long-term durability of the grout materials is difficult to determine. For assigning a relative durability value to the different grout compositions, the accelerated leach test will be used. The tests will provide conservative, relative dissolution rates of the grout materials under controlled laboratory conditions. It is understood that these dissolution rates are expected to be higher (much more conservative) than the actual dissolution rate of the grout materials when measured in SDA ground water saturated with calcite and atmospheric CO₂. Long-term durability is given slightly less priority than physical stability. The reason for this is that a lack of physical stability would allow unacceptable system degradation to occur within a few years if the ground surface contour collapsed and allowed ponding and infiltration of surface waters.

3.10.4 Waste Site Permeability (Weighting Factor = 4.0)

Reduction of the permeability of the buried waste site is an important mechanism to reduce the mobility of water borne and soil gas borne contaminants. The grout materials will be ranked according to hydraulic conductivity, the lowest hydraulic conductivity being most desirable. Variables related to waste site permeability are the grout temperature of set and grout isothermal shrinkage. In general, the lowest waste site permeability occurs when the grout material has low set temperature and low isothermal shrinkage, and therefore minimum crack formation. Low permeability grout is judged to have virtually the same priority as long-term durability because the primary goal of long-term durability is to provide long-term physical/chemical stability and thus minimize water infiltration into the waste. Low permeability becomes important when significant water can infiltrate the treated waste.

3.10.5 Chemical Stabilization (weighting factor = 3.0)

The composition of the grout may affect the chemical properties of the ground water and the chemical stabilization of potential contaminants. In general, the most desirable aqueous environment for the stabilization of uranium and other actinide contaminants in SDA ground water is one that has a pH of 8 to 10 and reducing conditions. Least desirable is one that has a pH greater than 10 and oxidizing conditions, equivalent to air. Chemical Stabilization is judged to have lower priority than Waste Site Permeability because the achievement of low permeability restricts contaminant movement to diffusion only and affects both volatile and nonvolatile contaminants.

3.10.6 Numerical Value of the Down-Selection

The down-selection for the cementitious grouts were based on the physical properties of the grout such as compressive strength, hydraulic conductivity, and leach resistance, and jet grouting properties such as set history, temperature of set, viscosity, density and pressure filtration, all applied to a weighting

criteria defined in the test plan. Table 21 presents the measured values and raw score for the measured grouts, and Table 22 presents the final score for the cementitious grouts.

Discussion of Scoring for the Various Grouts

Comparison of the four cementitious grouts (see summary Table 23) that passed the initial screening (recall that Saltstone did not meet the minimum screening criteria) show that the relative scoring for U.S. Grout (4150), TECT HG (4184), and GMENT-12 (3862) was relatively close while the Enviro-Blend (3010) was clearly a distant fourth. As expected, Enviro-Blend achieved a better leach index than any of the other grouts because of the presence of phosphate, but the other grouts were high enough in leach index and yet still have all the other desirable properties that the scoring came out higher. In fact, Enviro-Blend had virtually no resistance to interference tolerance and a relatively high shrinkage number such that a zero score was achieved for those parameters. Also, evaluation of the Waxfix paraffin-based grout was halted due to difficulties in achieving a reasonable distribution of the B-10 during a 5-day cooling period and therefore was also dropped. Therefore, using the agreed upon scoring system established in the test plan, three grouts were recommended for testing in the implementability phase including U.S. Grout, TECT HG, and GMENT-12.

Table 21. Measured values and raw score for the cementitious grouts.

PARAMETER	TEST DATA/TOTAL SCORE (0-100)									
	TECT HG		GMENT-12		U.S. Grout		Enviro-Blend		Saltstone	
INITIAL SET TIME (HOURS)	6	100	4.95	82	4.7	75	9.4	100	1.8	(Did not meet minimum requirement)
DENSITY (LBM/GAL)	18	100	15.4	100	13.7	75	14.8	75	13.3	100
PRESSURE FILTRATION (MIN-.5)	.008	100	.07	100	.03	100	.07	100	.023	100
VISCOSITY (MIN)	1.8	100	0.93	100	.9	100	2.7	100	1.8	100
INTERFERENCE TOLERANCE										
ORGANIC	12%	70	9%	50	5%	50	None	0	12%	70
SOIL	50%	75	50%	75	75%	100	None	0	50%	75
NITRATE (WEIGHT %)	25%	75	25%	75	75%	100	None	0	12%	50
pLEACH ANS 16.1(LI)	LI=10.3	80	LI=10.6	85	LI=9.9	75	LI=12.2	90	LI=10.6	85
HYDRAULIC CONDUCTIVITY CM/s	5.8e-9	100	7.3e-9	100	1.9e-8	100	1.5e-7	75	1.2e-8	100
SHRINKAGE	0.44%	50	1.82%	0	0.84%	25	3.16%	0	0.25%	50
TEMPERATURE OF SET DEGREES F	144	50	138	60	114	100	89.6	100	82F	100
EH/PH LEVELS	pH = 11.4 eH = 385 mV	25	pH = 10.7 eH = 193 mV	25	pH = 11.1 eH = 388 mV	25	pH = 10.7 eH = 365 mV	25	pH = 9.65 eH = 197 mV	75

Table 22. Final score for the various cementitious grouts.

Grout Property	Weighting Factor	Score TECT HG	Subtotal TECT HG	Score GMENT 12	Subtotal GMENT 12	Score U.S. Grout	Subtotal U.S. Grout	Score Enviro-Blend	Subtotal Enviro-Blend	Score Saltstone	Subtotal Saltstone
Initial Set time	5.0	100	500	81.7	408.5	75	375	100	500	0	0
Density	5.0	100	500	100	500	75	375	75	375	50	250
Pressure Filter	5.0	100	500	100	500	100	500	100	500	100	500
Viscosity	5.0	100	500	100	500	100	500	100	500	100	500
Interference Tolerance	4.5										
Organic		70	315	50	225	50	225	0	0	70	315
Soil		75	337	75	337	100	450	0	0	75	337
Nitrate		75	337	75	337	100	450	0	0	50	225
Leach	4.0	80	320	85	340	75	300	90	360	85	340
Hydraulic Conductivity	4.0	100	400	100	400	100	400	75	300	100	400
Shrinkage	4.0	50	200	0	0	25	100	0	0	50	200
Temp of Set	4.0	50	200	60	240	100	400	100	400	100	400
eH/Ph	3.0	25	75	25	75	25	75	25	75	75	225
Total Score			4184		3862		4150		3010		3692*
* Did not meet minimum requirement for set time(set time too fast)											

Table 23. Relative ranking of cementitious grouts.

Grout	Relative Rank
TECT HG	4184
GMENT-12	3862
U.S. Grout	4150
Enviro-Blend	3010

4. IMPLEMENTABILITY STUDIES

4.1 Introduction/Background

The implementability testing was conducted at the Applied Geotechnical Engineering and Construction test site in Richland, Washington, April 16-24, 2001. The full-scale implementability testing was designed to demonstrate the injectability of those grout formulations recommended from the bench testing. This testing provides essential information concerning the operational aspects and column development properties of chosen grout materials such that a down selection from three grouts recommended from the bench testing to one grout for field testing was possible. The three grouts that were chosen (see section 3.11) include GMENT-12, U.S. Grout Premium Grade, and TECT HG.

4.2 Objectives

The main objective of the implementability testing was to down-select a single grout from the three grouts chosen from the Bench testing discussed in the preceding chapter. In addition, data useful for the Field testing include those objectives listed in the test plan (Grant et al. 2000):

4.2.1 Test Objective 8

This objective is to evaluate the Field Implementability of the Grout Emplacement Process for Monolith Design and Application

Information relative to the functionality of hardware designs, safety equipment, grouting procedures, materials mixing, and delivery logistics will be collected during grout emplacement for the full-scale implementability tests. A combination of qualitative and quantitative data will be collected during and after grouting. A detailed examination of the grouted waste forms will also be performed to evaluate the quality and integrity of the grout and grout-soil columns verified by destructive examination.

4.2.2 Noncritical Test Objective C

This objective is to evaluate the Volume, Type and Expected Disposition of Secondary Waste

A quantitative analysis will determine the total volume and type of secondary waste generated as a result of the grouting process. The secondary waste determination will be used to group each type of waste according to disposal options for use in future in situ grouting operations.

Results of the applicability assessment will improve the estimates of cost and implementability associated with in situ grouting processing of buried waste at the SDA for the Operable Unit 7-13/14 Remedial Investigation/Feasibility Study.

The secondary waste determination will be used to estimate the total volume of secondary waste that may be expected during actual full-scale remediation of the SDA using in situ grouting. The estimate will be included in the applicability analysis section of the final report on the in situ grouting treatability study.

4.3 Test Hardware Description, Procedures

4.3.1 Hardware

The hardware consisted of water supply tanks, two in-series vortex mixers, and associated supply pumps, a JET 5 CASA GRANDE high-pressure injection pump, high-pressure injection lines, a CASA GRANDE C-6 rotopercussion drill jet grouting system, a typical mud balance, and the special thrust blocks discussed separately in this section. Figures 8, 9, and 10 show the Vortex mixer, high-pressure pump, and grout drilling rig set up on a thrust block, respectively.



Figure 8. In-line vortex mixers.

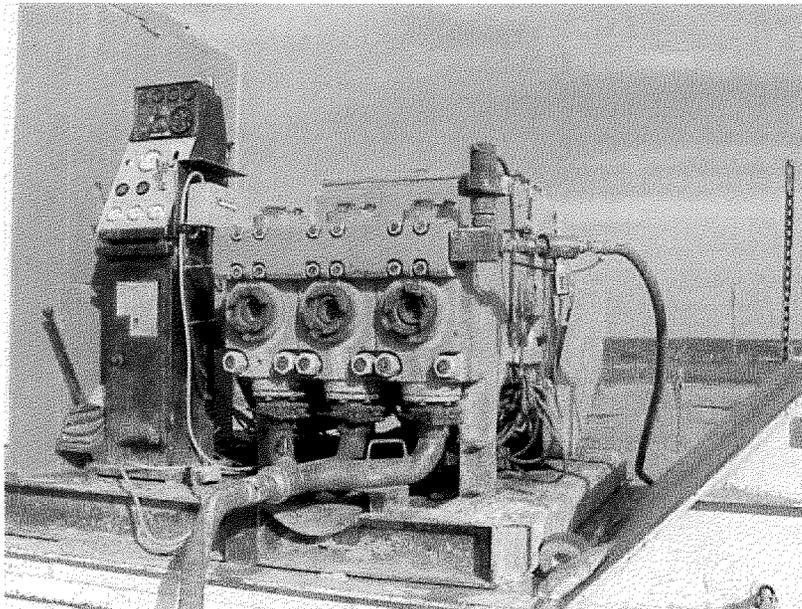


Figure 9. High-pressure pump.

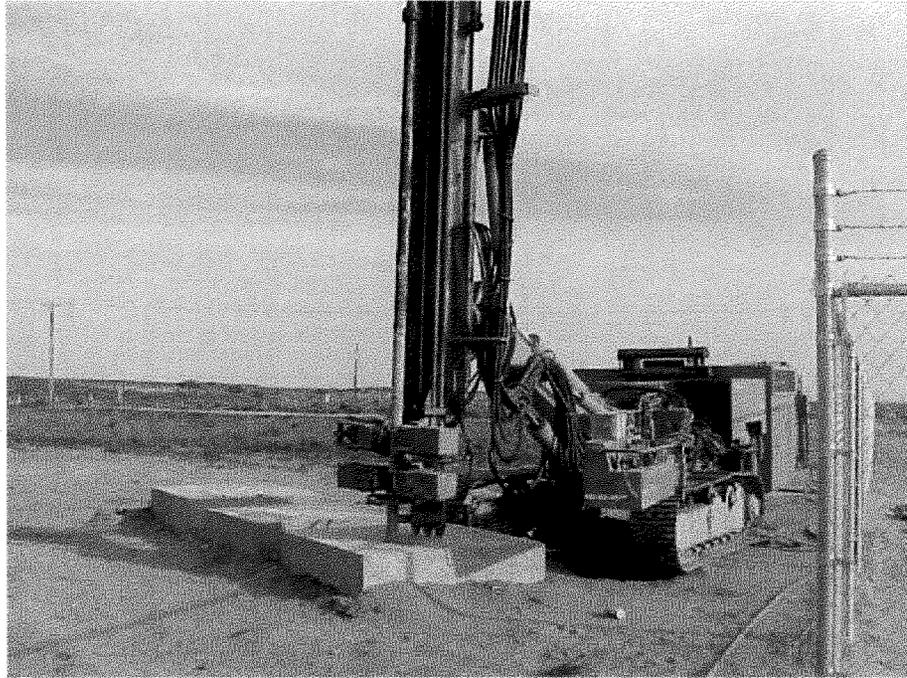


Figure 10. CASA GRANDE C-6 drill system on thrust block.

To measure grout flow, two in-line high-pressure flow meters were used, one a Jean Lutz-LT3n, C16M-B74, SP100MC21 pressure/volumetric flow device that measures the number of strokes of the positive displacement pump, and the other an in-line Halliburton turbine meter (Figures 11–12 show these devices).

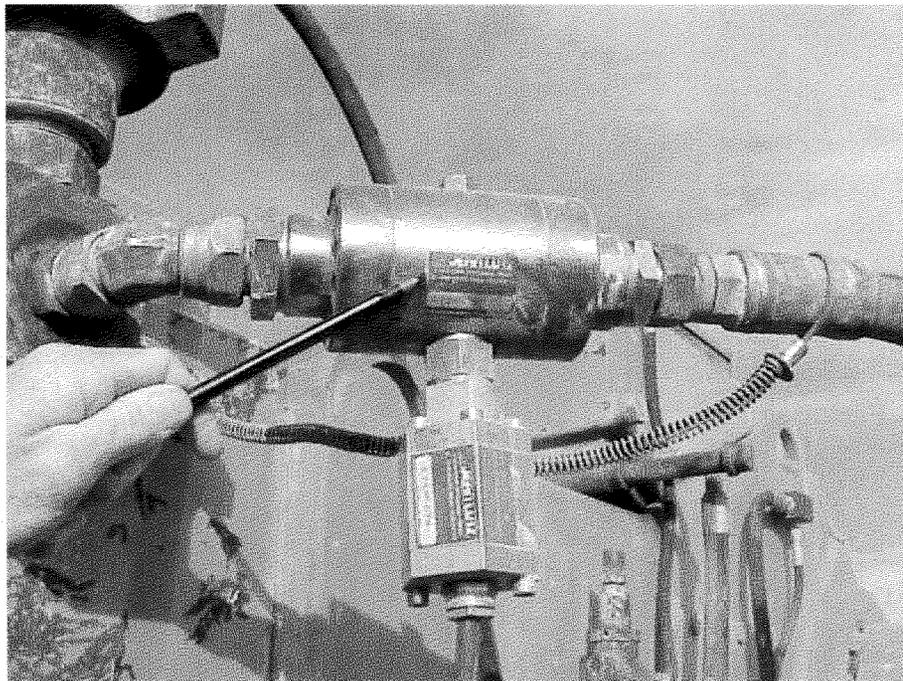


Figure 11. Jean Lutz pressure/flow meter.



Figure 12. Halliburton in-line flow meter.

When comparing data from the two flow measuring devices the integrated volumetric flow agreed with each other within 14%. In all cases, the Jean Lutz system measured about 14% lower than the Halliburton system. It is noted that the Jean Lutz system was purchased specifically for this test and was calibrated prior to testing by a factory representative and therefore is thought to be the most accurate. The Halliburton meter was used in previous INEEL testing (Loomis 1996) and the measured volume of grout that went into a pit was high relative to the amount of void volume in the pit. It was speculated in that work that the discrepancy was a water calibrated volumetric flow device erroneously measured the grout flow too high. However, the fact that the two systems agreed for a variety of grout types suggests that the Halliburton system was calibrated correctly in the past study in that for the present work it also was calibrated using water only.

4.3.2 Procedures

The implementability testing involved the following steps:

Construction of Site/Initial Testing

Initial preparation tasks included construction of a test area similar to disturbed soil conditions expected at the SDA. A pit 6.6 m (21 ft) long was excavated 1.2 m (4 ft) wide by 3.3 m (11 ft) deep and backfilled with equivalent INEEL-RWMC-type silty-clay soil obtained from a site near Benton Washington. Representativeness was verified by comparative soil composition tests. The backfilling was a loose pack without machine packing—the intent being to create a site with 30-50% by volume free voids. Next, three specially prepared mock-up thrust blocks were arranged over the pit as shown in Figure 13.

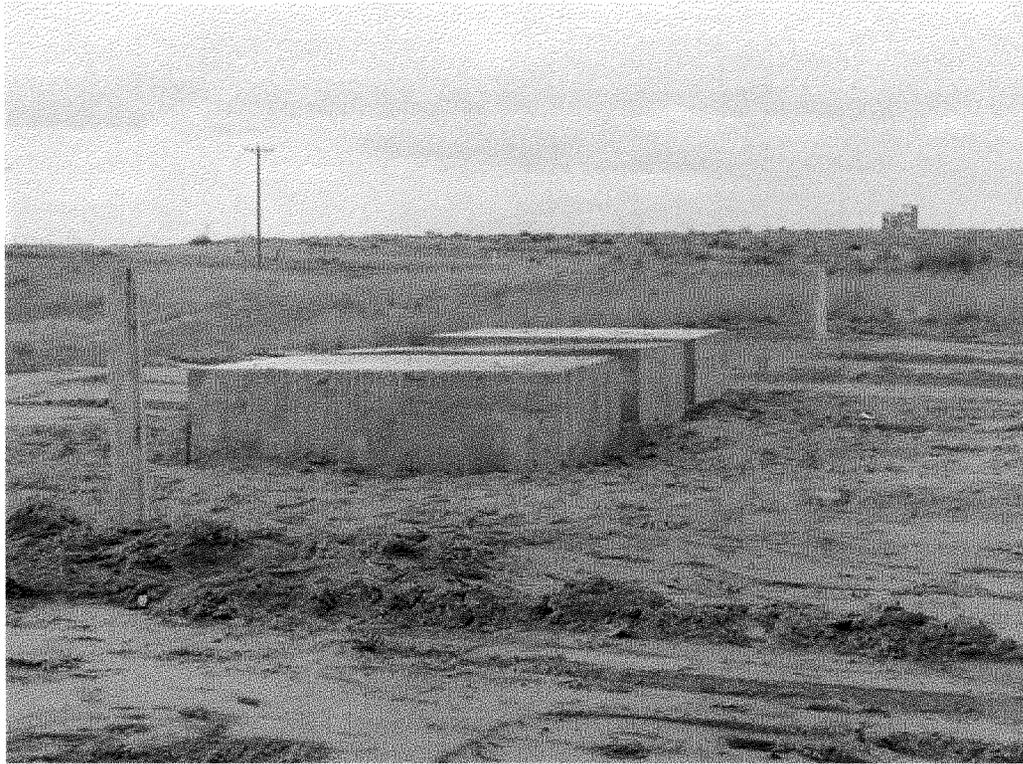


Figure 13. Thrust blocks in place on top of pit (without side berming).

The thrust block with three holes on a 50 cm (20-in) triangular pitch was designed to catch excess grout returns to the surface during grouting. The thrust blocks were lined with Styrofoam to prevent grout returns from sticking to the surfaces of the thrust block and the access holes for grouting were lined with brushes to provide wiping of the drill steel during withdrawal of the drill stem. The void space under the thrust block was 0.45 m^3 (16 cu. ft) which allows 449 L (119 gal) of returns for each block. Prior to grouting the three grouts, a nozzle optimization study was performed for those grouts that had never been field grouted before: GMENT-12 and U.S. Grout Premium Grade. In these nozzle studies each grout was tested with a 2.4-mm and a 3-mm nozzle by jet grouting 1.2 m (4 ft) high columns in a specially prepared RWMC-INEEL type soil region. The columns were allowed to cure overnight and then a trench was created adjacent to the columns and the columns were exposed, examined and photographed. Eventually, the columns were removed intact and broken in two pieces using a standard backhoe. The nozzle size that allowed jet grouting at 400 bar (6,000 psi) to create the largest column was chosen for the implementability testing involving the thrust blocks.

Implementability Grouting

- Samples of each grout batch were collected directly from the mixer before grouting was initiated and tested for density using an industry standard mud balance.
- The jet-grouting rig was positioned for grouting of the field trials. Basic procedures established during the Acid Pit Stabilization Treatability Study (Loomis et al. 1999) were followed. Grouting was performed with the following parameters: two revolutions per step, a step distance of 5 cm (1.97 in.) per step and a step rate dependent on the results of either the special a nozzle test or

based on an attempt to place the identical amount of grout in each triplex of columns. Injection pressure was nominally 400 bar (6,000 psi). The basic injection process was as follows:

- Position jet-grouting apparatus drill string over a hole in a thrust block
- Drill to 3.6 m (11.91 ft) below the top surface of the thrust block (includes the thickness of the thrust block-15 cm (6 in.), space under the thrust block –30 cm (12 in.), 73 cm (29 in.) of overburden, and 2.4 m (8 ft) of grout column)
- Commence high-pressure injection and retract rotating drill stem 8 ft.
- Discontinue high-pressure pumping
- Raise drill stem (allow grout to drain)
- Move to next hole and repeat the procedure.
- Place the thermocouple assembly down one hole in each thrust block following grouting.
- Place the 7 cm (2.75 ft) outside diameter by 5.1 m (17 ft) long polyethylene rod in one hole of the nine holes (At random, it was determined during testing to use the TECT HG test area.).

One three-column monolith was attempted for each grout. Figure 14 shows the layout of the thrust block with basic dimensions for the various features. Three grout types recommended from the bench testing made for a total of nine grout holes. Qualitative and quantitative data gathered during implementability testing included:

Qualitative Data

- Filter caking properties of the material
- Mixing problems such as excessive air entrainment, suspended solids, and material separation
- Equipment fouling and residual buildup inside pumping equipment
- Cracking of soil and heave outside of the thrust block
- Incomplete curing of grout columns
- Qualitative size distribution of soil inclusions in the columns
- Photographic record of the column excavation
- Relative ease of cleanout
- Sticking of grout returns to thrust block
- Durability of brushes on holes
- Other unusual operation occurrences.
- Pressure required to remove the 7 cm (2.75-in.) polyethylene rod placed in a grout hole for one of the grout holes (the rods are to be used during field testing to create holes for hydraulic conductivity testing during the field tests).

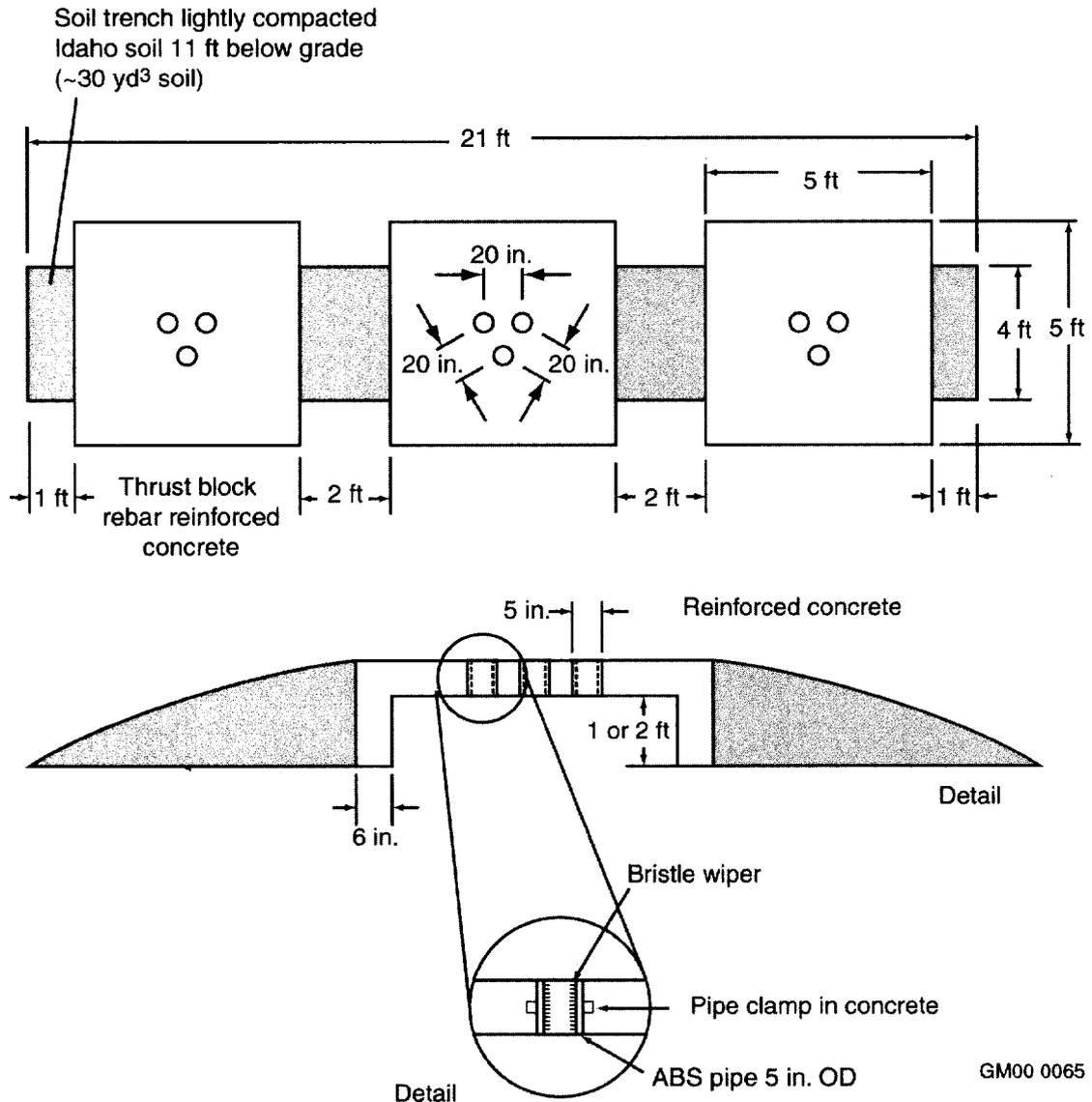


Figure 14. Implementability test layout.

Quantitative Data

- **Grout Returns.** Grout returns were measured by pumping a slurry mixture of bentonite and water into the thrust block void and subtracting the amount of water pumped from the measured inside volume of the thrust block. When the thrust blocks were removed, the amount of grout returns under the thrust block was also observed and compared to the water pumping method.
- **Curing Temperature Curve.** Temperature sensors were placed in one grouted hole for each grout type immediately after grouting operations were complete. A portable data logger was attached to the temperature sensors and used to measure and record the temperature of curing at intervals of approximately 20 minutes for the 5-day curing period.

- *Amount of Grout Injected and Injection Parameters.* The total amount of grout injected for each hole was recorded and the pressure, step size, rotation rate, and time on a step was also recorded.
- Column development measured to ± 1 inch using a tape measure.

Following a 1-day set, the thrust blocks were tested for volume remaining under the blocks using the bentonite/water slurry mix. Then, following a 5-day set, the monolith was exposed for examination. Following the cure time, the polyethylene rod was first removed using a standard backhoe and rigging then, the thrust blocks were removed. Following these preliminary tests, the grout columns were excavated using a combination of machine excavation and manual removal of surrounding soil. The first step after removing the thrust block and observing the amount of cured grout returns under the block was to remove the 73 cm (29 in.) of overburden material over the entire area of the columns. Next, directly in front of the row of columns, but not cutting into the columns, a separate trench 2.4 m (8 ft) below grade was cut. This trench was shaped for safe manned entry to further excavate the columns head on by hand. Once excavated, a combination of backhoes and laborers were used to cut surrounding soil away from the columns using hand held picks, crowbars, and shovels. Figure 15 shows a side view of the excavation method. A photographic record was kept and the physical condition of the columns was described. Once excavated and photographed, the columns were pulled down in one piece and isolated for further photographs and evaluation.

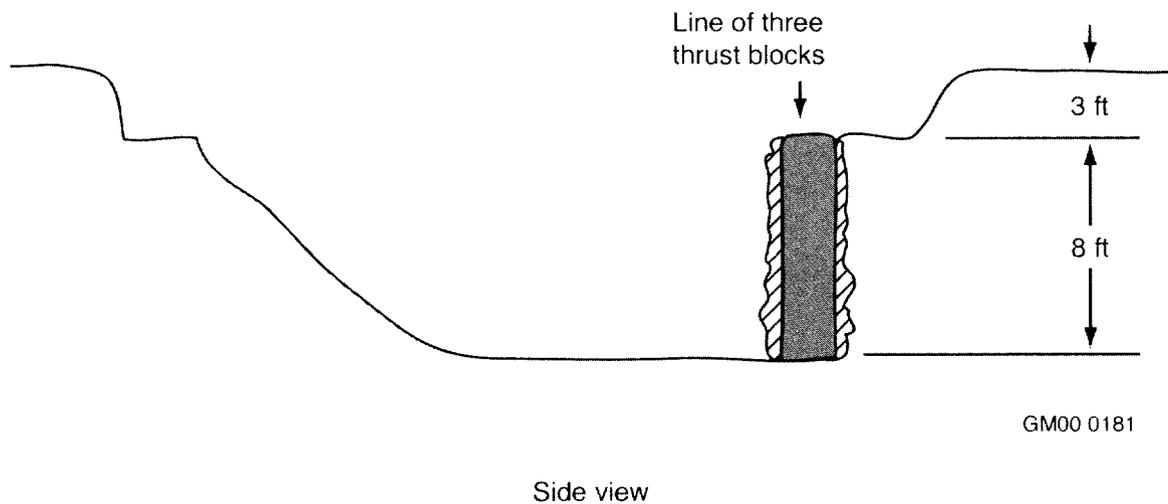


Figure 15. Side view of excavation of implementability tests.

One grout material was selected from among the three grouts used in this study for the field-testing. The grouts were evaluated using engineering judgment using the following criteria as a guide:

- Pumpable with minimal operational problems
- Ease of Mixing
- Optimal column development (a combination of maximum column diameter with low percentage of soil inclusions)
- Minimal grout return
- Cost and availability of grout

4.4 RESULTS OF IMPLEMENTABILITY TESTING

The results include (a) data from a special nozzle test to determine the optimum nozzle to allow jet grouting for those grouts that have never been jet grouted before, (b) grouting results when creating an interconnected three hole monolith under the thrust block for the three grouts, (c) results of curing temperature of the monoliths, (d) results related to placement and removal of the 7 cm (2.75-in.) polyethylene rods to be used as hydraulic conductivity holes, (e) results of quantitative determination of the volume of grout returns, (f) qualitative description and photographic record of the resultant monolith and finally, a discussion of results section in which the three grouts are compared and contrasted and a final single grout is recommended for field testing.

4.4.1 Special Nozzle Testing for U.S. Grout and GMENT-12

Prior to conducting the grouting tests with the thrust block, special nozzle optimization tests were performed to determine an appropriate nozzle for the two new grouts (GMENT-12 and U.S. Grout). The TECT HG had been grouted in prior studies (Loomis 1996, 1998) and a 3-mm nozzle was recommended. The nozzle tests involved creating (in INEEL-like soils) 4 ft high columns at a depth of 3 ft below grade. An attempt was first made to jet grout GMENT-12 and U.S. Grout with the 3-mm nozzles (two nozzles placed 180 degrees apart on the drill stem), and it was found that the system could not be pressurized with the high-pressure pump higher than 200 bar (3,000 psi) for either grout.

4.4.1.1 GMENT-12/U.S. Grout

For GMENT-12, use of a 2.4-mm nozzle resulted in achieving the desired pressure of 400 bar (6,000 psi), and a column was created with no grout returns. Based on preliminary calculations, the injection parameters included 5.1 s/step with a 5 cm step and 2 revolutions per step. Upon excavation following a 3-day cure, a cohesive column was revealed and removed in one cohesive piece using a standard backhoe. The measured dimensions of the column were 109 cm (43 in.) long and approximately cylindrical shaped, with an outside diameter averaging 63.5 cm (25 in.). With great difficulty, the column was broken in two pieces by dropping the bucket from a height of 0.61 m (2 ft) over 10 times. The column appeared to consist of mostly pure grout well mixed with soil, with some visible soil inclusions as shown in Figure 16. Some 0.5-cm diameter voids were found in the monolith (also shown in Figure 16). These pours were most likely from void redistribution in the soil and/or entrained air during grouting.



Figure 16. GMENT-12 mixtures of soil and grout.

A total of 208 L (55 gal) of grout were used to make the column at a step time of 5.1s which gives an injection rate of 1.89 L/cm (15.3 gal/ft). The volume of the column represented about 91 gal and the injected grout was 208 L (55 gal) with no returns resulting in 60% grout void filling which means the INEEL like soils were very dry (less than 10 wt% moisture) or the soils were only slightly compacted. This compares to estimates of a total void of 60% for the buried waste in the SDA.

A similar evaluation was made for the U.S. Grout material in that the 3-mm nozzle also could not support the 400 bar (6,000 psi) grouting pressure thus the 2.4-mm nozzle was recommended. The dimension of the resultant column was similar to that created for the GMENT-12 and was 61 cm (24 in.) in diameter and 1.2 m (4 ft) high.

4.4.2 Creating a Triplex Column Using the Thrust Block

A series of three connected holes were grouted in a 50 cm (20 in.) triangular pitch for each of the three grouts. For each of the cases, the grout was prepared in a vortex mixing system that allowed mixing at least enough grout for a single column such that the grouting was essentially performed as one operation with no curing of the grout between grout holes. Grouting was performed using a thrust block to collect any grout returns. The first grout injected was the GMENT-12 followed by the U.S. Grout and finally the TECT HG. Table 24 summarizes the volume of grout injected in forming the grout columns. What follows is a description of the grouting operation and data obtained during grouting.

Table 24. Volume (gal) of grout injected during the implementability tests (3.78 L/gal).

Grout Type	Hole number	Grout Flow (Jean Lutz Flow Meter)	Total Grout Injected
GMMENT-12	1	93	360
	2	139	
	3	128	
U.S. Grout	1	156	260
	2	104	
TECT HG	1	90	331
	2	158	
	3	83	

4.4.2.1 GMENT-12

A triplex column (completely interconnected set of three individually emplaced columns) was successfully created using the GMENT-12 grout. The grouting parameters were based on the initial nozzle tests and were set at 5.1s/step, 2 revolutions per step and a 5 cm step size with grouting pressure at 400 bar (6,000 psi) for all three positions grouted. Three gallons of water were mixed with each 27.2 kg (60 lb_m) bag of dry ingredients. During grouting, no grout returns to the surface of the thrust block were observed; however, when grouting the final hole 11.3-18.9 L (3-5 gal) of grout returns flowed from the side of the thrust block as the final inches of the third hole were grouted. Table 24 summarizes the volume of grout injected in forming the columns using the Jean Lutz metering system and shows that a total of 1,360 L (360 gal) of grout were injected with no returns to the surface of the thrust block (i.e., no overflow). This equated to about 1.8 L/cm (15 gal/ft) delivered to each hole during grouting.

4.4.2.2 U.S. Grout

Only two holes of the planned triplex column were grouted because excessive grout returns to the surface of the thrust block during the grouting of the second hole precluded grouting any further. The parameters for grouting the U.S. Grout were 5.5 s per step, 2.4-mm nozzle size, 5 cm step size, and 400 bar (6,000 psi) pressure at the high-pressure pump. A mud balance measurement was taken on the grout at 1.57 kg/L (13.1 lb_m/gal), which agrees with the density measurements in the bench study. The U.S. Grout was found to be easy to mix. There was a reduction in lumps of solid material that had to be mixed relative to mixing the GMENT-12 grout and about 90 L (24 gal) of water were used in mixing. With the U.S. Grout there was an initial plugging of a nozzle. This plugging was not attributed to the grout rather, it was the first grout hole of the day and debris in the lines could have accounted for the plugging. A total of 982 L (260 gal) of grout were delivered in the two holes during the grouting operation which essentially filled the thrust block with returns. In fact, the first hole had 589 L (156 gal) of grout in the 2.4 m (8 ft) column grouted and in the second hole only 393 L (104 gal) was injected [the column was between 1.2–1.5 m (4-5 ft) deep] when grouting was suspended because of the excessive return. Because of this complete filling of the thrust block with grout returns, it was decided to not grout the third hole. Further grouting was impossible because grout returns had no void under the thrust block to fill therefore, continued grouting would have caused massive spillage out the top the thrust block. The relatively low density U.S. Grout simply did not impart enough kinetic energy to the soil and simply could not overcome the resistance of the soil and copious grout product came to the surface as a grout return.

4.4.2.3 TECT HG

TECT HG was successfully injected in a total of 5 positions (two limited length-field trials and 3 holes using the thrust block). Initially, it was desired to inject a similar volume of grout for the TECT HG as the GMENT-12 so that a valid comparison could be made relative to column development in conditions of similar soil voids. To set these desired parameters, and to maximize the availability of grout material, the field trails only involved two-foot high columns in an iterative approach. These field trials were also necessary because the TECT HG grout is of higher density than the GMENT grout (specific gravity of 2.16 g/cc for TECT HG and 1.6g/cc for GMENT-12). The 3-mm nozzle was used and the mud balance reading on the TECT HG grout was 17.5 lb_m/gal [2.1 g/cc] again in agreement with the bench studies. In addition, the initial field trial was at 400 bar (6,000 psi), 5 cm/step and 5.0 s/step. In 0.35 m (1.15 ft) of column, 93 L (24.8 gal) of grout were injected or 2.6 L/cm (21.56 gal/ft), which was much higher than the average of 1.8 L/cm (15 gal/ft) injected in the GMENT-12 triplex columns. In the second field trial the time on a step was adjusted to 3.5 s/step and the result was 65 L (17.2 gal) for 0.35 m (1.15 ft) column or about 1.8 L/cm (15 gal/ft). Therefore, the first 2.4 m (8 ft) column of the triplex under the thrust block was grouted at 3.5 s/step with the result that 340 L (90 gal) of grout was injected for 1.38 L/cm (11.2 gal/ft) which was short of the goal of 1.8 L/cm (15 gal/ft). Therefore, using proportioning, the next hole was grouted using 4.5s/step with the result of 597 L (158 gal) of grout injected or 2.44 L/cm (19.7 gal/ft). This action overshot the desired 1.8 L/cm (15 gal/ft) so the last hole was grouted at 4s/step with a total of 317 L (83.75 gal) in a 1.7 m (5.7 ft) hole [the system ran out of grout before the total length of 2.4 m (8 ft) could be grouted] which resulted in 1.8 L (14.7 gal/ft), which was close to the desired 1.8 L/cm (15 gal/ft) average. Overall, in the three holes the total amount of grout injected was within 10% of that injected into the GMENT-12 pit. During grouting of these three holes under the thrust block, there were copious returns of grout pouring out the field trial holes that were located just off the side of the thrust block indicating communication between holes. Following grouting, a 7 cm (2.75 in.) polyethylene rod was easily hand inserted into one of the grout holes to evaluate this technique of creating a test penetration for hydraulic conductivity measurements during the field testing.

Possible explanations for this unpredictable behavior in grout delivery include (a) a random variation in step time or (b) millisecond periods in which the back pressure offered by the waste caused a millisecond reduction in flow not seen by the pressure/flow measurement systems. The step time was measured repeatedly during the implementability testing and was found to be fairly easy to set. However, it is possible when ranging out 2.4 m (8 ft) of hole there are approximately 50 steps and the step time could actually slip as it goes through the 50 steps. As far as the loss-of-pressure theory, it is unlikely that the response of the Jean Lutz would not pickup the millisecond oscillation in flow. Specifically, the pressure should have shown an oscillatory nature to account for each pressure variation.

The problem of predicting grout flow based on a given set of parameters from one hole to another was also evident in past grouting campaigns (Loomis 1997). The main parameters that is varied is the dwell time or time in a step. Table 25 summarizes the data discussed above with a common 400 bar (6,000 psi) pressure, 2 revolutions per step and a 5 cm step size:

Table 25. Gallons of grout per feet at various dwell times.

Dwell Time(s)	gal/ft (multiply by 0.12 to get L/cm)
5	21.56 gal/ft
3.5s	15 gal/ft
3.5s	11.2 gal/ft
4.5s	19.7 gal/ft
4 s	14.7 gal/ft

Examining the table at 4s dwell time 1.8 L/cm (14.7 gal/ft) were delivered which is the same as one of the 3.5s dwell time cases. In addition, for different 3.5s dwell times, there was a variation in grout delivered from 1.3 L/cm (11.2 gal/ft) to 1.8 L/cm (15 gal/ft). What is needed to resolve this issue is to perform separate effects flow tests in which grout is injected in air while measuring the grout flow for various dwell times. If this test shows consistent results with increasing integrated grout flow after increasing dwell time, then the variable-pressure idea has some merit. If the back pressure idea has merit, then during operations, the total delivered grout delivery rate for various regions of the pit will have to continuously be adjusted by varying the dwell time repeatedly.

4.4.3 Observation of Halliburton Reading Versus Jean Lutz Reading

During past testing performed in simulated waste (Loomis 97), the Halliburton flow meter was used to measure the volumetric flow rate of grout during grouting of Type-H cement, TECT, and molten paraffin. For the pits grouted with TECT and to a certain extent, the Type-H cement, the amount of grout injected did not match the estimated void volume of 60-70% voids. Both pits were injected with an amount of grout far in excess of these available voids. For the TECT pit, the amount of grout injected was 4,222 L (1,117 gal) in only 11 out of a total possible 18 holes. The total volume of the pit was only 6,104 L (1,615 gal) which means the amount of grout injected was 70% of the total pit volume. On a basis of amount injected per the amount of surface area covered (11/18 of the total surface area), the amount injected is 113% of the volume of that part of the pit. It is also noted that upon excavation, the total pit did not contain grout so the argument that the grout simply flowed to voids in other ungrouted parts of the pit is not valid. For the pit grouted with Type-H cement, a total of 5,428 L (1,436 gal) were injected into 18 holes covering the same volume as the TECT pit [6,104 L (1,615 gal pit)] which accounts for an 88% void filling in yet the amount of voids was expected to be only 60-70%. Because of these data, it was

suspected at the time that the Halliburton flow meter was for some unknown reason artificially reading high by as much as 20-30%.

With this background, the implementability testing used both the Jean Lutz and Halliburton flow meters in an attempt to reconcile the differences seen in the 1997 work for all types of grouts. Table 26 below summarizes the data.

Examining the data in Table 26 the average Halliburton/Jean Lutz ratio for GMENT-12 = 1.12, U.S. Grout = 1.13, and TECT HG = 1.17. If the Jean Lutz is considered the standard then the Halliburton measures grout flow approximately 14% high. This argument is in agreement with the data obtained in 1997 in the pit grouted with type-H cement in which it was concluded that 88% of the total excavated pit volume was grouted in yet the expected void volume was 60-70%. No conclusions can be made relative to the data taken relative to the pit grouted with TECT in 1997 because only a portion (11/18th) of the pit was grouted. Therefore as a backup measurement technique, under high pressure conditions, it is concluded that the Halliburton system consistently gives a 14% high reading for a variety of grout types. This is exactly why the Jean Lutz system was chosen in that it measures strokes of the pump independent of fluid type.

Table 26. Volumetric flow of grout during the implementability testing (3.78 L/gal).

Grout Type	Halliburton Reading (total gal)	Jean Lutz Reading (total gal)
GMMENT-12	105	93
	157	139
	144	128
U.S. Grout	176	156
	120	104
TECT HG	106	90
	186	158
	98	83

4.4.4 Special Drill Steel Drain Test

During grouting with the TECT HG grout the high-pressure pump was turned off, and the drill steel was raised to observe how long it took to drain the drill steel of grout via the nozzles. For several holes it took as low as 1min and as much as 5min to drain the grout material in the drill stem. The elapsed time of 5 min was recommended for the length of time to leave the sub assembly under the thrust block prior to bringing it above the thrust block during the field test. This action would ensure that the double bag around the sub-nozzle assembly (as a contamination control strategy) would not fill during the field testing and effect the operation of the drill string shroud assembly.

4.4.5 Ease of Mixing of Grout and Clean-out

Following grouting of the three types of grout, the grouting contractor was interviewed as to ease of use of the grouts to determine which of the three grouts (TECT HG, GMENT-12, U.S. Grout) was the easiest to mix and, following grouting, to clean-out. During grouting, the down time to clean out plugged nozzles was basically the same for all grouts. In only one case did a nozzle plug-just before the field trial for the TECT HG holes. This plugging was due to debris in the grout not an inherent problem with the TECT HG grout. The grouting contractor claimed that U.S. Grout mixes easier than GMENT-12 and TECT HG is the hardest to mix because of the required liquid component. In addition, GMENT-12 is rated medium difficult to mix with minor clods that have to be broken-up during mixing. GMENT-12 with an

all dry product is easier than TECT HG. When mixing U.S. Grout, superplasticizer must be put in very soon to allow mixing.

During clean-out the grouting contractor evaluated (on a scale of 1-10 with 10 the most difficult) the degree of difficulty for the clean out process. The results are listed below:

- TECT HG is hardest by far (some filter caking)-8
- GMENT-12 is second hardest with no filter caking-4
- U.S. Grout displayed some filter caking-4

These qualitative observations from the grouting contractor apply to using the small batch vortex mixer; however, even in a large batch plant mode the comments are still valid (approximately 1 cubic yard).

In summary, U.S. Grout is easiest to mix followed by GMENT-12 and the hardest was TECT HG. TECT HG is by far the most difficult to clean out with GMENT-12 and U.S. Grout similar but fairly easy to clean out.

4.4.6 Grout Returns Under Thrust Block

The amount of grout returns under the thrust block were measured by filling the remaining void in the thrust block with fluid and subtracting that value from the calculated volume of the total void under the thrust block. The lower the measured return, the less likely that contaminated grout returns will escape the thrust block during a hot application. A special mixture of bentonite (Wyoming Bentonite-Billings Montana) and water was prepared in the first vortex mixing system. The idea was to use a bentonite slurry to eliminate errors in measuring the volume under the block in that the slurry would seal any leakage paths under the block. A special test block was created in the local sand and the bentonite slurry mix effectively sealed the sand from slurry flow using a ratio of about 5.4 kg (12 lb_m) bentonite per 151 L (40 gal) of water. Using the flow meter on the vortex mixer, the slurry was pumped under low pressure into each of the thrust blocks. Because all of the holes in the thrust block used for U.S. Grout were sealed with grout returns (indicating a completely full thrust block) it was impossible to fill the U.S. Grout block. For the TECT HG thrust block a total of 94.5 L (25 gal) of slurry was placed in the thrust block and for; the GMENT-12 thrust block a total of 267 L (70.8 gal) of slurry was placed. Therefore, for the TECT HG grout, there were a total of 449 L (119 gal) minus 94 L (25 gal) or 355 L (94 gal) of grout returns and for GMENT-12, a total of 449 L (119 gal) minus 267 L (70.8 gal) or 181 L (48 gal) of returns.

In summary, U.S. Grout had the most grout returns at 449 L (119 gal) of grout returns even with only 2 holes grouted with a total of 982 L (260 gal) injected meaning 45% of what was injected came up to the surface. Next , TECT HG had the second most grout returns at 355 L (94 gal) with a total of 1,251 L (331 gal) injected meaning a 28% volumetric return, and finally, GMENT-12 had the least grout returns at 181 L (48 gal) with 1360 L (360 gal) injected for a 13% return. Comparing the amount of grout returns to the amount of grout injected in each of the blocks shows that GMENT-12 clearly had the best results of the three grouts tested as shown in Table 27.

Table 27. Comparison of grout take and grout returns (3.78 L/gal).

Grout Type	Grout Take (gal)/#holes	Grout Returns
TECT HG	332/3	94 plus 30 gal blowout = 124
GMMENT-12	361/3	48
U.S. Grout	260/2	119

Since only two grouted holes filled the thrust block for the U.S. Grout for a loose soil condition, it is anticipated that when grouting the soil surrounding the pit during the field test that the U.S. Grout would produce too much return to allow a safe operation.

4.4.7 Curing Temperature Results

The center line temperature of the triplex column was measured with thermocouples during curing to ensure that temperatures remained below 100°C. Above 100°C steam would be created and potentially entrain contaminants, which is undesirable. Figures 17, 18, and 19 show the curing temperature profile graphically for U.S. Grout, TECT HG, and GMENT-12, respectively. Tables in Appendix J summarize the time/temperature profile at mid-axial location for TECT HG, GMENT-12, and U.S. Grout. The thermocouples were inserted immediately following injection of each type of grout.

Examining the figures, the U.S. Grout reached a maximum centerline temperature of set of 48°C (118°F), 12 hours (720 min.) following insertion of the thermocouple and GMENT-12 reached a maximum temperature of set of 75.5°C (168°F), 14.3 hours (858 min) after insertion, and finally, TECT HG reached a maximum temperature of set of 74°C (165°F), 17.6 hours (1056 min) after insertion of the thermocouple. Therefore, all three grouts met the curing criteria in that the maximum temperature of set is below 212°F or 100°C.

4.4.8 Polyethylene Rod Removal Results

During the field test, a series of eight boreholes were required to perform hydraulic conductivity testing. To create these boreholes, a 7-cm (2.5-in.) polyethylene rod was inserted into a just-grouted hole. Following curing, the borehole was to be created by removing the polyethylene rod. A 7-cm (2.75-in.) polyethylene solid rod was inserted into one hole of the TECT HG triplex column. The polyethylene rod had a solid drive point attached to a metal rod that extended up through the center of the polyethylene rod. At the top of the metal rod was an “eye” lifting attachment.

The insertion was easily accomplished by manually pushing down the rod to full depth, which was 12 ft from the top of the thrust block (30 cm [1 ft] of thrust block, 0.9 m [3 ft] of overburden, and 2.4 m [8 ft] of grouted material). Allowing for 5 days of curing, the polyethylene rod was removed with some difficulty. The first attempt to remove the rod involved attaching the lifting ring located on the top of the polyethylene rod to a lifting strap attached to a backhoe bucket. The rod could not be removed with the standard backhoe in an extended position with the existing hydraulics. Next, the lifting strap was located in a chock-hold type arrangement using a pipe wrench for purchase on the smooth surface of the rod as shown in Figure 20.

This action allowed removal of the rod in about 60-cm (2-ft) lifts, with each lift demanding a change in position of the pipe wrench/strap combination. It is recommended that, during the field testing, a larger track-hoe be used to lift the polyethylene rod out using the designed lifting eye. If that fails, use the chocker method described above.

4.4.9 Destructive Examination of Resultant Columns

The destructive examination of the columns created under the thrust blocks first involved removing side burden material until the three monoliths were exposed as shown in Figure 21.

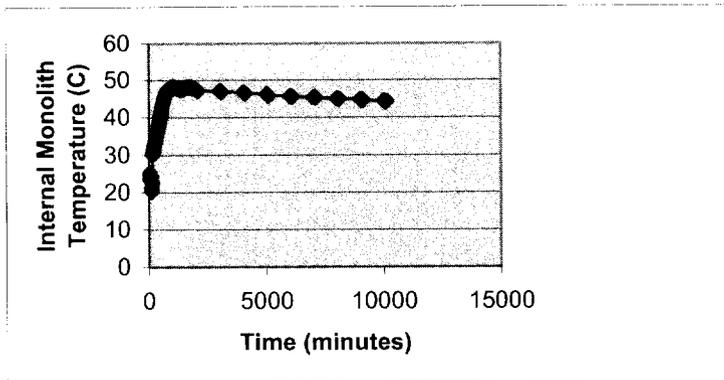


Figure 17. Curing temperature profile of U.S. Grout.

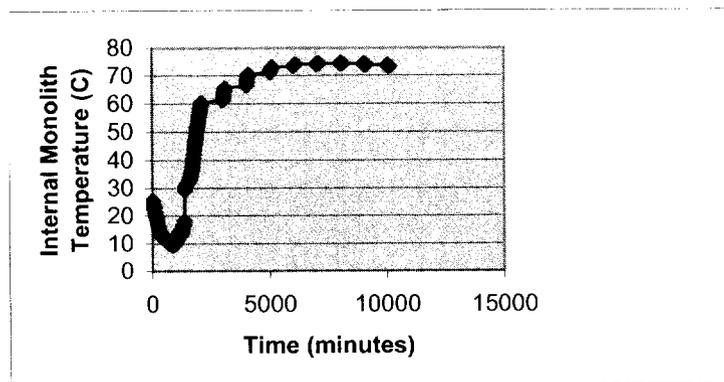


Figure 18. Curing temperature profile of TECT HG.

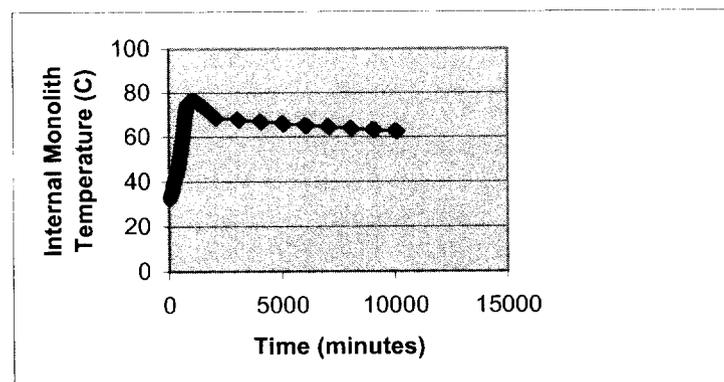


Figure 19. Curing temperature profile of GMENT-12.

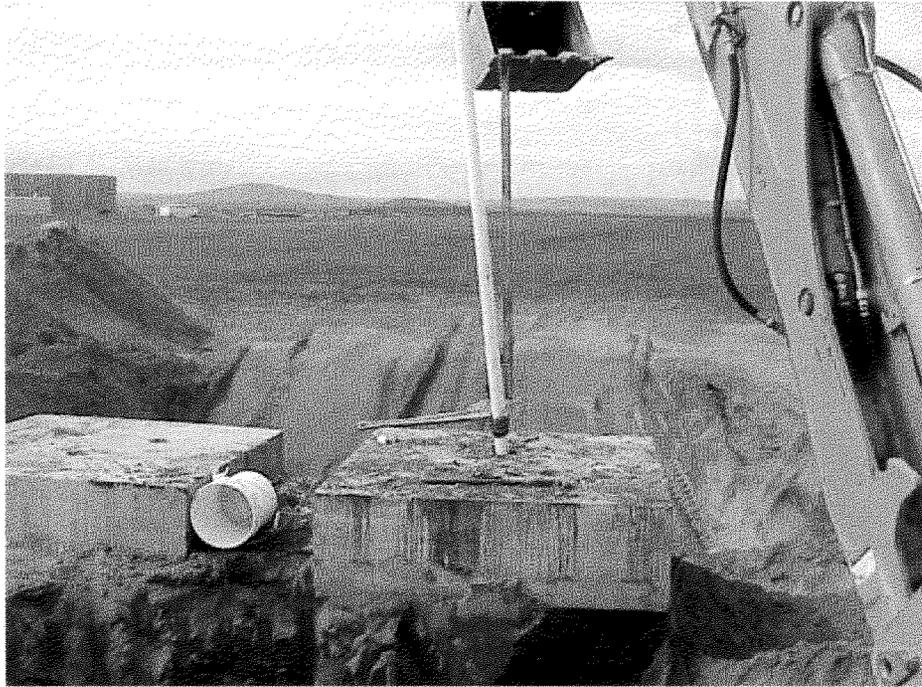


Figure 20. Lifting the polyethylene rod out of a grouted hole.



Figure 21. Excavation of the monoliths.

The thrust blocks were then toppled over into the created pit and the interior of the thrust block examined for sticking of the cured returned grout/soil on the interior surface of the thrust block (the interior surface of the thrust block was lined with closed cell foam liner). This was followed by complete excavation of the monoliths and removal in one piece for a photographic record.

4.4.9.1 Removal of the Thrust Block

Thrust block undersurfaces had been coated with foam to eliminate sticking of grout return and to allow possible reuse of the thrust block. Following grouting, the thrust blocks were examined for sticking of the grout. All thrust blocks were removed, and, as expected, the U.S. Grout thrust block was completely full of cured grout. The grout stuck to the surface of the foam and could not be removed, even after lifting and dropping the thrust block with a front-end loader. The TECT HG grout exhibited a similar effect in that it also showed the entire grout return stuck to the foam liner.

For the GMENT-12 grout, however, the entire return came out in one piece, leaving the thrust block empty during the process of rolling the thrust block off the top of the monolith. Figure 22 shows the underside of the GMENT-12 thrust block, with the solid block of cured grout returns basically fallen out of the block during excavation.

This compares to the U.S. Grout thrust block in which the entire grout return not only is stuck to the Styrofoam but the block is confirmed full of cured grout (Figure 23).

It is concluded that generally the blocks are not reusable and that the GMENT-12 grout exhibited the best tendency for not sticking to the Styrofoam. Reuse of the blocks would be a potential for the GMENT-12 grout only.

4.4.9.2 Examination of the GMENT-12 Monolith

The GMENT-12 monolith was isolated on three sides, photographed and measured. The GMENT-12 monolith is on the left side of the photograph shown in Figure 21.

Using the front-end loader, the monolith was completely removed in one stand-alone piece of the mixtures of soil and grout. The monolith was measured at 2.4 m (8 ft) high by roughly 1.2 m (48 in.) in diameter. This represents a volume of 2,838 L (751 gal), and 1,360 L (360 gal) of grout was injected with 181 L (48 gal) of returns, or a net 1,179 L (312 gal) into the monolith. This equates to a void filling of 41%.

An attempt was made to break up the monolith using a standard backhoe bucket by raising the bucket above the monolith about 60 cm (2 ft) and striking the monolith. Only small fragments could be obtained after repeated blows, suggesting a strong cohesive monolith (Figure 24).

4.4.9.3 Examination of the TECT HG Monolith

The monolith created by the injection of TECT HG grout in a triplex column was a solid cohesive stand-alone monolith as shown being moved out of the pit in Figure 25.



Figure 22. GMENT-12 thrust block (underside showing Styrofoam liner).



Figure 23. Underside of U.S. Grout thrust block.

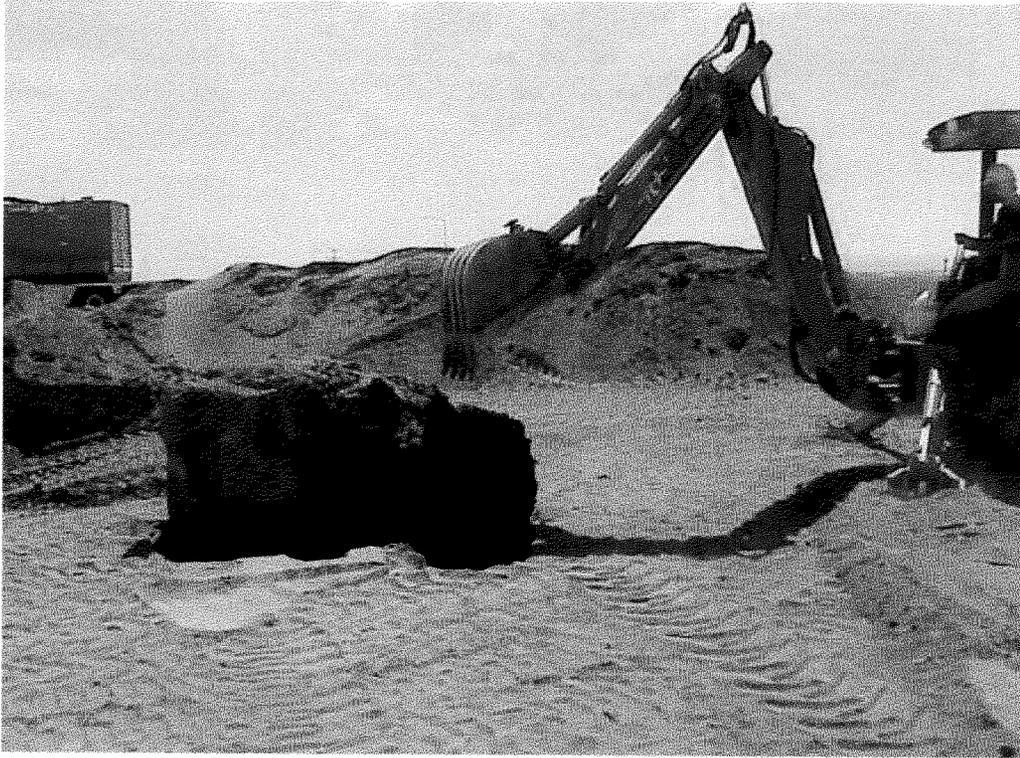


Figure 24. GMENT-12 monolith removed as one piece from the pit.

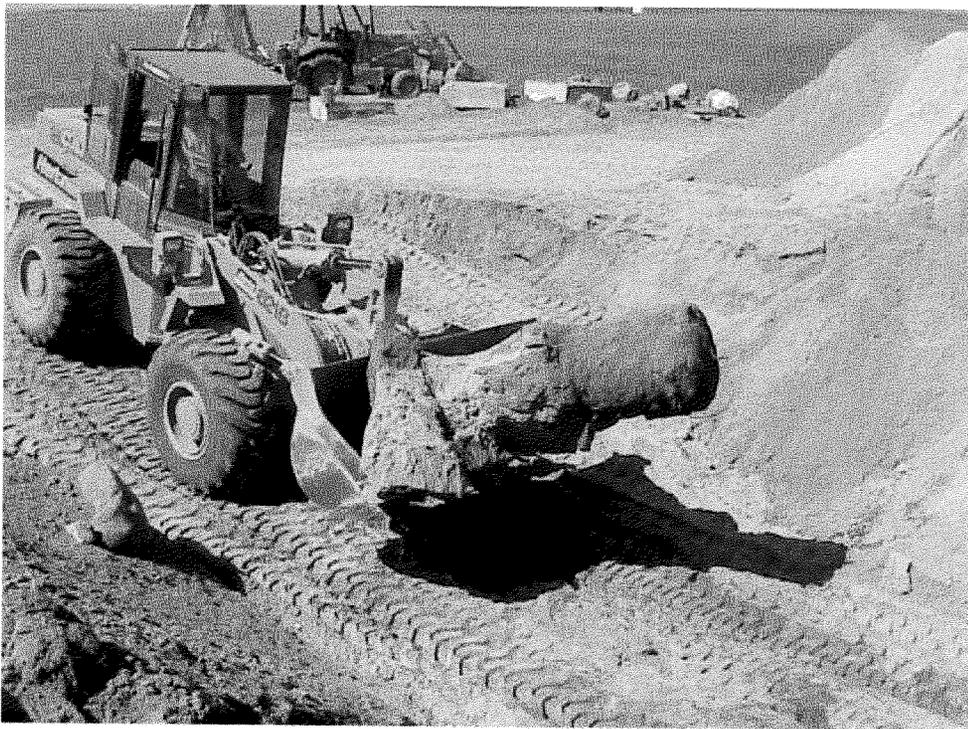


Figure 25. TECT HG monolith being moved as one piece.

The dimensions of the monolith averaged about 109 cm (43 in.) throughout the length of 2.38 m (7 ft 10 in.) with a reduced section or ledge in the top portion caused by running out of TECT HG grout during the grouting of the last hole. A total of 1251 L (331 gal) of grout were injected with 355 L (94 gal) of returns giving a net volume injected into the monolith of 895 L (237 gal). The size of this monolith equates to an approximate volume of 1950 L (516 gal) of column. Accounting for an approximately 0.6 m (2 ft) diameter by 0.6 m (2 ft) high reduction in the region not grouted due to running out of grout equates to a void filling of 45% which is in good agreement with the GMENT-12 created monolith. An attempt was also made to break up the stand-alone monolith with the backhoe and after repeated attack, only small chunks could be broken off again suggesting a solid cohesive monolith. The hole created by the polyethylene rod inserted into the TECT HG monolith produced a smooth hole for performing U.S. Bureau of Reclamation packer testing as shown in Figure 26.

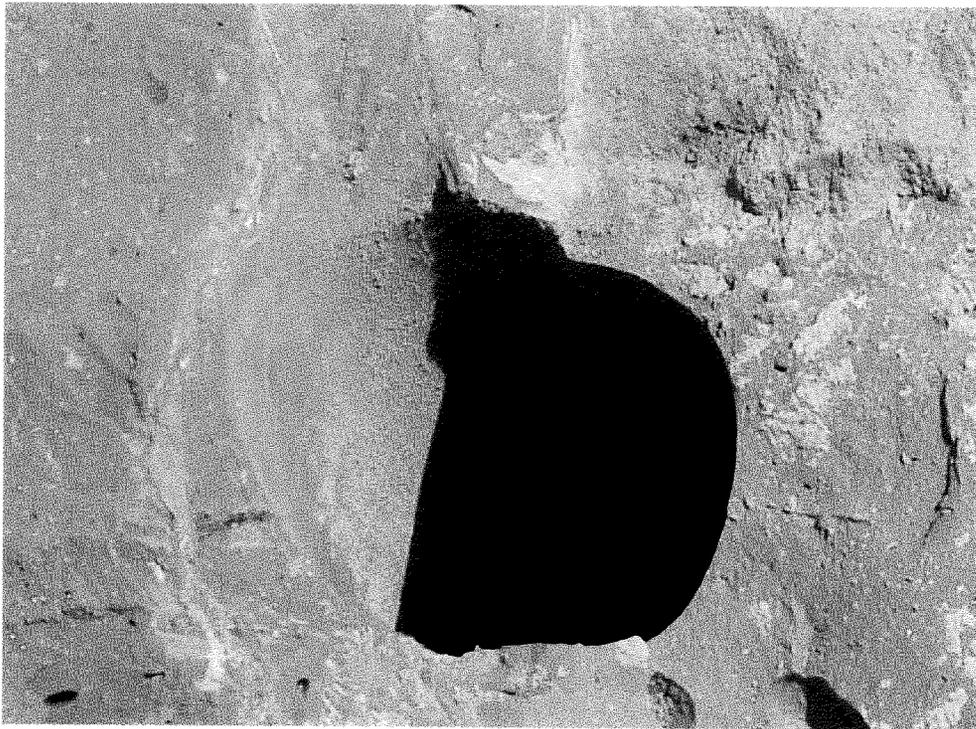


Figure 26. Hole created by insertion of the polyethylene rod.

4.4.9.4 Examination of the U.S. Grout Monolith

The U.S. Grout monolith also was a cohesive stand-alone piece of the mixtures of soil and grout as shown in Figure 21 on the right. The monolith was irregular shaped in that only 1 full column and 1 half column was grouted prior to filling the block with grout returns. The approximate volume of the column was that 1.2 m (4 ft) of the column had a mean diameter of 109 cm (43 in.) and the other half had a mean diameter of 84 cm (33 in.) which equates to an approximate volume of column of 1806 L (478 gal) of the mixtures of soil and grout. This compares to the amount of grout injected into the column of 532 L (141 gal) which means that the injection process filled 29% of the voids considerably lower than the void filling for the GMENT-12 and TECT HG grouts. It is possible that the relatively low specific gravity (U.S. Grout-1.6, TECT HG-2.16, GMENT-12-1.84) did not impart the same energy to the soil and therefore, the penetration of grout was lower.

4.4.10 Down-Selection of Grouts for Field Testing

Based on implementability testing of the three grouts, GMENT-12 was chosen as the single grout to carry forward for field testing. GMENT-12:

- Displayed the lowest grout returns/grout delivered ratio.
- Displayed the best ease of operation using simple dry ingredients and displayed a fairly straightforward cleanup.
- Was cost competitive with the other grouts.
- Produced a good monolith soil.
- Laboratory results for hydraulic conductivity, leach, physical strength, set conditions, where competitive.

5. FIELD TESTING

Field Testing was performed at the INEEL Cold Test Pit South which is located immediately south of the INEEL RWMC SDA. The testing was to involve a field preparation phase, a grouting phase, a hydraulic conductivity measurement phase and finally, a destructive examination of the resulting monolith. Due to a catastrophic failure of a fitting on the high-pressure pump and a resultant injury to a member of the subcontractor's team, only part of the grouting phase was performed. The project was not truncated due to safety issues, rather, there was a simultaneous compelling need for the remaining budget to cover the costs of other higher priority Environmental Restoration Projects. In addition, the cost of restart may have been prohibitive. Restart would have required new pressure relief systems and verification of operability, new procedures, and a rigorous operational readiness process. However, during this limited grouting operation, full contamination control data was obtained in enough detail to evaluate the contamination control features of the thrust block concept for jet grouting transuranic pits and trenches. What follows is a description of rest site construction, the testing hardware, mobilization processes, evaluation of the limited grouting operation, and finally, an evaluation of the contamination control system involving the thrust block and shroud systems.

5.1 Preparing for Grouting

5.1.1 Site Preparation

To perform the grouting demonstration, a pit simulating statistically average conditions in the INEEL SDA transuranic pits and trenches was constructed. The pit dimensions were 4.57 m (15 ft) by 4.57 m (15 ft) by 2.4 m (8 ft) deep. Waste in the pit was typical of that buried in the SDA including containerized cloth, paper, wood, asphalt, sludges, and metals. The backfill soil used in the demonstration represent exactly soil types, mineralogy, permeability, as seen in the SDA. Details of the pit construction and design rationale are documented in Shaw (2000).

5.1.2 Simulated Waste Preparation

A combination of SDA-wide waste volumes and specific details of Pit 6 at the SDA were used as a model for defining the simulated waste container volumes and waste material. Pit 6 was chosen as a model primarily because the average depth of buried waste is approximately 8 ft (2.4 m), simplifying the retrieval process for the treatability study. Table 28 shows the SDA-wide volume fraction of buried waste broken down into seven major categories. These include combustibles, organic sludge, inorganic sludge, nitrate sludge, metal, concrete, and asphalt. For example, on a volume basis, approximately 53% of the waste volume in the SDA comprises combustibles such as cloth, paper, plastic, and wood. Table 29 shows that the waste volume in Pit 6 approximately equals 50% of the excavated volume, of which 46% is drummed waste (55-gal [208-L] drums), 33% is boxed waste (wooden 4 × 4 × 8-ft [1.2 × 1.2 × 2.4-m] boxes), and 21% is cartoned waste (cardboard boxes of combustible material).

Applying the SDA waste loading rationale presented in Tables 28 and 29 to a 15 × 15 × 8-ft (4.5 × 4.5 × 2.4-m) deep test pit area, two 4 × 4 × 8-ft (1.2 × 1.2 × 2.4-m) boxes, 49 55-gal (208-L) drums, and 14 nominally 2 × 2 × 3-ft (0.6 × 0.6 × 0.9-m) polyethylene sacks were randomly configured in the test pit. Table 30 summarizes information relative to the type and contents of the simulated waste packages for the disposal pit. Metal debris including plate steel, tubing, and scrap metal was hand placed in two of the boxes along with concrete, asphalt, and wood. Boxes contain approximately 38% metal, 37% concrete and asphalt, and 25% wood as shown in Figure 27. Of the 49 drums, 25 contained combustibles that included cloth, paper, wood, and plastic, 13 contained inorganic sludge, six contained organic sludge, and five contained nitrate salts (shown in Figure 28). Three of the organic drums were metal sided drums, and

Table 28. Volume fractions of buried transuranic waste in SDA.

Waste Type	Volume (m ³)	Fraction of Total
Organic	3,696	0.059
Nitrate	2,480	0.043
Inorganic	7,361	0.124
Brick and concrete	7,570	0.117
Metal	7,445	0.121
Combustible	33,480	0.536
Total	62,032	1.000

Table 29. Pit 6 waste and soil volumes.

Total Excavated Volume	Soil Volume	Waste Volume
447,515 ft ³ (12,672 m ³)	223,617 ft ³ (6,332 m ³)	223,898 ft ³ (6,340 m ³)

Waste Type by Volume

Waste Type	Volume	Fraction of Total
Drums	102,272 ft ³ (2,896 m ³)	46%
Boxes	73,918 ft ³ (2,093 m ³)	33%
Cardboard	47,708 ft ³ (1,351 m ³)	21%

Table 30. Simulated waste packages for the disposal pit.

Waste Container Type	Number	Composition
Cardboard boxes (4 × 4 × 8 ft)	2	Metal debris (1/8-in. plate steel, tubing, piping, scrap metal), concrete/asphalt chunks (6-in. size), pulverized wood. Metal 38%, concrete/asphalt 37%, pulverized wood 25%.
Cardboard	25	Combustibles (cloth, paper, wood)
Cardboard	13	Inorganic (enough water to create a paste like consistency; 390 lb _m soil; 40 lb _m dry Portland cement; 36 lb _m NaNO ₃)
Cardboard	3	Organic (38 gal of Texaco Regal Oil; 65 lb _m Micro Cell-E;
Metal	3	35 lb _m kitty litter)
Cardboard	3	Nitrates (granular: 60 wt% NaNO ₃ ; 30 wt% KNO ₃ ; 5 wt%
Metal	2	Na ₂ SO ₄ ; 5 wt% NaCl
Sacks (2 × 2 × 3 ft) (polyethylene)	14	Cloth, paper.

two of the nitrate drums were metal sided drums. All other drums were cardboard drums to simulate the aging process that is expected to have occurred in the SDA transuranic sites and trenches. Fourteen sacks were filled with cloth and paper. A terbium oxide tracer was placed in each container (except nitrate drums) to simulate the mechanical movement of plutonium during operations. The combustible drums contained 3.5 oz (100 g) of tracer, the boxes 14 oz (400 g), the inorganic drums 7 oz (200 g), the organic drums 1.75 oz (50 g), and the sacks 3.5 oz (100 g). On a one-for-one basis it is estimated that this tracer loading represents maximum plutonium loading in the actual transuranic waste.

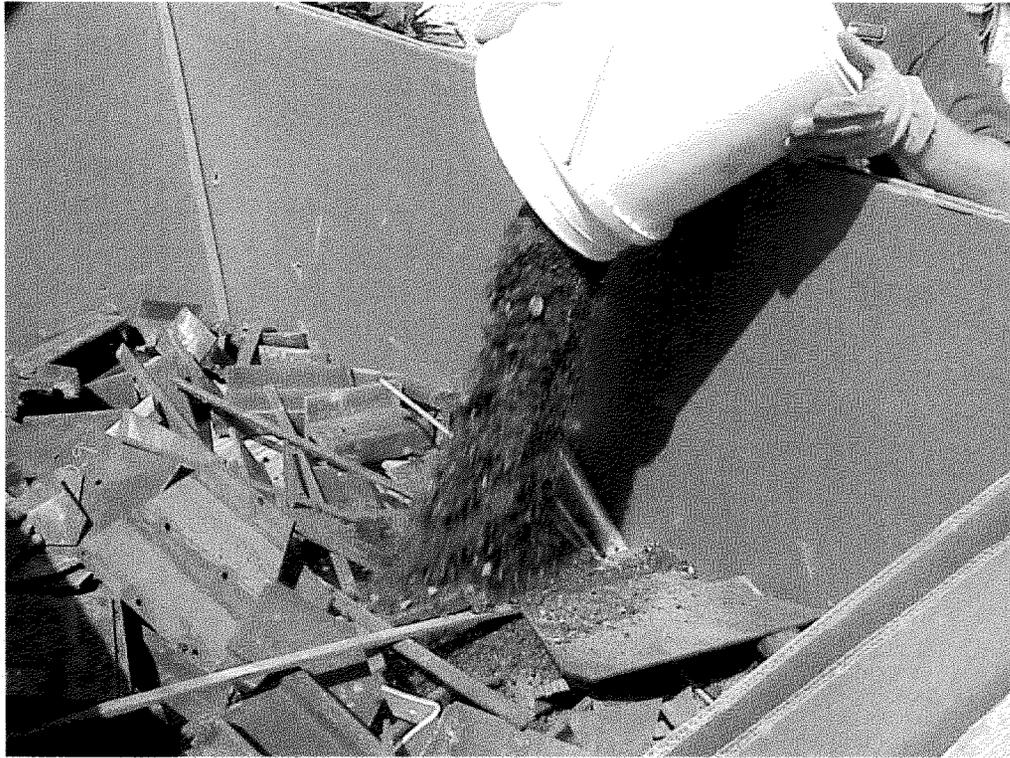


Figure 27. Box being filled with metal, wood, asphalt, and concrete.

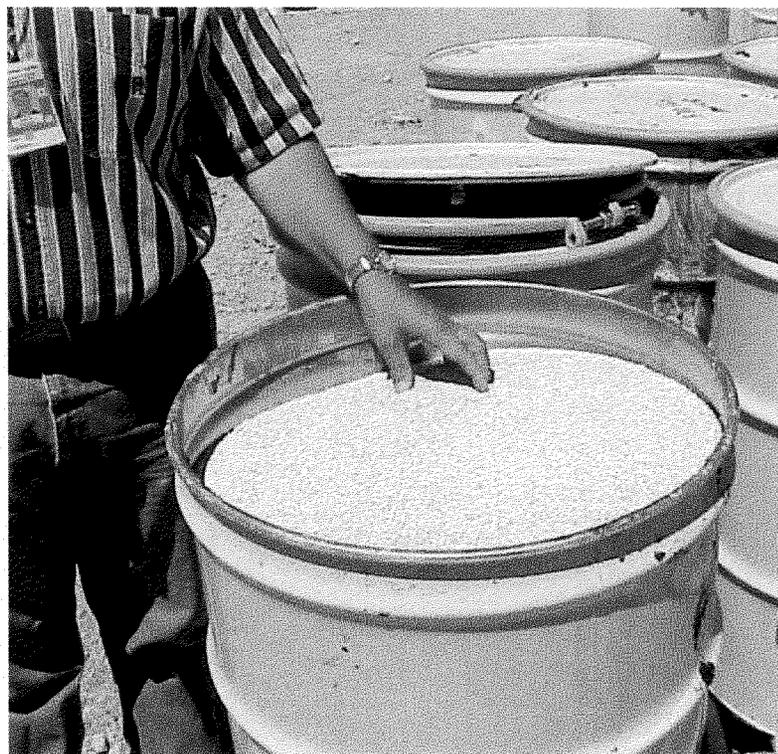


Figure 28. Drum filled with nitrate salts.

5.1.3 Waste Pit Construction

Except for the two boxes, the drums and sacks were placed in the test pit in a random orientation simulating the random dumping that occurred within the INEEL SDA. Figure 29 shows the general design features of the disposal pit including a 2-ft (0.6-m) compacted soil underburden, a 3-ft (0.9-m) overburden, a seam of simulated waste and soil 8 ft (2.4 m) thick, and standard thrust block approximately 17 in. (43 cm) thick with space for grout returns. Pit dimensions are 15 × 15 ft × 8 ft (4.5 × 4.5 × 2.4 m). The layout of the simulated waste in the Cold Test Pit South was designed generally to represent a random dump zone in the SDA pits and trenches. However, many of the drums were strategically located relative to drill hole locations (defined by the hole orientation on the top of the thrust block) to maximize the amount of information collected from the emplaced monolith. Because the hydraulic conductivity measurements (local packer tests) were to be made within the same holes used for grouting, positioning certain waste containers directly under these holes allows examination of the maximum effect that material in the waste container has on local hydraulic conductivity.

There are two general waste composition types: (1) material that will not generally affect grout curing such as combustibles and debris (cloth, metal, soil, asphalt, concrete, and wood), and (2) material that could interfere with grout cure (nitrates and organics). Because of the proportionally small volume of organics and nitrate sources in the SDA, the most representative monolith hydraulic conductivity conditions are near these interference materials but not within an actual penetration of the interference drum. Based on historical data, the volume of the nitrate and organic interference is approximately 10% of the volume of the pit while 90% of the volume is void or containerized soil or cloth, paper, wood, asphalt, metal, glass, and other debris (Vigil 1990).

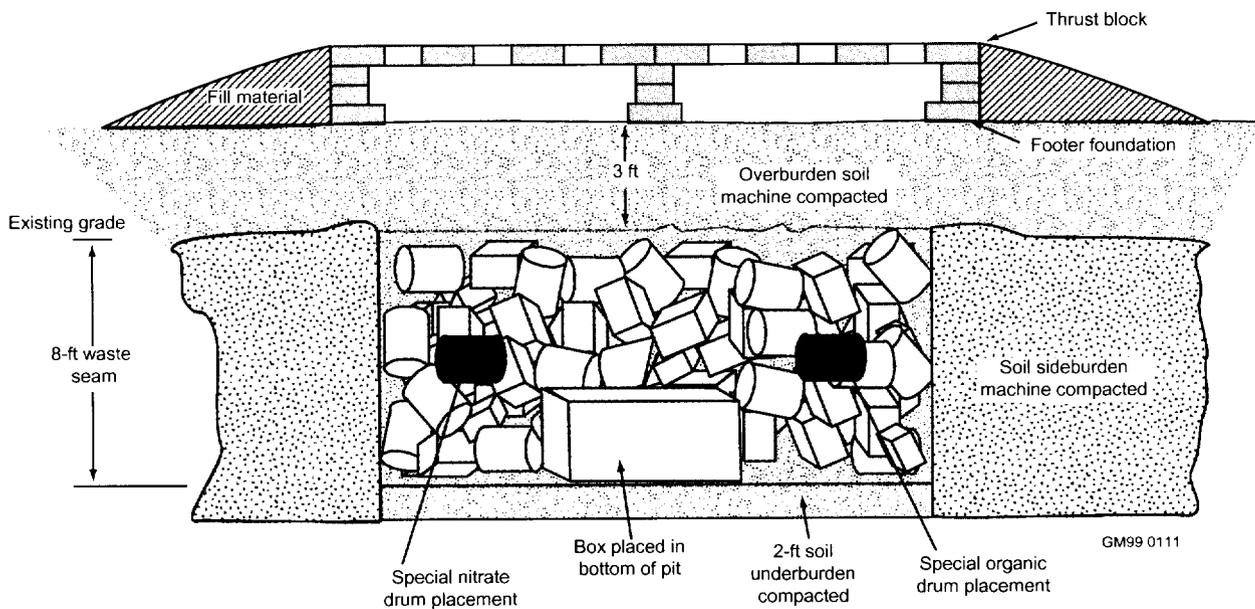


Figure 29. Design features of the long-term disposal pit.

In Figures 30–34, the placement of simulated waste in four 0.6-m (2-ft) layers is shown. The position of each waste form was surveyed after placement. Figure 31 shows the corners of Layer 1 being surveyed after placement and after being covered with a layer of dirt. Basically, each simulated waste container was surveyed such that once buried a three dimensional map of the waste could be recreated. This three dimensional map of each waste form is in Appendix E.



Figure 30. Lower-level 0–0.6 m (0–2 ft).

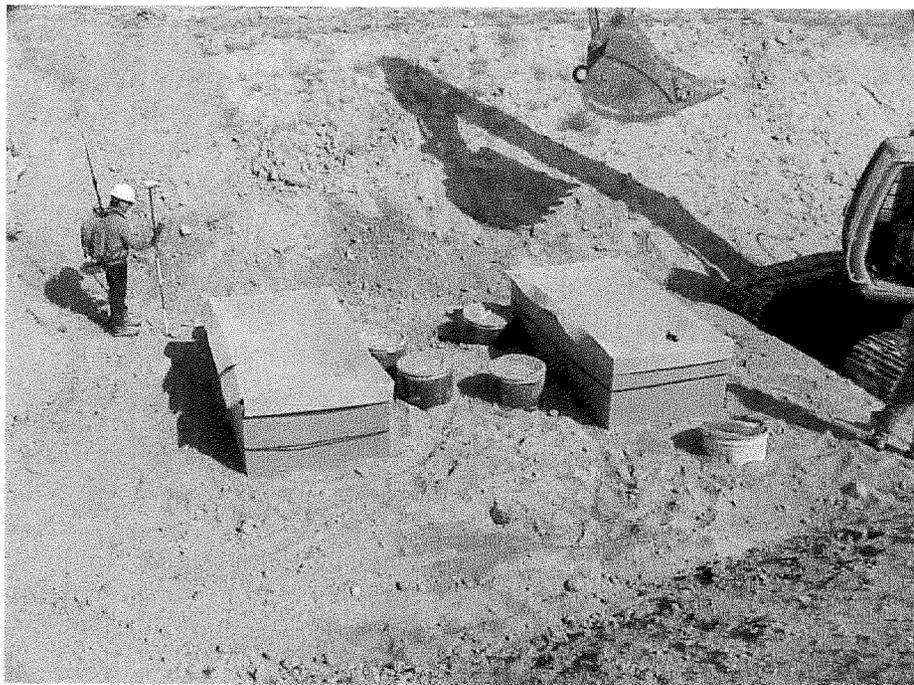


Figure 31. This picture shows the corners of Layer 1 being surveyed after being covered with a layer of dirt following placement.

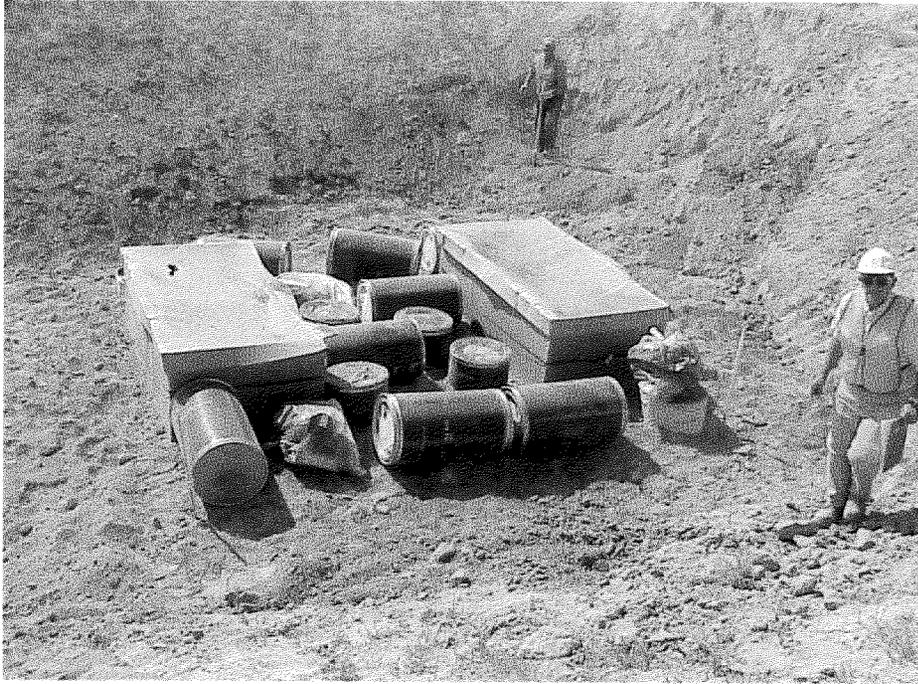


Figure 32. Layer 2, surrogate waste orientation 0.6–1.2 m (2–4 ft).



Figure 33. Layer 3, surrogate waste orientation 1.2–1.8 m (4–6 ft).



Figure 34. Top layer 1.8–2.4 m (6–8 ft).

Once the pit was constructed by completing a backfill of INEEL soil, a large 80 x 122 ft. weather structure was constructed over the top of the pit as shown in Figure 35.

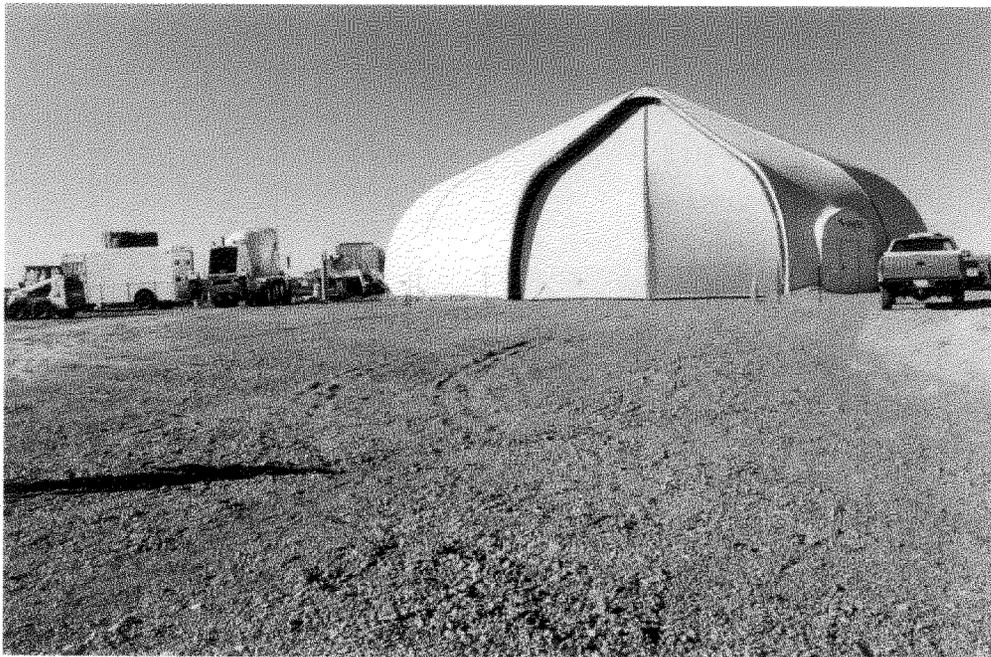


Figure 35. Weather structure constructed over the pit (Photo PNOL-520-1-1).

5.2 Equipment

5.2.1 Grouting System

The grouting system was identical to that used for the implementability testing, except that an elaborate removable contamination control shroud/drill string assembly was used in place of a simple drill string assembly. The major components outside the weather structure were the CASA GRANDE JET-5 pump, the vortex mixer and delivery system, and the high pressure hoses and fittings. Inside the weather structure was the CASA GRANDE C-6 drilling apparatus.

The shroud system was designed to provide a HEPA-filtered flexible double containment of the rotating drill steel during the grouting process. It consisted of a special sealed housing at the top of the drill steel that involved a double grease seal of the rotating drill steel against a canned seal material. To this housing seal was attached a double flexible inner and outer shroud. The space between the inner and outer flexible shroud had a dedicated passive HEPA filter and the space between the drill steel and the inner flexible hose had another dedicated passive HEPA filtration system. At the bottom of the drill steel was a cylinder to which the flexible inner and outer shrouds were attached. This cylinder was called the stinger, and the outside diameter of this cylinder was smooth and was the surface for attaching plastic sheets that were an inherent part of the thrust block glovebox system described next.

Figure 36 shows the drill string shroud in a nearly fully extended position, clearly showing the attachment of the plastic sheeting at the bottom of the stinger, the upper and lower HEPA filters covering the insides of the shroud, and the upper brass seal against which the rotating drill steel is sealed with a double grease seal.

5.2.2 Thrust Block/Contamination Control Features

The thrust block was placed over the pit and bermed with soil such that track mounted drilling system could operate on a level surface. The thrust block only covered part of the pit such that the grouting was divided into two separate operations, one operation with contamination control and the recording of appropriate data related to contamination control and a separate operation without contamination control. Figure 37 shows a schematic with the outline of the thrust block also showing the outline of the pit. The contamination control was to be in effect for holes 1-54 and holes 55-114 were to be accomplished without contamination control.

The thrust block holes [all on an 50 cm (20 in.) triangular pitch matrix] and all other holes [also on a 50 cm (20 in.) triangular pitch] on the pit were predetermined to coordinate with the location of various waste forms to ensure that certain holes corresponded exactly to certain simulated waste materials (specifically the nitrate salts and organic sludges).

The thrust block and shroud assembly on the drill string were specially designed to create a “glovebox” environment for the grouting process. The thrust block was made of carbon steel leaving a 43 cm (17 in.) vertical space to collect grout returns. By using carbon steel rather than concrete, allowed less support structures under the thrust block and more room for grout returns. Basically, the thrust block is a simple box with preformed holes on top that allowed insertion of the drill steel. The thrust block included elaborately designed double “plastic sleeve” ports for each hole in addition to a plastic diaphragm across the bottom of the hole. Referring back to Figure 2, details of the thrust block include the plastic sleeves, the diaphragm, a common pipe wiper to remove excess grout/soil/waste from the drill string when raising the drill steel out of the pit.

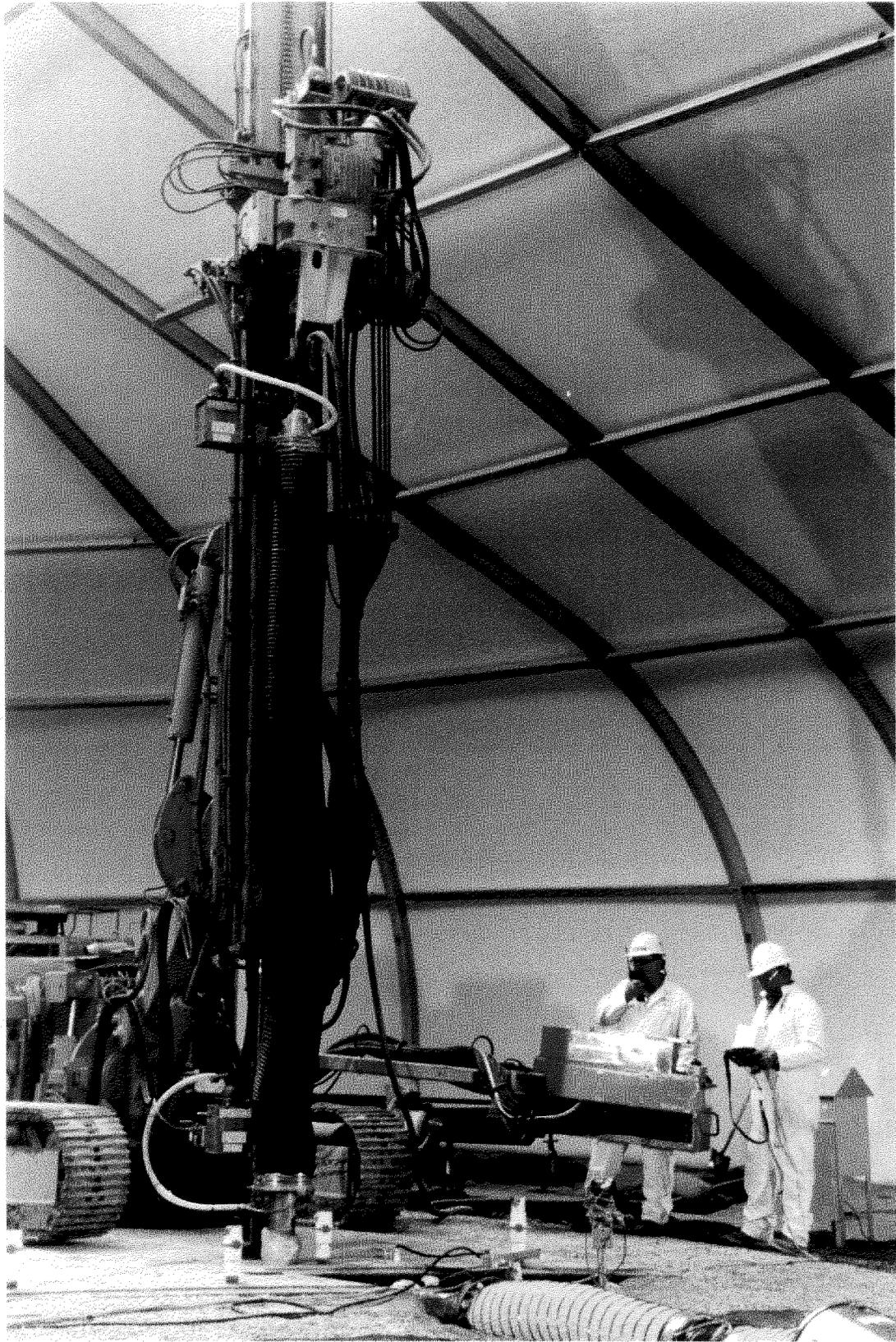
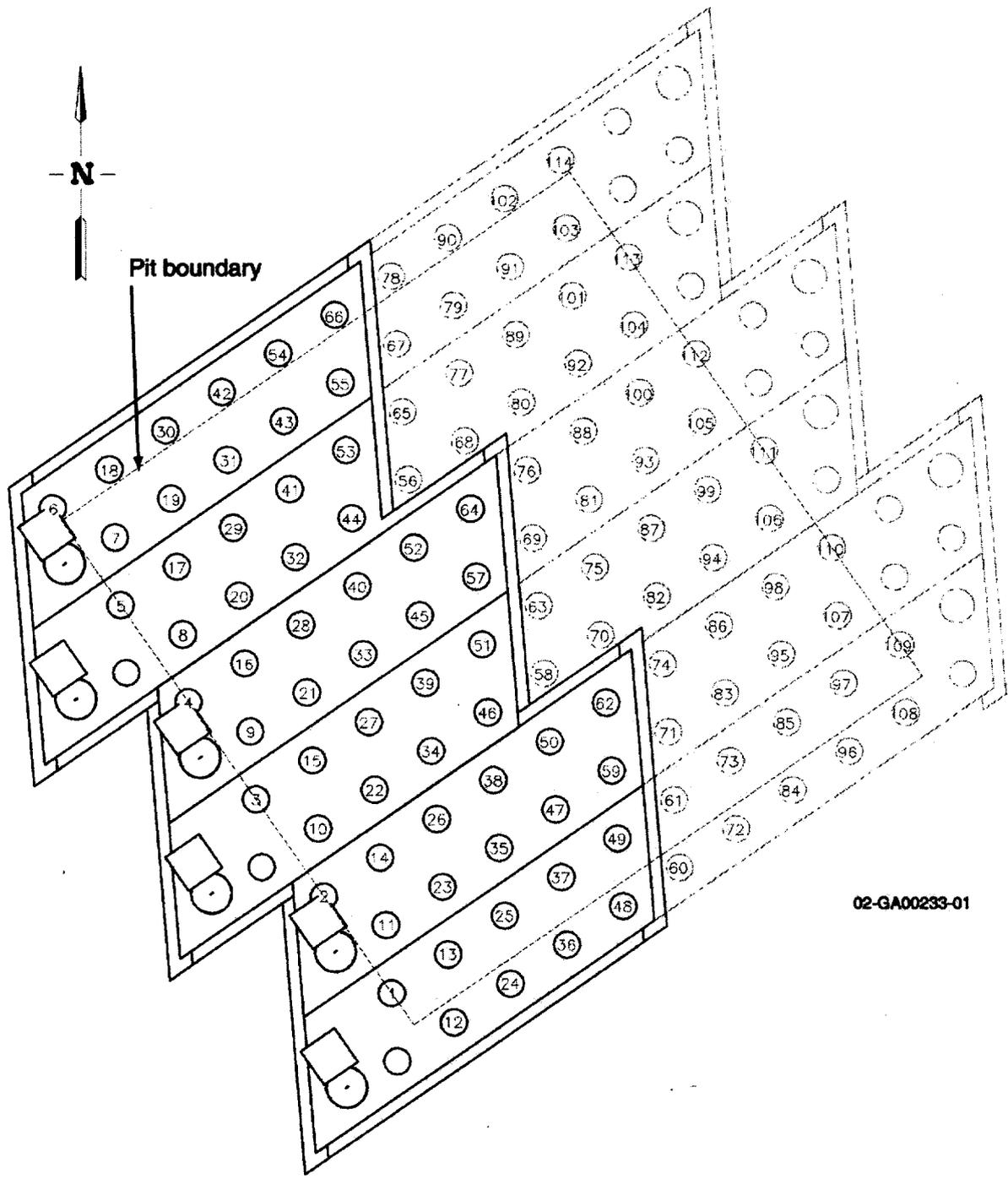


Figure 36. Drill string shroud in operation (Photo PN01-520-4-22).



02-GA00233-01

Figure 37. Schematic of the thrust block over the pit showing the hole numbering scheme.

The underside of the thrust block shown in Figure 38 shows the plastic diaphragm, which allowed pulling the double plastic sleeve around the drill steel without exposing the inner surface of the thrust block to the workers above. Figure 38 also shows the a special rigid plastic plate that aids in removing the plastic diaphragm material such that the material does not foul the drill string under the thrust block and cause this material to come up inside the plastic sleeves once the drill string is withdrawn.

Not shown in Figure 38 is the wiper blade just under the plastic plate which is a rigid rubber material that is solidly supported by brackets under the thrust block. This rigid rubber wiper allows the drill string steel to be cleaned as it is withdrawn from the thrust block.

Figure 39 shows the top surface of the thrust block with the double plastic sleeves removed from the holes and attached to the drill string shroud assembly. It was necessary to treat the 10-mil thick plastic sheeting with common baby powder to allow the plastic to be flexible enough to perform the elaborate sealing of the plastic on the drill stem stinger. Once the double plastic sleeve was attached to the drill steel, the diaphragm was punctured or pulled off by insertion of the drill steel against the rigid plastic plate. In addition, the top of the hole is slightly recessed and each hole had a solid metal top that allowed a flat surface on the thrust block.

Other design features of the thrust block included inlet and outlet holes for attaching a manifold for the HEPA air filtration system. Also, special vent manifolds were attached to the top of the thrust block to allow release of free air through special HEPA filters as the thrust block was filled with grout during a final thrust block fill phase discussed later. Additional penetrations in the top of the thrust block included special fill locations in which the final fill of grout was to take place. In these locations, the double plastic sleeve and diaphragm are also utilized.

The thrust block was bermed with dirt providing an airtight seal to the ground surface. The berming of the thrust block also allowed the drill platform to operate in a more or less level environment for all positions on the thrust block. In addition, the thrust block included ports and manifolds for a High Efficiency Particulate Air filtration system with inlet and outlet manifolds. This system also allowed keeping a negative pressure and a Data Acquisition System allowed measurement of negative pressure and temperature and relative humidity of the air going into the filters.

Figure 40 shows the drill string assembly in position on top of the thrust block. This figure shows the outlet manifold for the HEPA filtration system, the special vent ports used for final filling, and the camera view port with camera inserted. Also shown is the double flexible shroud around the drill string.

In the background, is a control trailer to which is fed the data from the HEPA filtration system (relative humidity, pressure relative to atmosphere under the thrust block) and most importantly the video taped view of the grouting operation under the thrust block. Also shown in Figure 40 are the holes with flush-mounted metal tops.



Figure 38. Detail of the underside of the thrust block showing plastic diaphragm and the rigid plastic plate.



Figure 39. Plastic sleeves extended out of thrust block in preparation for testing (treated with talcum powder) (Photo PN01-520-3-5A).

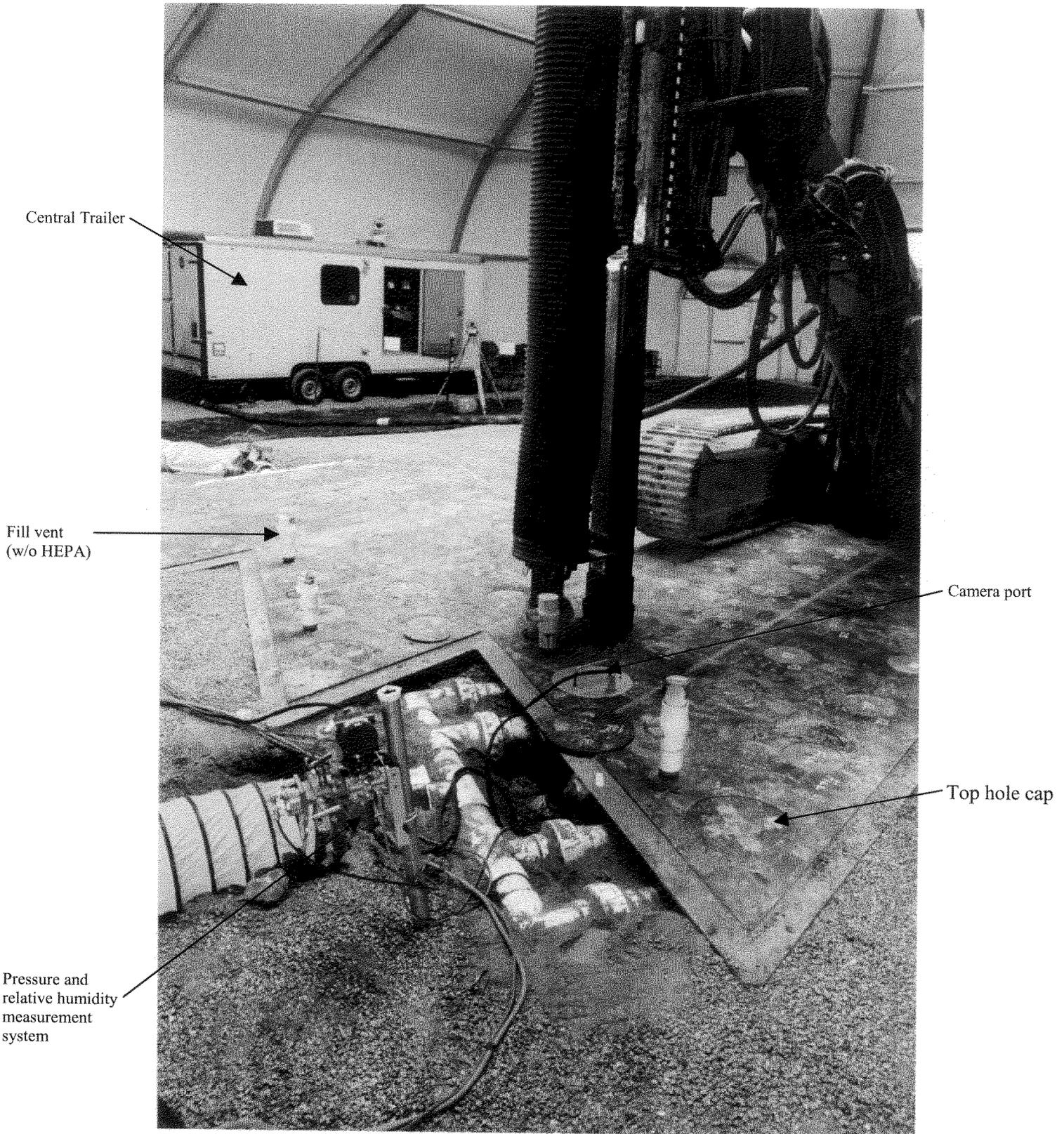


Figure 40. Drill string on top of thrust block (Photo PN01-520-4-15).

Figure 41 shows the passive HEPA filters on the inner and outer shrouds (one at the top for the space between the drill steel and the inner shroud and one at the bottom for the space between the inner shroud and the outer shroud).

Figure 42 shows another view of the grouting operation with the strategically placed high volume air filters arranged around the thrust block.

Another design feature of the thrust block included a Lexan viewing chamber for locating a remotely controlled Television Camera that extended down into the cavity under the thrust block. Since the thrust block consisted of separate panels allowing supporting “I” beams underneath, each panel had a viewing well. Within the well was placed a TV camera for online and recorded operations. This allowed monitoring of the amount of grout returns under the block during all phases of grouting. These data were feed back to a control trailer located within the weather structure and a second hand held viewing camera was utilized by the grouting operator. In this manner, excess grout returns could be visually monitored and the grouting operation could be immediately stopped if excess returns were observed. Figure 43 shows a view underneath the thrust block which is essentially the view as seen by the camera.

5.3 Grouting Procedures

To grout an individual hole, the following basic procedures were followed:

- Place the drill rig over a hole to be grouted.
- Remove top hole cover.
- Bring the double bag out of the hole and extend both bags to their full extension.
- Place a strap wrench on the bottom of the double bags to keep the two bags from extending into the hole when the bags were placed on the stinger
- The inner bag was sealed onto the stinger at a high point using plastic strapping, folded over, and forced down to the next lowest point (shown in Figure 44)
- The outer bag was then also strapped with plastic straps, folded over and also pulled down to the line of the elevation of the inner bag.
- When the inner and outer bag were in position, the strap wrench was removed and the drill string was inserted breaking the lower plastic diaphragm.
- Once fully inserted into the waste, jet grouting was started as the drill string was withdrawn in predetermined steps until the nozzles were at the top elevation of the waste. At this point, the drill string was withdrawn through the overburden without grouting.
- The nozzles were positioned in the space under the thrust block, and the drill stem was allowed to drain its grout.
- Once the drill string was withdrawn into the stinger, the entire drill string/shroud assembly was tilted back allowing access to the bags.
- The bag was twisted off and taped (shown in Figure 45).
- The twist off and taped area was cut with either a pipe shear or a special heat knife.

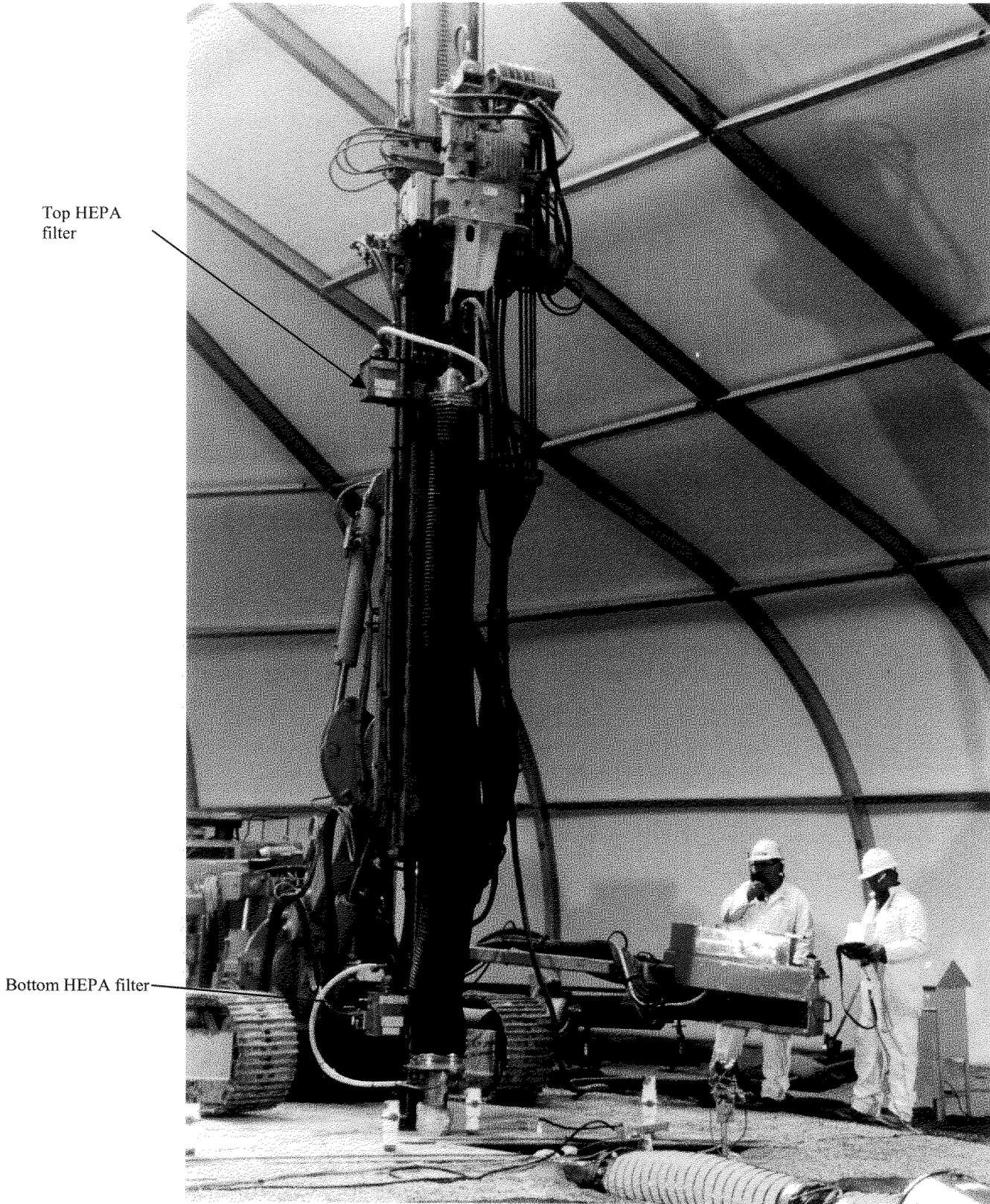


Figure 41. Drill string being inserted into waste pit (Photo PN01-520-4-22).

Figure 42. Air samplers in weather structure (Photo PN01-520-4-25).





Figure 43. View underneath thrust block.



Figure 44. Placing plastic sleeve material on drill string stinger (Photo PN01-520-4-10).

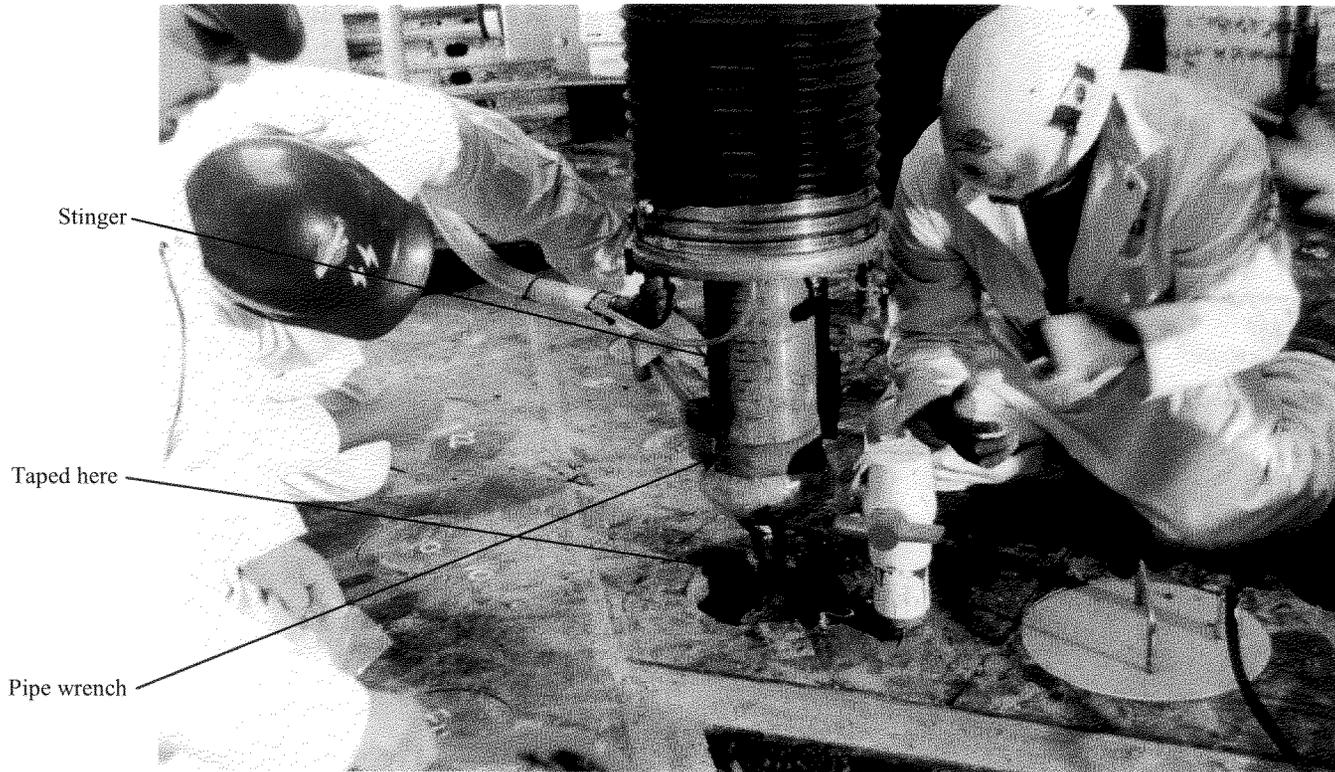


Figure 45. Twisted and taped plastic sleeves ready to cut (Photo PN01-520-5-2).

- This procedure was repeated until the number of plastic bags (upper part of the bags) on the stinger filled the approximately 35 cm (14 in.) of surface. It is noted that the last bag still had a twisted and taped bottom thus maintaining contamination control.
- After the stinger had no more room for bags because of an accumulation of upper bag material, the entire region of upper bag material was taped creating a new smooth work face (the surface of the Duct tape was similar to the surface of the original stinger). To facilitate dropping off the last twist off/taped bottom into the next hole by insertion of the drill string, the bottom taped zone on the stinger had to be perforated. A heat knife and/or a pocket knife was used for this operation. This was mandatory because on that last application of Duct tape, insertion of the drill steel would not pull off the last bag rather the drill steel would simply puncture the bag leaving excess plastic material on the bottom of the stinger.
- The process was repeated until either the shroud required a change out due to nozzle plugging or there was a compromise in the shroud contamination control system.
- Once the space under a specified series of panels in the thrust block was grouted, the remaining void was to be filled with a bentonite slurry mix. This filling was to be done to allow assessing the amount of void left in the thrust block and therefore to calculate the total volume of the grout return realized during the grouting operation by subtracting the bentonite slurry volume from the design volume under the block. In an actual hot operation, this void would be filled with grout to create a nearly impervious top cap. The vents on top of the thrust block would be fitted with a special passive HEPA filter and the air within the void under the thrust block would be expelled out these vents.

5.3.1 Special Procedures for Plugged Nozzles

Plugging of nozzles during jet grouting was an expected event based on past grouting experience (Loomis 1997, 1999). Plugging can occur due to intrusion of wet clay into the nozzles during the drilling sequence but can also be accentuated by grouts that filter cake. Another common source of plugging is debris within the grout delivery system all the way from the delivery truck to the pumping equipment. In the past (even in hot applications—Loomis 1999), there was always a trickle-flow of grout allowed. However, because of the use of the glovebox-like system in the thrust block using the plastic sleeves, the trickle flow was stopped, and the system was allowed to drain between grouting holes. Special testing was performed during the implementability testing and it was found that in the 1 to 5 minute timeframe the grout had drained to the point where no trickle flow of grout was observed. Therefore, the procedure was to stop all active pumping of grout and allow the system to drain into the thrust block. Until visually using the remote camera under the thrust blocks, no flow was observed coming out of the drill stem.

If in the event that a nozzle plugged, rotopercussion of the drill system was to be used until the nozzles were clear of debris or grout. If that failed, a complete new drill string/shroud assembly was to be employed and the old assembly taken to a separate area (simulating an adjacent glovebox) and the nozzles cleared. During the special System Operations testing at the shroud manufactures plant, it took nominally 1 hour to replace the old shroud with a new one. Once cleared, a new plastic bag was attached to the bottom of the drill assembly and the outside of the bag decontaminated. To this end a total of three complete drill string shroud assemblies were fabricated and on hand for the testing procedure.

Grout Mixing at Batch Plant

Grout was mixed in Idaho Falls-50 miles from the Cold Test Pit South in a specially designed batch mixing plant shown in Figure 46. The system involved mixing dry ingredients of GMENT-12 with water in a high shear mixer also shown in Figure 46. The mixture was transferred to Ready Mix trucks (clean-new dedicated trucks for this project). Grout was delivered in 3,024 L (800 gal) batches three times a day. Since each hole was to use nominally 378 L (100 gal) of grout it was possible to support grouting up to 24 holes per day. Enough dry ingredients were purchased to support 7 days of grouting operation. Since it was planned to only grout 114 holes (which would in a perfect application take nominally 5 days) there was an extra 2 day's supply of grout at three loads per day. In short, there was nominally an extra 18,144 L (4,800 gal) of grout that could be wasted during the anticipated 5-7 days of operation.

Table 31 contains a summary of the detailed mixing operations.

Table 31. Summary of GMENT-12 grout batch mixing times and grout data.

Mix Date	Batch Invoice No.	Plant Departure Time	Job Site Arrival Time	Marsh Funnel Time (sec)	Grout Specific Gravity
October 11, 2001	16669	8:40	10:05	52	1.86
October 11, 2001	16690	13:22	14:40	57	1.87
October 12, 2001	16697	6:49	8:00	51	1.85
October 12, 2001	16713	10:35	12:00	61	1.87
October 12, 2001	16738	14:13	15:30	60	1.87
October 15, 2001	16772	6:55	8:00	55	1.87

All batches were 4.0 cubic yards.



Figure 46. Batch mixing operation (Photo PN01-0520-8-9A).

The dry grout product was mixed with water using a HighShear® 1,000 grout mixer with two MK 2 Colcrete colloidal mixers each driven by a 30 HP electric motor. The dry grout was weighed using a portable batch plant provided by Valley Ready Mix, Inc., and the water volume was measured using a Fill-Rite Series 900 flow meter supplied with the grout mixer. Prior to production mixing, a test batch was prepared to verify the accuracy of the mix proportioning. The specific gravity of the mixed grout is used as a quality control parameter to insure that the desired ratio of water to dry material is achieved.

During grout production, the grout is mixed in 1.33 cubic yard batches. The desired amount of water is added to the grout mixer through the flow meter. Once the water in the mixer has flooded the pumps and has risen above the pump inlet, the pumps are started and the addition of the dry grout material is initiated. The addition of water is stopped upon reaching the desired volume. Mixing of the grout continues as the remainder of the preweighed dry grout material is added to the mixer. After addition of the water and dry material is complete, mixing continues until the mixture is free of any lumps of partially mixed material. With the high shearing action of the Colcrete colloidal mixers, the high flow rates, and the relatively low viscosity of the GMENT-12 grout, a strong vortex is formed in the grout mixer during mixing (see Figure 47). The vortex is very effective in drawing any poorly mixed materials through the high shear Colcrete colloidal mixers where it is thoroughly mixed with the water, producing a homogeneous product. Once all of the dry grout material is in the mixer, the mixing is completed in about 60 to 90 seconds, and the grout is ready to be discharged into a concrete truck for transportation to the INEEL Cold Test Pit site.

Three batches of 1 cubic m (1.33 cubic yards) each were used to produce the 3 cubic m (4 cubic yard) loads that were used for the INEEL Cold Test Pit project. After the three batches of grout were loaded onto the concrete truck, the grout was blended by the concrete truck. Following the blending by the truck, the grout was sampled and tested. The specific gravity and Marsh funnel time were determined and recorded on the batch ticket. A time stamp was placed on the ticket prior to the truck leaving the plant. Mixing, screening, and testing of a 3 cubic m (4 cubic yard) batch took about 30 to 40 minutes.



Figure 47. Grout mixer vortex view after 60 seconds of mixing.

Initially, the grout was to be screened as it was unloaded from the concrete truck at the INEEL Cold Test Pit site. When the first load was screened at the site, some unexpected material consisting of small hard fragments and fibrous material was retained by the screen. Subsequent batches of the grout were screened at the mixing location as the grout was discharged from the grout mixer into the concrete truck. The grout was passed through a double layer of standard wire window screen as it was loaded into the concrete truck. The foreign material is not usually found in the raw materials and has never been encountered in previous field or laboratory testing of the grout. The source of the foreign material has not been positively identified at this time.

5.4 Evaluation of Grouting Operations

In 2 days, a total of 12 holes were successfully grouted with 4,936 L (1,306 gal) of grout injected into the void space of the pit as shown in Table 32. This grout was emplaced into the voids of the pit with minimal grout returns; therefore an average of 408 L (108 gal) of grout was delivered in each hole and there was nominally 51 L (13.6 gal) of grout delivered per linear foot of waste. There was an attempt to grout the 13th hole at the end of the second day of grouting but this hole was abandoned due to plugging of the nozzle. At the start of the third day of grouting, the operation was terminated when attempting to grout the 14th hole due to an injury accident to a grouting subcontractor. A high pressure fitting at the exit to the pump catastrophically failed causing a piece of the fitting to strike the subcontractor with a resulting serious injury accident (see following section on lessons learned).

What follows is a description of the grouting process for the 12 holes leading up to the accident. Complete logbooks for the grouting operations are contained in ER-004-02. The thrust block filling operation described above was not performed and only a limited amount of contamination control data was obtained; however, a large body of operating experience was obtained prior to the accident in a limited amount of holes grouted and this information is contained in this section.

Grouting started on hole number 1 as shown in the schematic in Figure 37. The first 6 holes were considered edge holes, and for these holes the plan was to inject a lower amount of grout in that the hole spanned the edge of the pit and was a mixture of pure soil with low voids and debris waste with considerable voids. For holes in the interior, there were expected to be mostly debris type material with considerable voids. Appendix G contains an analysis of the expected void fraction and concluded that in general, there would be an expected void of 60%. Therefore, during the grouting campaign, it was planned to attempt to fill the pit with 60 to 70% of the volume of the pit using the 114 holes. What would dictate a local change to that rate of filling would be the amount of grout returns observed using the remote TV camera. If the returns became unacceptable (enough to fill the void space locally with a viscous return of grout and soil/waste mixture) then the grouting operation locally would be terminated and moved to a new hole.

The relative humidity and temperature of the air for the thrust block HEPA filtration system were continuously measured and it was found that at the start of each day, the humidity was higher and as the air flow continued under the thrust block, the humidity reached a lower steady state. On the first day the humidity was measured at 89% and 54F and during the day it reached a more or less steady value during grouting of 69% Relative Humidity and 58F. During the second day, the Relative Humidity continuously decreased as the day wore on with the first reading upon turning on the fans was 98% Relative Humidity reducing to around 67% Relative Humidity by the end of the day with 14 C (57F). This high relative humidity at the start of the day followed by lower values as the day wore on is basically attributed to the air flow removes fluid from the air. From an operational standpoint, using water based grouts does raise the humidity and the system when continuously running has a nominal 67% Relative Humidity which must be factored in to HEPA filtration system designs.

Table 32. Summary of grouting during field testing (3.78 L/gal).

Hole	Elapsed Time	Grout Delivered	Pressure at the Pump	Grout returns	Comments
1	First hole of day-29 minutes from start to finish	129gal/4s/step	400 bar	None	Observed a 2-minute drain of the nozzles using the remote camera
2	44 minutes	135gal/4s/step	400 bar	None- a pint of neat grout came up to the surface as the drill string was withdrawn but the material went back down the hole	Took 34 minutes to reposition the drill stem and attach the plastic sleeves. The plastic sleeve with about 2.5 gal of neat grout fell onto the top surface of the thrust block when moving to hole number 3. Area was decontaminated.
3	80 minutes (includes cleanup time for spill)	120 gal/3.5s/step	400 bar	None	No problems; various solutions to the drain of the drill steel discussed
4	82 minutes	114 gal/3s/step	400 bar	None	Nozzles plugged-used rotopercussion to unplug them. Resistance in drilling at 2 ft into waste and 3.5 ft into waste
5	51 minutes	113 gal/3s/step	400 bar	1-quart of viscous grout returns possibly in a nitrate drum	The grouting was split as the nozzle plugged after grouting a few feet. The nozzle was unplugged by using rotopercussion. Small drip of neat grout as draining of drill steel incomplete and grout fills the bottom of the bag
6	52 minutes	85.44 gal/3s/step	400 bar	½ gal return during grouting; however no net return as when the drill steel was withdrawn, the return went back down the hole	Started using “Chem-wipes” on the top surface to contain any returns due to the leaky bag as the drill steel never completely empties of grout.
7	57 minutes	104 gal/3.5s/step	400 bar	½ gal of viscous material as drill string withdrawn	Hole 7 ended the first day of grouting.
8	First hole day	91 gal/3.5s/step	400 bar	None	Utilized compressor to blow out remaining fluid in the drill steel. About 2.5 gal of material came out.
9	39 minutes	88 gal/3.5s/step	400 bar	None	No comments
10	36 minutes	125 gal/4.5s/step	400 bar	4 in. of material heaved up as the nozzles got near the top of the waste	Increased flow for an interior position.
11	37 minutes	96 gal/3.5s/step	400 bar	None	No comment
12	37 minutes	104 gal/3.5s/step	400 bar	6 in. cone of viscous returns came up around drill stem	None
13	Hole abandoned	None	NA	NA	Nozzle plugged with system at 500bar-dangerous situation as grouting subcontractor relieved pressure

Table 32 contains the data on the grouting operation for the first 13 holes. Basically the operation was progressing as planned with several exceptions as discussed below. There was an efficiency of operation realized as the process proceeded in that there was a marked decrease in the time to grout a hole as the process proceeded from hole 1 to hole 13. It appeared that all 114 could be grouted using the technique involving the thrust block as planned. The following issues arose during the grouting process of the first 13 holes described in Table 32 and (ER-004-02) contains a detailed log book of all major data taken during the field testing:

- Debris in delivered grout
- Draining of the excess grout in the drill stem, spillage of neat grout on the top surface of the thrust block, draining the drill stem of grout utilizing an air bleed point
- Expediting the time to perform the grouting process/wasted grout
- Plugging of the injection nozzles
- Plugging of the injection nozzle with lost time to relieve pressure
- Complete detachment of the inner and outer flexible hose on the shroud assembly, filling of the lower HEPA filter on the drill string shroud with grout, deterioration of the inner shroud due to mechanical contact with the drill string
- Collapse of outlet air line in HEPA filtration system for the thrust block
- Limited view of volume under the thrust block because of length of camera stem relative to the Lexan tube.

5.4.1 Debris in Delivered Grout

During delivery of the first two loads of grout there was considerable small debris observed on the screen at the entrance to the vortex mixing system. In fact, the debris was considered a serious enough problem to preclude jet grouting with the relatively small 2.4-mm nozzles used for the GMENT-12 grout. This was particularly puzzling to the grout vendor in that new delivery trucks were utilized for this project. The solution to the problem was to double screen the material at the batch plant. Upon a closer examination of the material on the screen, it was determined that it looked like material from a mouse nest that had collected in a pump somewhere in the system. After the raw material was double screened at the plant, the quality of the grout product delivered to the site improved.

5.4.2 Draining, Spillage, Cleaning

During implementability testing, once the drill string was brought to the surface, the draining of the drill steel of excess grout was timed and it was found that the grout drained out in the 1 to 5 min time period. Therefore during the field testing it was planned to allow a 5-min drain of the drill steel after the nozzles were brought to the space under the thrust block and in direct line with the remote TV cameras. After the first hole, it appeared that the drain time of a few minutes would allow essentially a complete drain of the drill stem and thus the taped plastic sleeve would not fill with grout and cause spillage onto the top surface of the thrust block.

Following successful grouting of the first hole, testing proceeded without note to the second hole and the same procedure was followed (i.e., allowing a few minutes of draining coordinated by viewing

the nozzles with the remote TV camera). During the move between hole 2 and hole 3, the taped and twisted plastic sleeves filled with grout to the point that the frictional fit of the sleeves on the stinger caused by the plastic ties was insufficient to hold the head of grout and the sleeve fell off the stinger. About 2 L of grout spilled onto the top surface of the thrust block. From a contamination control standpoint, spilling neat grout that had been in contact with the nozzles and corner drive point was a potential disaster. However, the spillage was indeed neat grout that had been in the clean drill steel and the only chance of contamination was dripping of grout down the outside of the drill steel or small amount of contaminants that were in the nozzle area. Terbium contamination dripping down the outside of the drill steel was unlikely considering the extensive wipe that had occurred as the drill string was withdrawn across the wiper material in the bottom of the thrust block. In addition, the first and second hole both were on the edge of the waste and there was a high probability that the drill string had not encountered the terbium tracer. Therefore, the drill steel was rebagged and the area was deconned with Chem-Wipes and water and grouting proceeded to the third hole. When grouting the third hole, an attempt was made to allow more time to drain the drill steel and rotopercussion was applied until draining of drill steel seemed likely. This process took up to 15 minutes while watching progress under the thrust block with the remote TV camera. In addition, it was decided to tape the bottom of the cut twist-off sleeve to avoid leakage of grout onto the top surface of the thrust block. As an added precaution a separate plastic bag was also taped to the bottom of the twist off area.

As grouting proceeded, it became apparent that the system required a bleed point in the line to break the vacuum holding up the fluid and indeed that was implemented with positive results. The line was broken at a hose connection where the high pressure hose entered the tent. In addition to a simple bleed, compressed air was also blown into the line thus completely clearing the line of fluid between moves. This temporary fix was employed for the remainder of the holes; however, it was recognized that it was only a temporary fix for the treatability study. What was needed was a positive high pressure bleed valve in the high point of the drill system swivel (where the hose interfaces with the drill steel). This would require a design and fabrication effort beyond the scope of the treatability study.

5.4.3 Excess Time to Perform Grouting/Wasted Grout

From a time-motion standpoint, the time to perform the grouting operation improved as the number of holes progressed. The early holes proceeded slowly with the first 6 holes taking an average of 53 minutes and the last 4 holes took an average of 37 minutes. The very first holes took up to 80 minutes per hole but as the process progressed, the last holes grouted took on the order of 30 minutes. This improvement reflected the learning curve on the combination of placing the plastic sleeve on the stinger, drilling/grouting/draining, and twisting off the sleeve and cutting the taped area. Further expediting could be accomplished by using only one plastic tie on the double bag rather than the two that were used for the first 13 holes which could reduce the time to around 25 minutes per hole. Change out of the drill string shroud was performed just prior to the accident. It took 1 hour and 10 minutes to remove the shroud and another 30 minutes to rebolt the shroud in place. It was obvious that the operation could be performed in a few minutes if the mounting system utilized an alignment bar and easier access to the bolts (this also applies to the HEPA filter mounting bolts). This operation was performed with a crew for the first time and they were basically on a learning curve for the shroud replacement.

Grout was to be delivered three times a day 3024 L (800 gal) at a time. The grout was mixed in Idaho Falls and delivered in Ready Mix trucks and there was a lead time of at least 2 hours to order a new batch. In other words, depending upon what was happening 2 hours prior to delivery was expected, the grout had to be mixed and delivered. What would happen is that problems would develop during that 2-hour window and in the extreme case, the on site delivery truck had not delivered any grout to the pumping equipment at the time the new truck came. This resulted in multiple dumping of full trucks of

grout during the 3 days of grouting. In fact, there was 18,144 L (4,800 gal) delivered and only 4,936 L (1,306 gal) injected. What was needed was a portable ready-mix plant at the Cold Test Pit South.

5.4.4 Plugging of Injection Nozzles

Plugging of the injection nozzles is an inherent weakness for the in situ grouting in that grouting particulate grouts there is a potential for “filter caking” or deposition of particulate in low cross section regions such as nozzles. In addition, there is a tendency in clay soils to mechanically plug the nozzles with wet clay during the downward drilling process. In past demonstrations, to combat this tendency, a trickle flow of grout was maintained when moving from one hole to another. In the earliest tests, this small trickle of grout was simply allowed to freely flow over the grouting area but in a hot treatability study in the Acid Pit (Loomis 1999), a cone and catch cup was placed around the drill steel which allowed the flow but contained it to the catch cup. In all other prior grouting demonstrations and treatability studies, there was physical access to the nozzles and clearing of the nozzles usually involved simply lowering the pressure in the system and clearing the nozzle with a simple thrust of a piece of wire. However, in the current treatability study with the “glovebox” concept, there were two drawbacks: 1) since the system was sealed by the twist off of the plastic sleeve, trickle flow was not allowed because there was no volume to collect this trickle flow during the extensive time period between holes and 2) because of the glovebox nature there was no access to the nozzles for physical removal of the plug. Because of this, considerable plugging of the nozzles occurred which caused much of the delays. Interesting enough, much of the plugging was cleared by utilizing rotopercussion of the drill system while the nozzles were under the thrust block and visible to the remote TV cameras. However, after grouting 12 holes, an attempt was made to grout the 13th hole and plugging occurred which was not clearable by using rotopercussion. What was used during the first 12 holes was a combination of short bursts of pressure from the high pressure pump combined with rotopercussion as the drill stem was inserted into the waste. Since it took nominally 1 hour to replace the drill string/shroud assembly, it was desirable to develop a technique to clear the nozzles by mechanical insertion of a wire perhaps in a special glovebox adjacent to the drill/grouting operation.

5.4.5 Plugging of Injection Nozzle with Lost Time to Relieve Pressure

As mentioned in the preceding discussion, plugging of the nozzles was a common occurrence and the technique of pulsing the high-pressure pump while applying rotopercussion to the drill stem was routinely applied as the drill stem was inserted. When grouting hole number 13 this operation resulted in a nozzle that would not unplug and the system was pressurized to 500 bar (7,500 psi). The subcontractor procedure for this relatively dangerous situation is to relieve the pressure gradually by opening a relief bolt in the manifold immediately adjacent to the pump. After considerable effort involving approximately 1 hour of gradually relieving pressure on the system, the system was once again readily to grout, however, no amount of rotopercussion could break loose the nozzle plug and an attempt on grouting hole number 14 was abandoned.

5.4.6 Detachment of Shroud, Wearing of Material on Shroud, HEPA Filter Clogging

During the second day of grouting (holes 8-14), a slight detachment of the top shroud was observed and the system was moved outside the weather structure and the area taped with duct tape for completion of the days work with the understanding that there would be a shroud change-out before starting the next days work. The fact that the weld broke at the top was very confusing in that it was considered to be a non-moving part with little stress. At the end of the day, it was decided to install a new shroud assembly and destructively examine the old shroud assembly. Two findings were evident, 1) the inner and outer shroud weldment had broken most likely due to the extensive rotopercussion applied during the grouting operation, and 2) the inner shroud material had indeed worn against the rotating drill stem near the bottom

as the drill was inserted. This was caused by an inherent twist in the shroud as the shroud was compressed. It was thought that by putting the proper twist in the inner shroud during construction could have circumvented this event. Another possible solution is to employ “spacers” in the design to keep the inner shroud from touching the rotating drive string. In addition, by using a better weld technique at the top, the weldments should have withstood the rotopercussion operation. After consultation with the manufacture of the drill string shroud, it was clear to them that a simple tack type weld was insufficient and a more robust weld should have been applied at this attachment point. Another interesting event occurred related to the shroud wear near the bottom. It was observed that when tipping the drill string to the horizontal positions which is done from time to time during normal operations, there was a filling of the bottom HEPA filter with fluid which rendered it ineffective. Once observed, the HEPA filter was changed out with a new one from one of the spare shrouds. Most likely, although not proven, fluid grout entered the space between the inner and outer shroud via the tear on the under shroud. It is unlikely that the fluid in the bottom HEPA system came from evaporated fluid under the thrust block in that during operation of the thrust block system, the relative humidity was measured at about 67% @ 57°F air temperature.

5.4.7 Collapse of Air Line in Thrust Block HEPA Filter

The air flow in the thrust block HEPA filtration system was adjusted to allow a negative pressure operation without a totally collapsed air line in the outlet of the thrust block. At first, the negative pressure under the thrust block was operated at 1.27 cm (0.5 in.) of Water which resulted in a complete collapse of the outlet line air hose. To avoid burning out the HEPA filtration pump, the negative pressure under the thrust block was cut back to the tenths of inches of water negative pressure. The range of operation for the first day of grouting was -0.076 to -0.152 cm (-0.03 to -0.06 in.) of water with most holes grouted at -0.03 in. of water. For the second day of grouting, the system range of -0.05 to -0.127 cm (-0.02 to -0.05 in.) of water with most of the holes grouted at -0.05 cm (-0.02 in.) of water. The problem with the outlet hose was that it was a collapsible plastic vent hose which would be better suited to a hard PVC piping type system.

5.4.8 Limited View of Volume Under Thrust Block

In general, the remote camera in the thrust block performed perfectly and gave a high quality video recording of the top surface of the soil under the thrust block. Since only the first few rows were grouted, there was no problems seeing the drill string go in and out of the ground and indeed, the removal of the plastic sleeve from the previously grouted hole was also visible. However, because of the length of the camera and the length of the LEXAN camera port as designed, there was a poor view of back rows of holes. What was needed was a deeper LEXAN well or a different camera design. In general, the camera provided invaluable feedback during grouting and would be considered mandatory for this type of operation.

5.5 Evaluation of Contamination Control

Contamination control of the finely divided plutonium particles was a central thrust of the treatability study. Operating with no spread of the terbium tracer above background was considered the performance standard of the thrust-block/glove-box approach. To assess this, a series of smears and air monitoring with high volume filters, were obtained as part of the grouting procedure. It was planned to take a smear sample of the top surface of the thrust block on every third hole grouted starting with hole number 1. Since the grouting process was truncated by the accident, only a small data set was obtained for this measurement. Other data included taking a smear sample on the inside surface of the drill steel (inside the shroud), a smear sample on the outside surface of the inner shroud, a smear sample on the inside surface of the outer shroud. Other data included an extensive background measurement for the

local air for terbium tracer. A total of 11 individual backgrounds were obtained using the seven high volume samplers located around the thrust block and during grouting there was a composite of the 6 high volume filters taken for each day of grouting for comparison to the background. Finally, what little grout returns came to the surface were evaluated for the presence of total organic compounds and the terbium tracer as a stand-in for plutonium. There were several events that could have adversely affected the contamination control data (meaning terbium above background appearing on the top surface of the thrust block or in the air samplers). These events included a spill of neat grout as the twisted-off plastic sleeve filled with grout in the drill stem that was not drained and several minor drips of the same grout that occurred due to poor draining of the drill steel prior to the “bag-out” procedure.

What follows is a discussion of the results of the contamination control data.

5.5.1 Results of Thrust Block and Drill String Smears

Table 33 below summarizes the ICP-MS evaluation of smears taken before, during and after grouting. The smears were standard 100 cm² swipes of surfaces using a standard fiber smear material. Smears were taken on the top surface of the thrust block, the outside surface of the drill string, the outside surface of the drill string shroud, the outside surface of the inner shroud, and the inside surface of the inner shroud. When a value of the smear is expressed as less than 11.8 ng/smear it means the reading is at the detection limit for that run based on the use of known tracers in the ICP-MS system (see complete data set in Appendix L). Examination of Table 33 shows that the smears taken before grouting on most surfaces showed a ICP-MS reading of “less than 11.8 ng/smear except for one smear that had 16.3 ng/smear on the north edge of the thrust block. Either this was a residual particle of terbium from the pit construction process or a statistical representation of a real background. At this point, it is assumed to be the upper limit of background smears taken prior to grouting. In general the number is no higher than 11.8 ng/g.

The smear taken after grouting hole number 1 showed a similar terbium concentration as the background as expected. This was expected in that the glove sleeves on the drill string and drill string shroud were designed to contain the movement of any tracer. In general, however, after the plastic bag (formed by the twist-off of the plastic thrust block sleeves) filled with grout and fell onto the top surface of the thrust block, the smears taken on the top of the thrust block were elevated in terbium reading relative to the background. The readings for these post spill thrust block smears varied between 14.2 to 35 ng/smear compared to the background values of “less than 11.8 ng/smear.” The spilled material was primarily neat grout; however, material on the outside surface of the thrust block was potentially contained terbium tracer due to the fact that the drill string was driven into the simulated waste containers and could have been immersed in neat grout held back by the bag. On a later chapter discussing the destructive examination of the limited monolith it is shown that a drum containing combustible had been punctured and grouted. Combustible drums contain 100 g of terbium oxide tracer.

Likewise, some of the smears taken on the drill string when drill string shroud no 1 was destructively examined show elevated values (12.2-32.2 ng/smear) as expected since the drill string was driven repeatedly through the simulated waste containing the terbium tracer. However, half of the readings on the used drill string were at background type levels indicating that the drill string wiper material was partially effective in wiping off the mixtures of soil and grout or mud-like material on the outside surface of the drill string.

Smears were taken before and after grouting on the surfaces above the grease seal at the top of the shroud and it was found that even though grease was present after use on this top surface, the after grouting ICP-MS terbium reading was at the background type reading. This is important in that it means the grease seal was working to contain the spread of terbium tracer around the rotating drill string.

It appears that even though the shroud basically separated at the top and was worn through the inner shroud as the drilling proceeded (due to rubbing of the inside surface of the inner shroud on the drill string), the smears taken on the inside surface of the outer shroud and the outside surface of the inner shroud showed only background type readings. However, when examining the inside surface of the inner shroud, the ICP-MS values of the smear was elevated (21.8 ng/smear) relative to background as expected.

In summary, evaluation of the smears show that the shroud/thrust block contamination control system appears to work as designed as long as fluid in the drill string can be drained to disallow filling of the bag formed by twisting off the plastic sleeve containing grout. In addition, even though the inner shroud was worn by rubbing on the drill steel, there was not a spread of contaminants outside the shroud and the in depth contamination control strategy afforded by the thrust block/drill string shroud system worked as planned.

Table 33. Summary of ICP-MS evaluation of smears.

Location of Smear (100 cm ²)	Terbium Concentration (ng/smear)
Thrust block background-north edge	16.3
Thrust block background-center	Less than 11.8
Thrust block background-south edge	Less than 11.8
Drill String Background no 1-entire surface	Less than 11.8
Drill String Background no2-entire surface	Less than 11.8
Drill String Background no 3-entire surface	Less than 11.8
Drill String Shroud Background –Shroud 1	Less than 11.8
Drill String Shroud Background-Shroud 2	Less than 11.8
Drill String Shroud Background-Shroud 3	Less than 11.8
Above Shroud Grease Fitting no 1	Less than 11.8
Above Shroud Grease Fitting no 2	Less than 11.8
Above Shroud Grease Fitting no3	Less than 11.8 plus dup at less than 11.8
On Top Surface of Thrust Block Hole#1	Less than 11.8
On Top Surface of Thrust Block Hole#3(near spill)	21.5
On Top Surface of Thrust Block Hole#4	35.2
On Top Surface of Thrust Block Hole#7	Less than 11.8
On Top Surface of Thrust Block Hole#10	14.2
Inside Surface Outer Shroud (Shroud no 1-shroud used for holes 1-12)	Less than 11.8
Outside Surface Inner Shroud (Shroud no 1)	Less than 11.8
Inside Surface Inner Shroud (Shroud no 1)	21.8
Collected above Shroud no 1 Grease Fitting –contained grease	Less than 11.8
Collected above Shroud no 1 Grease Fitting-second sample	Less than 11.8
Top of Used Drill String no 1 first sample	Less than 11.8
Top of Used Drill String no 1 duplicate of first sample	32.2
Top of Used Drill String no 1 second sample	Less than 11.8
Middle of Drill String no 1 first sample	Less than 11.8
Middle of Drill String no 1 second sample	28.0
Bottom of Drill String no 1 first sample	Less than 11.8
Bottom of Drill String no 1 second sample	Less than 11.8
Bottom of Drill String no 1 third sample	12.2
Water sample from clean-out of first day's operation	0.88 ng/mL

5.5.2 Results of Contaminant Spread to the HEPA Filter

The HEPA filtration system for the thrust block was dismantled and samples of the filter were processed for ICP-MS evaluation for terbium tracer. Table 34 shows the results of this evaluation.

Table 34. ICP-MS evaluation of thrust block HEPA filtration system.

Location in Thrust Block HEPA Filter	Terbium Concentration (micro g/g)
HEPA prefilter 1	0.017
HEPA prefilter 2	0.038
HEPA prefilter 3	0.063
HEPA filter 1	0.177
HEPA filter 2	0.175
HEPA filter 3	0.174

This data set is inconclusive in that there is no established background sample for the ICP-MS; however, the data are presented as a reference for future reference for any follow-on work involving in situ grouting contamination control studies. It is noted that there is a large variation in the HEPA prefiltration in that the third sample shows a factor of almost 5-in. terbium concentration over the first sample. This indicates that most likely terbium tracer had advanced from under the thrust block to the prefilter of the HEPA filtration system. Also, since the three evaluations of the HEPA system are essentially identical, it is most likely that no tracer advanced past the pre filter material. Even though the HEPA filter values are elevated above the pre filter values, this represents a variation in the ICP-MS process for digesting the filter materials in that the prefilter is of different material from the HEPA filter.

5.5.3 Results of Air Monitoring

During the grouting operation air samples were taken using seven strategically located samplers as shown in Figure 42. The filters used in these high-volume air samplers were composited and evaluated for terbium tracer using ICP-MS as one sample. The results were expressed as ng/g of filter per average cu. ft. of air that passed through the seven high-volume samplers. Table 35 presents the results of the ICP-MS evaluation for samples taken during grouting (one set of seven per day) along with 14 background air samples. The average or mean background reading of terbium concentration per air volume for the 14 backgrounds was 0.026 ng/g/cu. ft. of air with an average deviation from that mean of 0.0059 ng/g/cu. ft. of air. This means that the reading during grouting was between 0.015 to 0.0378 ng/g/cu. ft. then there is a 2-sigma or 95% confidence that the reading is at background levels. If the reading during grouting was between 0.02 to 0.032 ng/g/cu. ft. of air than there is a one-sigma or 67% confidence that the reading is at background. Examining Table 35 on Day 1 and Day 2 of grouting (0.015 and 0.012 respectively) there is a 95% confidence that the air monitoring at background levels meaning no spread of contaminants. This is significant in that there was a definite spill of potentially terbium contaminated material on the top of the thrust block during these two days of testing that could have dried and been aerosolized by the continual personnel travel on the top surface of the thrust block. For Day 3 (there was no grouting that day only set-up leading to the accident), the air monitoring reading was 0.021 ng/g/cu. ft., which is below the mean value of 0.026ng/g/cu. ft. The Day 3 value, however, is only within one sigma or 67% confidence that the reading is at background. It is noted that the drill string no. 1 shroud was mechanically disassembled exposing the drill string to the inside the weather structure at the end of the Day 2 grouting and this was a possible source of a higher reading than for Day 1 and 2, during which there was continual personnel traffic inside the weather structure and on the top of the thrust block that was not seen on Day 3.

Table 35. Results of air monitoring for terbium tracer (0.028 cubic meters = 1 cubic foot).

Sample Collection Period	Average Total Air Flow (ft ³)-average of 7 high volume filters	Terbium concentration in the composite filters (Ng)	Terbium concentration weighted by the Air Flow (ng/g- ft ³)
Background Run No. 1-9/13/01	6539	176.4	0.027
Background Run No. 2-9/13/01	6827	172.2	0.025
Background Run No. 3-9/18/01	6970	137.9	0.019
Background Run No. 4-9/18/01	6366	172.7	0.027
Background Run No. 5-9/19/01	7113	137.2	0.019
Background Run No. 5dup-9/19/01	7113	181.4	0.025
Background Run No. 6-9/24/01	7865	197.3	0.025
Background Run No. 7-9/24/01	6712	86.3	0.012
Background Run No. 8-9/25/01	9071	132.2	0.014
Background Run No. 9-9/25/01	6521	188	0.028
Background Run No. 10-9/26/01	9885	194.3	0.019
Background Run No. 11-9/26/01	5407	183.4	0.039
Background Run No. 12-9/27/01	7828	170.0	0.022
Background Run No. 12dup-9/27/01	7828	164.7	0.021
Background Run No. 13-9/27/01	7346	192.6	0.026
Background Run No. 14-10/01/01	8709	170.5	0.019
Sampling First Day of Grouting 10/11/01	15,404	233.6	0.015
Sampling Second Day of Grouting 10/12/01	17820	218.0	0.012
Sampling Third Day of Grouting 10/15/01	8772	191.0	0.021

5.5.4 Results of Contamination in the Grout Returns

Post grouting grab samples of returned grout material in the vicinity of select grout holes where obtained, analyzed by ICP-MS for terbium tracer and compared to a similar analysis for soil samples and a neat grout sample. Determining the terbium tracer content in the grout return samples under the thrust block reflects on the expected amount of contamination that might be expected during a hot operation. Table 36 shows the results of ICP-MS analysis for the common soil samples, neat grout samples and the grout return samples for holes numbered 5,6,7,9,10,11,and 12. These holes displayed some visual evidence of grout returns using the remotely controlled camera and therefore were targeted for analysis.

Table 36. ICP-MS analysis of soil and neat grout backgrounds compared to grout returns under the thrust block.

Sample Location	Terbium concentration (Tb microg/g)
Surface Soil in Weather Structure	0.660
Surface Soil in Weather Structure	0.719
Average Soil in Weather Structure(background)	0.689
Neat Grout Sample 1	0.693
Neat Grout Sample 2	0.676
Neat Grout Sample 3	0.690
Average clean Soil/Grout Samples (Considered Background)	0.687+/-0.013
Grout Return Hole 5(West Side of Hole)	0.686
Grout Return Hole 5 (East Side of Hole)	0.619
Grout Return Hole 5(SW side of Hole)	0.665
Grout Return Hole 6(N side of Hole)	0.673
Grout Return Hole 6(SW side of Hole)	0.665
Grout Return Hole 6(E side of Hole)	0.696
Grout Return Hole 7(E side of Hole)	0.660
Grout Return Hole 7(S side of Hole)	0.706
Grout Return Hole 7(W side of Hole)	0.668
Grout Return Hole 9 (N side of Hole)	0.702
Grout Return Hole 9 (E side of Hole)	0.649
Grout Return Hole 9(SE side of Hole)	0.667
Grout Return Hole 10(N side of Hole)	0.136
Grout Return Hole 10(SW side of Hole)	0.554
Grout Return Hole 10(E side of Hole)	0.696
Grout Return Hole 12(N side of Hole)	0.688
Grout Return Hole 12 (SW side of Hole)	0.808
Grout Return Hole 12 (NW side of Hole)	0.731

The surface soil samples and neat grout samples were averaged to obtain a background of 0.687 microgram terbium per gram of sample (with an average deviation from the mean of 0.013 microgram/g). Table 36 shows that for holes 5,6,7,9,and 10, the readings for the grout return samples were at background meaning no release of the terbium tracer which is located in all containers except the nitrate salt drums. However, the measurement of the terbium content for hole 12 showed elevated levels (levels beyond the average background and average deviation from the average). It is noted that for hole 12 in Table 32 there was mention that a small 15 cm (6 in.) cone of “viscous” returns came to the surface meaning that a simulated waste drum containing the terbium tracer had been hit. Grout returns showing

terbium tracer were expected for all holes except the holes directly over the nitrate salts. Grout returns with terbium tracer was anticipated for those holes over an organic sludge drum (there was maximum terbium tracer and essentially zero voids in the organic sludge material).

The conclusion reached in this data is that the grout returns have the terbium tracer and if more of the pit had been grouted, it is anticipated that more of the returns would have shown tracer. The fact that holes 5,6,7,9, and 10 show no tracer is mainly due to the fact that these first holes are more on the edge of the pit and further grouting would have shown movement of the tracer material to the surface in the form of grout returns.

5.5.5 Comparison of Clean-out to Background Water Samples

Clean water was used to clean the drill steel and originated in a Rain for Rent container. Background water samples from this Rain for Rent system were evaluated for terbium tracer and a comparison was made to a single data point obtained during the grouting operation of a drill stem clean out water. A single sample of the clean-out water was taken during the grouting operation and is reported in Table 33 as 0.00088 micro grams/g (0.88 ng/mL). When comparing the background water samples to this single data point it could be concluded that there was terbium tracer in the clean-out water. However, the extremely small numbers reported in this background and in the clean-out water sample stress the detection limit of the system, and the conclusion is that “no terbium” is present in the clean-out water (see Table 37). To make definitive statements about terbium in the clean-out water would require a better statistic. During the Acid Pit Project (Loomis 98) no contaminants were detected in the clean-out water and a similar procedure was followed.

Table 37. Summarizes the results of the ICP-MS evaluation of clean-out water samples.

Location of Sample	Terbium Concentration (micro g/g)
Rain for Rent Sample no 1	Less than 0.0002
Rain for Rent Sample no 2	Less than 0.0002
Rain for Rent Sample no 3	Less than 0.0002

5.5.6 Discussion of Contamination Control Results

Although only a limited set of contamination control data was obtained due to truncation of the project, it is concluded that the contamination control system was working as planned. Terbium tracer was found in those parts of the system within the “glovebox” of the thrust block/drill string shroud assemblies but not on parts of the system associated with manned entry. The contamination control features of thrust block/drill string shroud concept worked as planned. There was no terbium tracer spread to the high-volume air monitors, even though neat grout with potential terbium contamination was spilled onto the top surface of the thrust block when the sack containing grout drippings fell off the drill string stinger. In fact, inductively coupled plasma-mass spectroscopy of smears taken on the top surface of the thrust block following cleanup of the spill showed terbium contamination. However, even with eventual extensive foot traffic and movement of the drill rig, there was no spread to the high-volume filters. The idea is that the grout locks the tracer material up in larger, less easily aerosolizable particles. It is speculated that if the bag had not dropped, there would only have been terbium tracer within the containment of the drill string shroud and under the negative pressure of the thrust block.

5.6 Destructive Examination of the In Situ Grouting Pit

Using a backhoe with thumb attachment, the limited in situ grouting monolith (October 2002) was excavated, revealing a solid monolith with the usual inclusions of compacted clay soil. The shape of the

monolith followed the shape of the holes grouted, which extended across the 4.5 m (15 ft) of the pit with the width of the monolith generally varying between 40-66 cm (16-26 in.) wide with the exception of the Northwest end of the pit. At this position a metal sided drum was embedded in a monolith and the width was 92 cm (36 in.) including drum and monolith. Based on records obtained during construction of the pit, this drum was determined to be a painted white metal-sided drum containing nitrate salts. The drum embedded in the monolith is shown in Figure 48.

The section exposed was just the top of an 8-ft monolith of grouted waste and represented only grouting in 12 holes (approximately 2 rows of 6 holes each row). To the backhoe operator, the monolith was obvious in that there was a large resistance to digging compared to the surrounding soils. Figure 48 shows the top of the top of the monolith. Figure 49 shows the same region during construction of the pit with the white metal sided nitrate drum in the left corner.

The white metal nitrate drum was removed for further examination and the surrounding grout actually adhered to the drum surface as shown in Figure 50.

Examination of the drum showed that the drum apparently was filled with grout. The drum lid was removed and the face of the contents looked like solid grout. The end was struck with a pick to a depth of about 12.7 cm (5 in.) until the "J" seal of the plastic sack within the drum was revealed completely embedded in what appeared to be neat grout. The end of the drum is shown in Figure 51.

The exterior of the drum was examined and it was determined that the drum had been punctured for only about 22.8 cm (9 in.) on the side of the drum. This puncture is shown on the left side of the drum in Figure 52. The cylindrical shape of the drill steel is shown on this picture. This hole corresponds to hole number 7 in which 393L (104 gal) of grout was injected in 8 ft of grouting. This corresponds to a maximum of about 1 gal of grout delivered for every inch of penetration; therefore, 22.8 cm (9 in.) of grouting in the drum could have involved the placement of about 34L (9 gal) of grout in the drum. It is possible that following grouting, more grout and soil/grout mix gravity fed into remaining voids in the drum.

A hole was made in the metal of the drum in the middle, and the contents were examined. This hole is shown in Figure 53. Also shown in Figure 53 are two holes on either end made by the backhoe during excavation. Contents of the drum observed in the end holes was neat grout and in the middle was a low compressive strength mixture of nitrate salts and grout. This suggested that the grouting action had penetrated to the center positions of the drum, even though the drum had been punctured on the edge.

The actual grout delivery to the interior of the drum had two sources: 1) the limited jet grouting in the maximum of 22.8 cm (9 in.) of travel of the rotating drill stem, and 2) any gravity feed of grout and soil/grout mixture that would have occurred after grouting. Most likely, the two tears in the metal sides showing "neat grout" represent a thin layer in which there were edge voids between the plastic sack and the inside diameter of the drum. Basically, the idea is that grout readily filled the top of the drum with a large void and other voids on the outside of the sack and the interior of the sack with the nitrate represent a very low compressive strength mixture of grout and nitrate salt. Nevertheless, the drum seemed to be embedded in a wall of the mixtures of soil and grout such that the grouted nitrate drum was part of the matrix.

The drum had been weighed prior to building the Cold Test Pit and the recorded weight was 173 kg (381 lb_m). The drum was weighed following excavation and the drum weighed 204 kg (450 lb_m), indicating that a total of 31 kg (69 lb_m) of grout had entered the drum. The grout has a density of approximately 1.8 g/cc (112 lb_m/ft³) or 1.8 kg per liter (15 lb_m per gal). This 31 kg (69 lb_m) extra in the drum accounts for 17.3 L (4.6 gal) of grout delivered to the drum.



Figure 48. Top of monolith showing grouted drum embedded in northwest edge.



Figure 49. Top layer of in situ grouting pit during construction with white nitrate drum in corner.

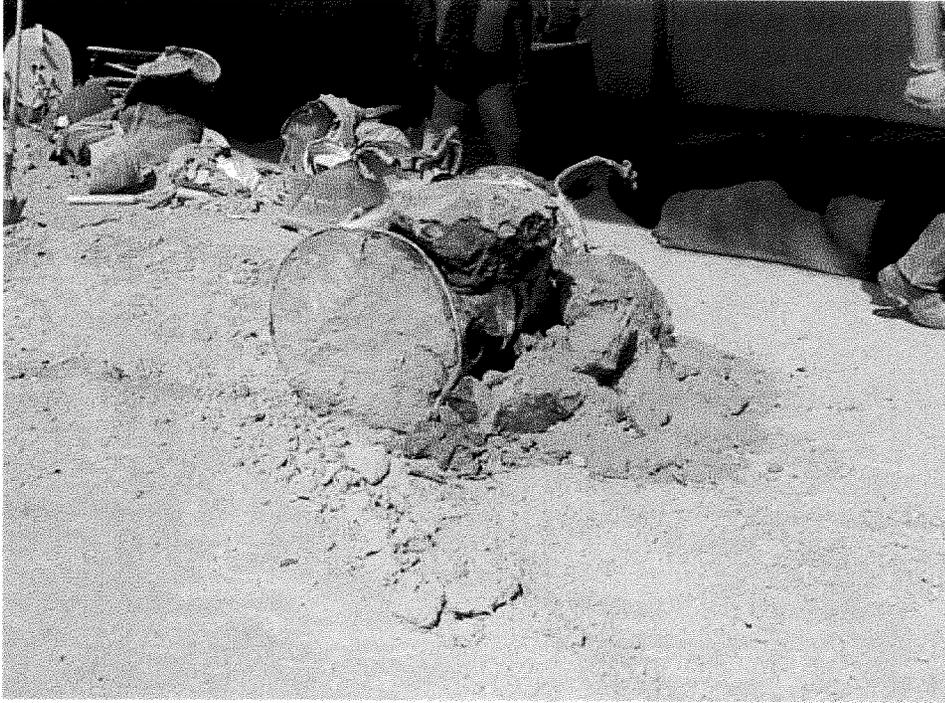


Figure 50. Nitrate drum removed from monolith with grout adhering to the surface of the drum.

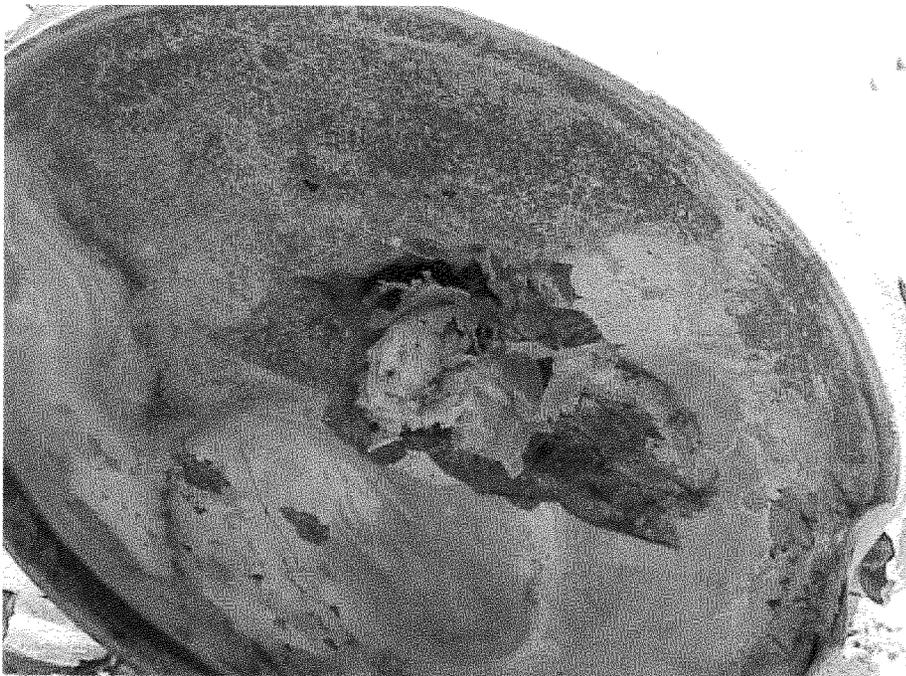


Figure 51. Drum with lid removed showing solid grouted interior with “J” seal visible about 12.7 cm (5 in.) into the top.



Figure 52. Puncture of drum by the drill steel (left-hand side of drum), showing the cylindrical shape of the drill steel.

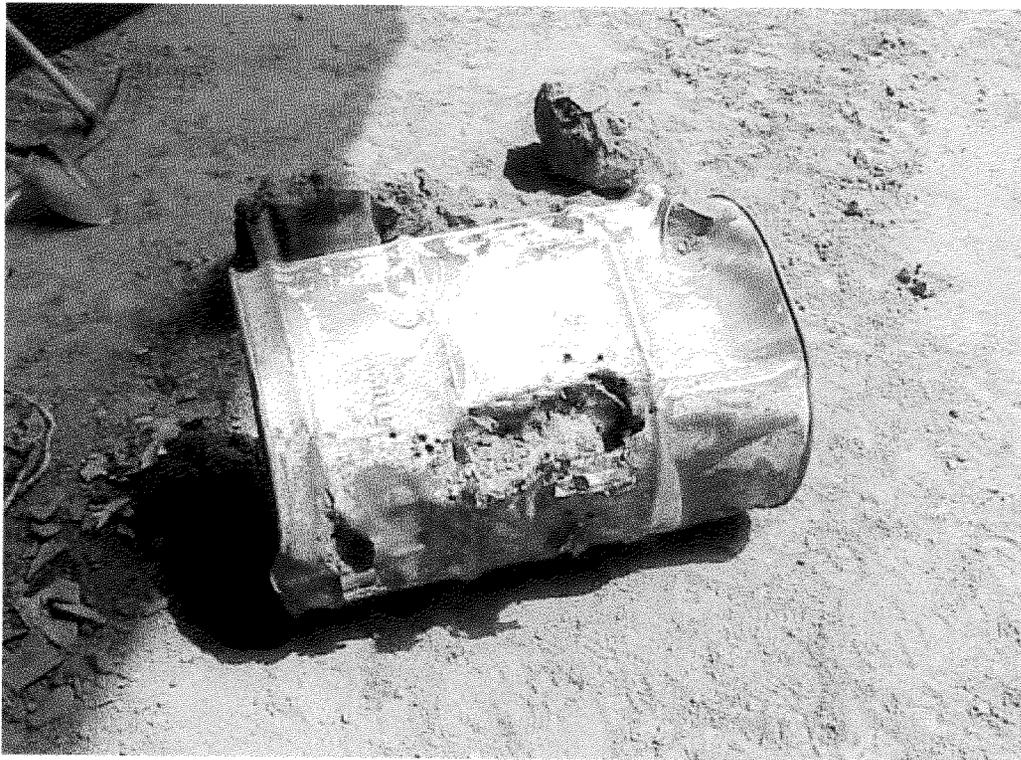


Figure 53. Hole in metal side of drum showing grouted nitrate salts in middle and neat grout on ends.

Accounting for a 5% weighing error, the most that could have been delivered to the drum would be the error ($0.05 \times 450 \text{ lb}_m = 22.5 \text{ lb}_m$) plus the 69 lb_m or 41.5 kg (91.5 lb_m). At 41.5 kg (91.5 lb_m) there could have been at a maximum a total of 23 L (6.1 gal) delivered to the drum which is much lower than the theoretical amount of 34 L (9 gal) delivered based on the 22.8 cm (9 in.) of travel of the drill stem. With a 0.61 m (2 ft) diameter in the drum there is approximately 7.2 L (1.9 gal) of volume per inch of drum height. Therefore, if 23 L (6.1 gal) had been injected into the drum, a linear total of 8.1 cm (3.2 in.) of empty drum length could have been completely filled. This then supports the idea that the grouting action only filled the peripheral edges where there was a large void. If the drum had been struck straight on by the drill stem and further that there was a full 60.9 cm (24 in.) of travel then the amount of grout delivered into the drum would have roughly tripled (as much as 102 L [27 gal]). This action would have certainly filled all voids in the drum and most likely even overflowed a mixture of nitrate salts and grout into voids surrounding the drum.

As the excavation proceeded, two more recognizable waste forms were photographed including a grouted drum containing combustible material and material from the $1.2 \times 1.2 \times 2.4\text{-m}$ ($4 \times 4 \times 8\text{-ft}$) box. Figure 54 shows the grouted combustible drum, and Figure 55 shows the grouted boxed material.

The fact that these two simulated waste containers had grout means that the terbium tracer in the containers could have spread to the top surface of the thrust block and to other positions within the contamination control system.

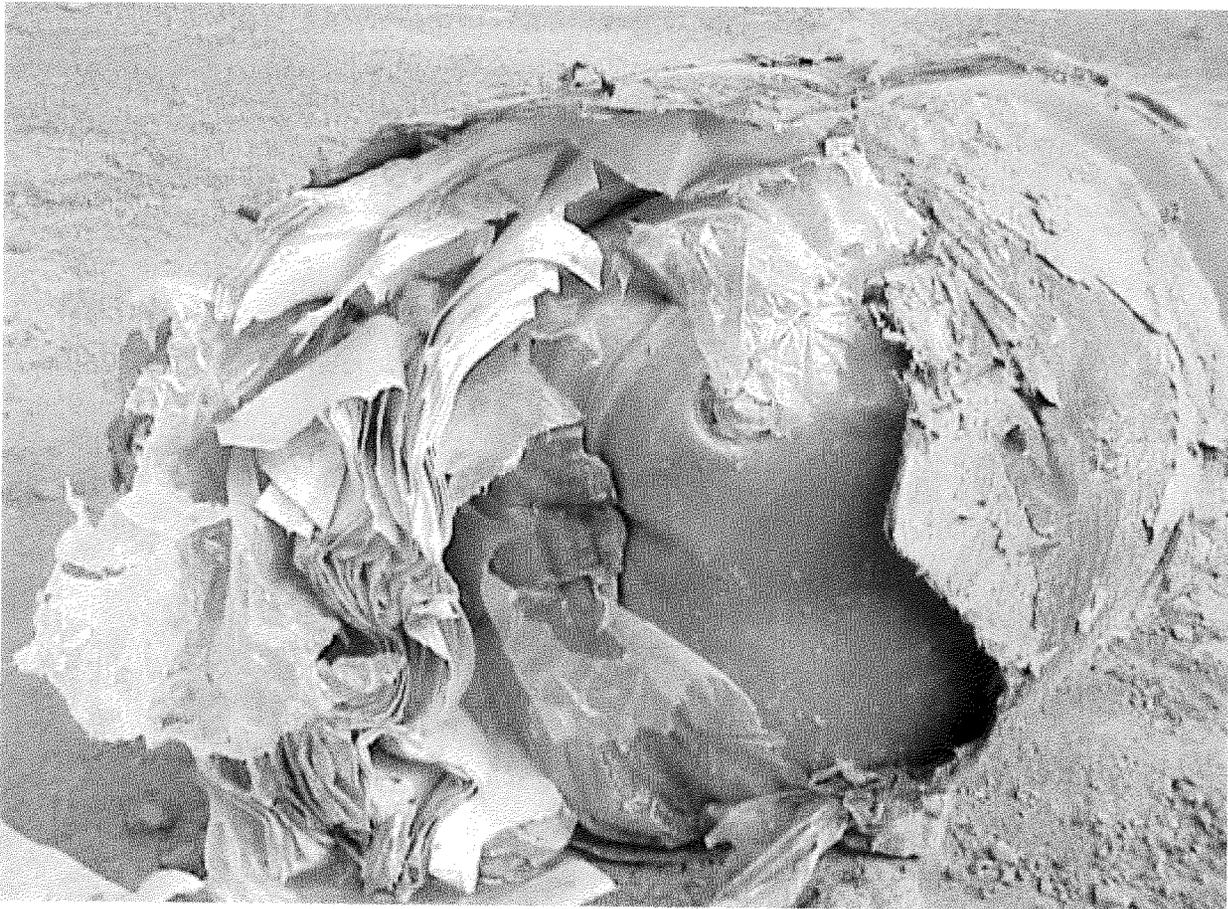


Figure 54. Grouted drum containing combustibles.



Figure 55. Grout in contents of 1.2 x 1.2 x 2.4-m (4 x 4 x 8-ft) box.

6. LESSONS LEARNED

During the limited field demonstration, several lessons were learned. Some of the lessons were related to operations of the system, and others are related to the accident due to the catastrophic failure of a high-pressure fitting.

6.1 General Lessons

Using the thrust block concept requires a complete new design of a vacuum relief system within the drill string. The new design should allow complete automatic draining of the drill string of neat grout prior to moving the drill string to a new hole. Following grouting, simply letting the drill string drain of fluid through the nozzle was not sufficient in that the vacuum created by partially draining the drill string held up fluid. This fluid, once jostled upon moving the system, caused fluid to drain into the bag formed by using duct-tape to tie off the plastic sleeves in the thrust block. In one occurrence, the bag like container formed by the taping process was filled with several liters of neat grout and the bag fell off onto the top surface of the thrust block potentially spilling contaminants. The solution is a high-pressure vacuum relief valve located in the high pressure delivery line. This valve could be remotely operated only at low pressures to open the system allowing complete draining of the drill string.

When the plastic sleeve is installed onto the stinger of the drill string, the process could be simplified by attaching the two bags as if they were one bag rather than attach each bag individually. This action would save operations time in that placing the inner first bag on the stinger is difficult when working within the confines of the second outer bag. The plastic bag material should be a very soft pliable material that doesn't easily stick to metal surfaces and to other similar plastic surfaces.

Prior to restart each day, residual cured grout and other debris should be removed from the drill string by the grouting subcontractor by repositioning all drill strings with shrouds in the vertical position with the sub assembly removed. Water should be flowed through the entire system using the JET-5 pump in the lowest pressure mode while applying rotopercussion to the system to remove all residual cured grout product. This can be done outside the test area at the mouth of the tent. In an actual hot application, this process could be done in a glovebox environment.

As a refinement of the existing drilling sequence, the nozzles should be positioned away from the cameras under the thrust block with a no flow situation, and a trickle flow of grout started with a combination of the low pressure feed system and triplex pump as needed. Only after the trickle flow starts, the nozzle should be placed into the gravel layer and the flow gradually increased as the system is drilled into the pit. In this manner, the cameras can be protected and plugging can be reduced.

It is recommended that the grout plant be located adjacent to the grouting operation to avoid wastage of grout. The output of the plant should be double screened and the supply hopper to the high pressure grouting pumps should be double screened.

If operations are to be performed in the winter months, it is recommended to provide the outside pump personnel and equipment with a temporary weather structure. This would make it easier to avoid ice formation in pumping equipment and improve radio communications between outside pump operators and inside drilling operators. When the air temperature is -1°C (30°F) with a 48-km (30-mph) wind, personal protective gear makes it difficult to use radios.

In an actual hot application, there should be a glovebox adjacent to the drilling area into which the drill string can be inserted. Within the glovebox, the drill string can be extended past the stinger and allow operation access to the nozzles for nozzle unplugging. The bag formed by twisting off the thrust block plastic sleeve would be left in the glovebox (it would automatically fall off into the glovebox when the

drill string was extended past the stinger) and a new “bag” of plastic would be placed on the stinger as the drill string comes out of the glovebox. In this manner, changeout of the entire drill-string shroud assembly would be avoided for something as common as a plugged nozzle.

6.2 Lessons from the Accident

The grouting subcontractor should install a high pressure relief valve and a redundant pressure relief plug system which would allow emergency bleeding of the system. The primary system would be a valve and the secondary system would be a simple plug located in an easily and safely accessed area. This emergency plug should allow safe easy access for wrenches and needed hammers in the event of a system pressurized by nozzle plugging. Once the plug has been forced open in an emergency, it should be replaced with a new plug/and or fitting. In addition, as part of the emergency procedure, the relief system should be cleaned or replaced if used to ensure proper operation.

The grouting subcontractor should check the setting on the automatic shut off feedback switch periodically. This will require a pressurization procedure using water and may require special plumbing to accomplish the testing. The incident at the Cold Test Pit suggests rapid uncontrolled overpressurization by the triplex pump not shown on the gauges. If a nozzle plugs, the operator needs automatic backup help independent of the gauges.

The grouting contractor should use only pressure gauges that operate smoothly at all pressures. It is speculated that the gauge used during the field test sticks at lower pressures and unsticks when the pressure gets higher. For instance the gauge can read 20 bar when it is really at 500 bar about to go to 1,000 bar with a few more strokes of the triplex pump. If not already used, a read-out of the Jean Lutz system may be implemented. It is recommended that there be two gauges, one that is used during low-pressure operations and another that is used during high pressure operations. It may be required that the low-pressure gauge must be valved in to operate at low pressures and valved out when operating at high pressures.

The grouting contractor should use only rated equipment and fittings such as valves, hoses, and tie-downs. The tie-downs and fitting documentation should accompany the fittings for an operating pressure expected of 400 bar (6,000 psi) and safety limits at the design pressure of the pump.

The idea of using 3-mm nozzles for U.S. Grout and GMENT-12 grout should be reevaluated with the new pressure gauges. It is possible that during the nozzle optimization tests at the subcontractor Richland site the pressure gauge showed a pressure of 200 bar (3,000 psi) when in fact the system was pumping at a much higher pressure. Evidence of this was various obvious overpressurization events that occurred when performing the initial nozzle optimization tests performed prior to the implementability testing. What is needed is another separate effects test in soil at the INEEL Cold Test Pit involving improved pressure control and both the 2.4 and the 3-mm nozzles. If the 3-mm nozzle could be used instead of the 2.4-mm nozzle, the potential for nozzle plugging would be greatly reduced. This basically could be accomplished in a 2-day test involving grouting at the end of the week and digging up the columns on Monday following a weekend cure. Using the improved pressure measurement systems, If the system cannot be pressurized to 400 bar with the 3.0-mm nozzles, then, there would be no need to dig up the columns. However, if the system can be pressurized to 400 bar with the 3-mm nozzles, a series of three columns 4 ft high need to be created only varied by the step time. The suggested step times are: 3s, 4s, and 5s.

Because of possible mechanical loads on the Jean Lutz system during the accident, this system should be recalibrated using a barrel filling technique before operations resume.

A blast shield around the outlet to the high-pressure pump is recommended to deflect any future blowout due to catastrophic failure of any fittings in the vicinity of the Jean Lutz and High pressure pump. The system should be attached to protect the tent, the driver, the grout control man and the pump operator. The shield could be designed for easy-on/off attachment.

Although there were many safety issues that required addressing following the accident, the overall technology was working. The main problems involved the long delays due to nozzle plugging. In fact, nozzle plugging could be considered one of the root-causes of the accident. Some slight modifications to the procedures would greatly eliminate the nozzle plugging that was observed including: 1) use of the 3 m nozzles, 2) complete dynamic clean-out of the Applied Geotechnical Engineering and Construction system, 3) double screening of material at the batch plant, and 4) observing a positive trickle flow prior to nozzle insertion into the gravel using the cameras.

7. VENDOR BID ESTIMATE

Applied Geotechnical Engineering and Construction prepared a vendor bid. The grouting vendor was asked to prepare the cost estimate as if it was the company bidding on the job. This company was the grouting contractor on this and past INEEL projects. This bid estimate was prepared using two different approaches to grouting 11 acres in the SDA. The first approach was to use the thrust block or cover block approach as described in the field testing portion of this report. The other technique was for an x-y positional system gantry crane approach as described in Appendix A. For both approaches, the cost estimate did not include the costs of remedial design, Federal Facility Agreement and Consent Order management and oversight, remediation oversight, and remedial design. However, the costs reflected that the contractor set up a permanent operation with a 10-year duration with onsite management. Appendix I gives the details of both of these cost estimates.

To summarize, the gantry crane approach was much lower in total cost over the 10-year period than the cover block or thrust block approach. This is almost entirely due to the cost of the cover blocks relative to the cost of the gantry crane systems. The total cost for the gantry crane approach was \$251M and the cost of the thrust block approach was \$621M. For the thrust block approach, the cost of the thrust blocks were \$283M of the total of \$621M. Both approaches assumed using the same grout (GMENT-12 @ \$2.55/gal for the dry ingredients) and the use of an on site batch plant. Both approaches used 20,000 holes per acre and assumed the same escalation of 3% starting with 2002 dollars. Both approaches used 10% profit and 30% overhead in the estimation. Again, Appendix J gives detailed assumptions and costs for the two approaches.

INEEL cost estimating made a preliminary evaluation of this bid and found the following points:

- the bid did not include a “basis of estimate”
- estimate should be broken down into capital and operation costs
- concrete for footings on page J-6 is inconsistent with spread sheet
- more detail required on cost of thrust blocks (it is a multi-million dollar term and requires more detail)
- units are missing on spread sheet
- not clear whether state and local taxes included.

8. DISCUSSION OF RESULTS

The results presented in Volume 1 of this report represent both detailed bench data as well as full-scale qualitative demonstration of the technology during field applications. In general, there was a continuous down-selection of grouts from a total of six grouts to one grout for field application. Even though only one grout was chosen for the field test, all of the grouts tested had desirable properties for application to in situ grouting. What follows is a discussion of results relative to the bench, implementability, and field testing work.

8.1 Bench Studies

Bench testing resulted in a logical down-selection of grouts from six promising candidates to three grouts recommended for field studies. The three grouts selected showed excellent durability properties (high leach indexes and pH's compatible with INEEL soils), low hydraulic conductivities, high tolerance to interferences, and high strength. The three grouts were: GMENT-12, TECT HG, and U.S. Grout. In a study involving the microencapsulation of volatile organics by the neat grout mixtures, there was an unexpected retardation in the release of the various volatile organics (TCE, TCA, PERC, CCl₄) to a control volume over a period of 90 days of testing. The reference organic sludge mixture released the VOCs immediately to the control volume but when uniformly mixed with the grout, the release was on the order of "hundredths" of a percent of the source term per 10-day testing period. This can translate to a much retarded release of the material. The other three grouts (Waxfix, Enviro-Blend, and Saltstone) displayed properties that require some work to be considered applicable to field use. During testing, it became obvious that certain areas required more work. These areas include 1) suspension of the boron-10 in the Waxfix material, 2) the too early gel time for the Saltstone grout, 3) the poor strength for neat grout and neat grout and interferences, and a too-high hydraulic conductivity for the Enviro-Blend material. Another area that required more work was the unexpected result that the macroencapsulation test displayed a too-high release of Volatile organics relative to the microencapsulation testing for the three grouts recommended for field work. It is suspected that end plug cracking lead to this poor performance.

8.1.1 Boron-10 Suspension in Waxfix

Basically, no physical or chemical testing was performed on the Waxfix grout due to the poor suspension results of the glycerin/sodium tetraborate solution in the curing Waxfix. Instead of the desired 1g/L of boron-10 in the cooled matrix, there was an almost complete settling of the boron material. This was most unfortunate in that Waxfix in prior studies had demonstrated the capability once grouted to penetrate all voids in the buried waste such that the resultant monolith was virtually water-proof and self healing relative to crack formation. The problem of boron suspension could not be solved in time to commit to a complete evaluation of the Waxfix grout; however, this study will an important addition to the Remedial Investigation/Feasibility Study database and should be undertaken as soon as possible. Waxfix has the potential to not only support the concept of grouting for in situ disposal but also has great potential to support cheaper, safer retrieval of buried transuranic waste (see Appendix I). One such concept was developed called RETRIEVABLE DISPOSAL discussed in Appendix I. This concept involves grouting with Waxfix, retrieving the waste, shredding the retrieved material using cryogenics, and then mixing the shredded material with low temperature polyethylene. The resultant material is poured into polyethylene boxes with lifting lugs and disposed of in the pits from which the material was originally retrieved. After this process, the waste is considered disposed but easily retrieved should a better disposal site be determined.

8.1.2 Too Early Gel Time for Saltstone

There were two grouts from the Savannah River Plant that were investigated in this study including Saltstone and Tank Closure grout (reformatted as GMENT-12). The test plan (Grant et al. 2000) called for adjustments to these grouts to make them “jet groutable” in that neither of these materials had been applied in that way. There was only time and budget to “adjust” the formulation of one of the grouts-Tank Closure grout. In fact, the effort to reformulate the base ingredients in this grout were so extensive that the University of Akron subcontractor renamed the grout GMENT-12. Meanwhile the Saltstone grout was tested with minimal reformulation. As a result, the Saltstone grout had an initial gel time of less than 2 hours, which cannot support jet grouting in the field. If an effort were made to adjust the admixture to support jet grouting, it is possible that the Saltstone could be a viable grout.

8.1.3 Poor Performance of Phosphate-Based Grout

It was recognized that including at least one phosphate based grout in the initial list was prudent because of the excellent scavenging properties of the phosphate material for hearing metal contaminants. Evidence of this is in the Phosphate Beds in South Eastern Idaho and the high thorium concentrations. This natural analog geological evidence is useful when convincing regulators about the concept of in situ disposal. To this end, a vendor of a phosphate based strippable paint product used in Rocky Flats remediation was contacted. The vendor was American Minerals, Inc. American Minerals agreed to develop a “grout” material at no cost to the government and supply the material for our testing protocol. The development time was short and the resultant Enviro-Blend grout was developed and tested during the Bench testing. While it displayed the highest Leach Indexes during ANS 16.1 testing, it also displayed the poorest performance for hydraulic conductivity and tolerance to interference materials. It is possible to improve the properties of the neat grouted tested in this program; however, until these properties are improved, the Enviro-Blend grout cannot be recommended for in situ jet grouting.

8.1.4 Cracked End Plug for Macroencapsulation Testing

The results of evaluation for Volatile Organic release in a special sealed volume test using gas chromatography was performed for both micro and macro encapsulation scenarios. The results of the microencapsulation testing (the grout and organic sludge was intimately mixed and allowed to cure) was encouraging in that there was a large unforeseen decrease in the release rate such that the release of VOCs could be retarded for hundreds of years. Macroencapsulation testing involved filling a hollow cylinder made of neat grout with the organic sludge. After sealing the central hollow containing the sludge with a cap made of the grout and further sealing the grout cap with epoxy, the cylinder was placed in the same sealed volume test facility as was used for the microtest. Surprisingly, there was not a marked decreased in the offgas rate of VOCs for the macro compared to the micro testing. Examination of the end plug seal showed a visible cracking which could allow a tortuous but a definite flow path for the VOCs to the chamber air. To perform this experiment correctly would involve accounting for the crack by allowing the hollow cylinder to cure, place the sludge in the cylinder, seal in a cured plug of neat grout, and then, apply various coats of water based epoxy resin coat by coat until the cracks are filled. At this point, the 90-day test could be re-run with the expected result that the VOC release would be hardly measurable in that it was expected that the release would be diffusion controlled.

8.1.5 Durability

The ANS 16.1 leaching protocol provides a conservative durability estimate compared with grout dissolution into the natural ground water because the SDA ground waters are virtually saturated in calcium (with respect to calcite, CaCO_3) and silicon (with respect to chalcedony, microcrystalline SiO_2) whereas the ANS 16.1 leach tests specifies demineralized water, which remains unsaturated. The effect of

composition difference between the pore water and solvent water is illustrated by considering the Fick's law relationship given as $F = A(D_p\Delta C)/\Delta X$ (Kemper 1986), where F is the grout material flux, A is the area, D_p is the diffusion coefficient of material p , ΔX is thickness of the diffusion medium and ΔC is the difference in concentration between the pore water composition and the surrounding ground water composition.

In the case of the SDA, ΔC is virtually zero and the grout material flux would also be virtually zero, indicating that the material loss rates would probably be significantly slower than those used in the computations. In addition, the fact that the ground waters are saturated with calcium and silica means that these materials would probably reprecipitate at the boundary of the waste form. This is borne out by the fact that caliche, natural deposits of calcite, is forming in the SDA soils at the present time. (J. R. Weidner, personal observation, 1991) The data indicate that all the tested grout materials would provide mechanical stability and chemical buffering for thousands of years and easily meet a 1,000-year durability goal (Armstrong 2002).

8.2 Implementability Testing

During implementability testing it was demonstrated with full-scale field equipment that the three grouts recommended from the Bench Testing (TECT HG, U.S. Grout, GMENT-12) could be applied for in situ grouting. All three grouts could be mixed and delivered at 400 bar (6,000 psi) via jet grouting. Two of the grouts (U.S. Grout, GMENT-12) required using a 2.4-mm nozzle and the third grout could pressurize the system using a 3-mm nozzle. The size of the nozzle is important in that the larger the nozzle, the less prone to plugging due to small debris in the system or the effects of filter caking in a stagnant condition (as was required with the thrust block contamination control strategy). Also demonstrated at the implementability testing was the ability to place a 7 cm (2.75 in.) polyethylene rod into the just grouted hole to create, once cured, a borehole for performing hydraulic conductivity tests. In addition, it was further demonstrated that a thermocouple probe consisting of a 1.27 cm (1/2 in.) copper pipe could be inserted for measuring the centerline temperature of the pit. It was demonstrated that the fluid in the drill stem could be drained in a matter of a few minutes; however, what was observed was a stoppage of flow out the nozzles, in that during Field testing it was shown that there was still fluid "held up" by a vacuum that was spilled out the nozzles when tilting the drill stem and moving between holes.

A single grout –GMENT-12- was chosen from the three grouts based on factors such as basic cost, ease of mixing and clean-up of the grout, grout returns in creating a triplex column, and formation of the monolith. It must be mentioned that all three grouts displayed the capability to be jet grouted and form solid stand-alone monoliths in an INEEL type soil condition (tightly packed silty-clay soils). This clay soil condition is thought to be the more restrictive for jet grouting in that there are low voids. For a buried waste case involving soil and debris there is a marked increase in easily accessible void fraction in the debris. U.S. Grout had the lowest specific gravity and therefore displayed the largest amount of grout returns during grouting (when grouting two holes, the space under the simulated thrust block was filled with grout and the third hole could not be grouted). With a lower specific gravity grout, there is not as much kinetic energy imparted to the surrounding medium as with the higher specific gravity grouts, the velocity of the grout being the same. Even though the U.S. Grout displayed a too-high grout return for use on the thrust block concept, this grout would certainly be recommended as a candidate grout for using the x-y positional system discussed in Appendix B.

8.3 Field Testing

Even though an injury accident occurred after successfully grouting only 12 holes, much data on using the thrust block concept and actual data on the capability of the thrust block to contain the terbium

tracer was obtained. The project was not abandoned because of technical/safety issues rather, there was a need for the remaining budget for more pressing INEEL projects at exactly the same time frame as the resultant extensive accident investigation. In short, the remaining budget was needed elsewhere. Completion of the testing would have resulted in, a better statistical approach to evaluating contamination control data, more data on durability of the shroud, knowledge of the hydraulic conductivity of the cured pit, and an extensive evaluation of the monolith and physical and chemical testing of select samples of the monolith during destructive examinations. These monolith samples would have completed an evaluation of the durability of the monolith and supported the data obtained from the Bench testing and the analytical studies on durability found in Volume 2 of this report.

Prior to the accident, there was a learning curve to using the thrust block concept. Since a trickle flow of grout in the nozzles had been utilized on all other grouting at the INEEL (Loomis 95, 97, 98) this test represented the first attempt at grouting without allowing a continuous flow. During implementability testing, the drain of the drill stem was noted to be on the order of minutes and in fact this knowledge was applied for the first two holes. For the first hole, the process worked as planned. When moving from the second hole to the third hole, the sack formed by the twist and tape action of the thrust block sleeve filled with draining grout. Gravity pulled the sack full of fluid off of the stinger and the potentially terbium contaminated neat grout material flowed onto the top surface of the thrust block. This lead to measurable terbium tracer on some of the thrust block smears. This event lead to two actions. One action was to separate the high-pressure hose at the fitting near the weather structure wall and relieve the vacuum in the drill stem (caused by the draining fluid which holds up material in the drill stem). In fact, compressed air was introduced to blow the grout out the nozzles. The other action was to provide a separate bag at the bottom of the sack to help contain any dripping that may occur due to sack filling in the twist-off section. In a hot application, however, it would be desirable to have a special self cleaning relief valve in the system to relieve the vacuum and the possible automatic actuation of compressed air to blow out the remaining grout. Another major issue was the amount of nozzle plugging and time spent using rotopercussion to unstick plugged nozzles. This issue may be related to the grout chosen for the test (GMENT-12). In prior studies using the TECT HG grout there was an allowed trickle flow for most of the grouting; however, there were times when the grout was stopped and start-up was accomplished without much plugging of the nozzles. What was needed in the implementability testing was a separate-effects test to determine which of the grouts displayed the least nozzle plugging in a stopped flow condition. As an alternative to excessive use of the rotopercussion hammer for nozzle clearing during hot application, it is recommended to use a glovebox adjacent to the grouting area with glove ports to allow clearing the nozzles with a wire inside the glovebox.

At the time of the accident, all systems were working as planned with minor modifications required. One modification is the need for a better view of the void space under the thrust block using the remote TV cameras which would involve a deeper Lexan well for the TV camera. Another minor modification would be to provide a hard pipe for the inlet and outlet of the thrust block HEPA filtration system to avoid collapse of the hose. It was obvious that a better weld connection of the shroud to the top bracket was required as well as an engineered twist in the shroud material itself to avoid the rotating drill steel touching the inner shroud.

During the testing, grout was mixed in Idaho Falls at a Ready Mix plant and transported 50 miles to the INEEL Cold Test Pit South three time a day (3024 L [800 gal] per trip). This distance factor lead to a poor utilization of mixed grout in that many loads were dumped unused because of schedule delays in grouting. In order to meet schedule, a batch was prepared based on grouting performance several hours earlier. When the grout actually arrived at the Cold Test Pit, the grouting system may not have been functioning for the entire 2 hours and a full truck of grout was still available. The obvious solution to this problem is to utilize a ready-mix plant at the Cold Test Pit South.

Data quality objectives were listed in the test plans covering the bench, implementability and field testing phases of the treatability study. Most of the data quality objectives discussed in the test plans were addressed by the treatability study; however, there are definite missing gaps in data due to truncation of the field testing program. All of the data quality objectives were met for the bench and implementability testing phases and even with only limited testing in the field testing phase, many of the objectives associated with the field testing involving the thrust block and contamination control system were addressed. Overall, the main data quality objective relating to implementability of the in situ grouting process using the thrust block contamination control system was demonstrated. Only minor design changes are required as discussed above. The overall grouting process is not as rapid on a time per hole basis compared to that expected using alternative grouting concepts (the x-y positional system discussed in the report); however, the thrust block concept process could be applied for a variety of applications in buried waste regions. For instance, the thrust block concept could be used to grout a series of interconnected columns (say 10 hole columns) at various regions within a pit to support a cap and leave the thrust block in place. Another application would be to grout small very specific hot spots within a buried waste region. For this case, the relatively long time to grout a hole would not matter and the complications of using an x-y positional system would not be warranted for such a limited application. The time issue only becomes important when grouting hundreds of thousands of holes over a 10-year period. Finally, to fully evaluate the missing data quality objectives (those relating to the characteristics of the emplaced monolith like void filling, and monolith durability) , would require completion of the grouting in the pit followed by hydraulic conductivity testing and excavation of the monolith with further chemical and physical testing of samples from the resultant monolith.

9. DATA EVALUATION RELATIVE TO DATA QUALITY OBJECTIVES

Because the treatability study was performed under CERCLA guidance, the results of bench, implementability, and field studies are compared to data quality objectives defined in the bench test plan (Grant et al. 2000) and in the implementability and field test plan (Loomis 2000). Table 38 makes that comparison for the bench testing results, and Table 39 provides that comparison for implementability. Table 40 covers field testing. It is noted that, because the field testing was not completed, many data quality objectives were not met.

Table 38. Data quality objectives compared to bench testing results.

Data Quality Objective	Measurement	Discussion of Results
Test Objective 1-Estimate the Durability of the Grouted Waste Monoliths	ANS 16.1 leach testing for Sr, Al, Ca,Si, KNO3	High leach indexes between 10-15 for constituent materials suggests long durability
Test Objective 2 Evaluate the Hydraulic Properties of the Grouted Waste Monoliths	Hydraulic Conductivity	There are a variety of grouts with low hydraulic conductivity on the order of e-9 cm/s suggest essentially no flow through neat grout regions and for the case with soil/nitrate/organic interferences on the order of e-7 cm/s to e-8 cm/s suggest low hydraulic conductivity in a monolith application
	Shrinkage	Screening test results show relatively high shrinkage numbers in the range of 0.25% to 3% as measured as a drop in level in a curing cylinder of neat grout.
	Porosity	Data not taken in that neat grout samples dissipated upon roasting. Testing protocol designed for aggregate concrete.
	Tensile strength	Relative high tensile strength in the range of greater than 600 psi for neat grouts and greater than 200 psi for neat grouts mixed with interferences. Enviro-Blend grout displayed essentially no tensile strength for both neat grout and neat grout mixed with interferences.
Test Objective 3-Identify Grout Material Suitable for Monolith Application	Tensile Strength	See above
	Shrinkage	See above
	Hydraulic conductivity	See above
	Porosity	See above
	Estimate Fracture Development	Fractures observed in macroencapsulation tests in the end plug. Other than this obvious fracture, none observed
	Measure change in test cylinder height	See measurement of shrinkage. The range was a drop in height of 0.25 to3%.
	Measure fracture development; tensile strength	Not directly done by measurement-issue addressed in Volume 2 of this report
	Measure Free water	See height of cylinder discussion

Table 38. (continued).

Data Quality Objective	Measurement	Discussion of Results
	Hydrogen ion activity	PH measured for leachate for the ANS 16.1 testing. System is completely alkaline and moderately oxidizing basic with pH in the range of 10-13 for the leachate water. Compatible with INEEL soil conditions.
	Oxidation Reduction Potential	Measured range in 225-390 mV suggesting moderately oxidizing conditions
	Compressive Strength	Relative high compressive strength for a large variety of grouts for both neat grouts and grouts with interferences. Range of compressive strengths for neat grout 1,500-9,000 psi range and for grouts with interferences 600 to 5,000 psi range.
	Density	Range of density was sg = 1.6 to 2.16 for cementitious grouts tested
	Viscosity	All grouts showed to be jet groutable with Marsh funnel at 56 to 165s.
	Cure time /temperature of cure	Only Saltstone showed a too fast cure time. All other grouts showed initial gel times greater than 2 hours. All grouts had a temperature of set lower than 100°C.
Test Objective 3-Evaluate Chemical Buffering Properties of grouted waste forms	PH and eH measured in ANS 16.1 leachate water.	Demonstrated chemical compatibility with INEEL buried waste soil conditions (see ph/eh discussion above)
Test Objective 4-Determine the effects of soil, organic materials and nitrate salts on grout properties	Compressive strength	All grouts showed high compressive strength (greater than 1,000 psi) with 50 wt% soil/12 wt% nitrate salts, and 9 wt% organic sludge.
	Leach of Sr tracer	High leach indexes relatively unchanged over the case for neat grout (range 10-15)
	Hydraulic conductivity	A large variety of grouts showed only a 1-order-of-magnitude change in hydraulic conductivity with interferences present
	Fracture development/tensile strength	Fracture not observed except in macroencapsulation end plug. Tensile strength remained above 200 psi with interferences
	Free water	Not reported with interferences
	Compressive strength	Compressive strength greater than 1,000 psi for all grouts except Enviro-Blend with the interferences present
Noncritical Objective B-Determine the effectiveness of retaining volatile organic compounds	Microencapsulation testing	Exhibited on the order of "hundredths of a percent" of source term release per 10-day period. Surpassed expectation for VOC entrapment.
	Macroencapsulation testing	Exhibited results similar to the microencapsulation testing ; however, the macro testing was expected to display less release than the microencapsulation testing. Results flawed by presence of

Table 38. (continued).

Data Quality Objective	Measurement	Discussion of Results
		cracks in the end plug
Test Objective 1-Confinement during retrieval-properties of grout based on additives	Sodium tetraborate added to molten paraffin and cooling and separation properties examined	Almost complete separation of the sodium tetraborate during the slow cool down.
Test Objective 2-Confinement during retrieval-Evaluate the combustion hazard of the paraffin based grout	Department of Transportation Oxidizer testing	Not done due to failure of boron suspension results
Test Objective A-Confinement during retrieval-non critical	Btu content	Not done due to failure of boron suspension results.

Table 39. Data quality objectives compared to testing results (implementability).

Data Quality Objective	Measurement	Discussion of Results
Test Objective 6-Evaluate INEEL administrative feasibility for in situ grouting process implementation	Observe pit construction and layout/stage equipment/grout equipment parameter settings/time to grout/total grout returns/heaving/temperature of cure/	Implementability testing performed at vendor site in a timely manner with down-time less than a total of 4 hours. All three grouts (U.S. Grout, TECT HG, and GMENT-12) dry/wet materials were easily shipped to the vendor site. Pit was built to specification and equipment easily arranged to expedite grouting. Key data included time to grout, total grout returns and pit heave. In addition, the temperature of set was easily measured. Basically all data called for in the test plan was obtained.
Test Objective 8-Evaluate field implementability of the grout emplacement process for monolith design and application	Pit construction and layout, position of thrust block, stage equipment, set parameter settings	Silty-Clay soil Pit constructed as per test plan with 3-simulated thrust blocks with 12 in. space for grout returns located on top of pits. Equipment oriented to optimize grouting process and pretest nozzle setting tests recommended U.S. Grout and GMENT-12 should use the 2.4-mm nozzle while the 3-mm nozzle should be used for the TECT HG grout. These nozzles would allow pressurizing the system to 6,000 psi.
	Measure dry/liquid components and mixed amounts of grout during testing	Mixing of grouts simplified to use dry (and some cases wet ingredients) and water in a vortex mixing system. One column batch produced at a time. Some concerns that there was sufficient GMENT-12 in that an excessive amount was used during nozzle optimization testing; however, enough product was left to create a triplex column
	Measure total depth of drill rig and grouted region	Drill stem was inserted 11 ft and the bottom 8 ft was grouted as measured by a mark on the drill string
	Measure parameter settings (injection pressure, step distance, step time, drill string rotation	Injection pressure was always 6,000 psi with the step distance always set at 5 cm. The step time was varied depending upon

Table 39. (continued).

Data Quality Objective	Measurement	Discussion of Results
	rate, total injection volume, nozzle size/total volume of grout returns, heaving, grout physical properties such as density and viscosity	the measured amount of grout that went into the pit. It was attempted to keep the amount of grout injected for each grout the same; however, the injection of U.S. Grout had the volume under the thrust block filled with grout after only two holes. The density of the grout was measured with a mud balance prior to injection and the density was in agreement with the bench values. There was no heaving of the blocks however, in a weakened area for the TECT HG pit there was a grout return outside the thrust block. The weakened are was caused by test holes that had been grouted to set the injection parameters just prior to grouting the triplex column. For both the U.S. Grout and the TECT HG there was remaining space under the thrust block
	Temperature of cure	Measured for each pit less than 100C
	Excavation of columns	All three grouts created stand-alone grout columns
	Rock Quality description	No free water was observed in surround soils nor under thrust block; however, surround soils displayed a wet nature, the monoliths were cohesive enough that all three monoliths could be brought out of the pit in one large piece. Banging on the monolith with a standard backhoe bucket required 10-15 blows from 3 ft to obtain small take-a-way sample.
	Grout Quality(set hardness, impeded curing, free water, fracture development, soil inclusions, mixing,	See above, monolith consisted of cured mixtures of soil and grout with occlusions of clay soil similar to that observed in prior INEEL testing, no fractures could be observed in the monoliths even after removal with the large front-end loader, no incomplete curing was observed.
	Column development(diameter, height, overlap)	Column diameter was nominally 48 in. and 8 ft high for three holes on 20 in. center. No ungrouted regions were observed within the column
Test Objective 3-Identify grout material to support monolith application during in situ grouting	Equipment check-out, time to grout, grout returns, heaving of pit, temperature of cure	GMENT-12 was chosen as the grout to carry to the field testing based on groutability, mixability, monolith formation, and other factors discussed in the main text of this report.
Test Objective 7-Determine contaminant release during in situ grouting	Grout returns	U.S. Grout was eliminated as the choice for field testing on the amount of grout returns which were excessive and would have compromised contamination control systems. U.S. Grout, should be considered as a candidate grout for application of in

Table 39. (continued).

Data Quality Objective	Measurement	Discussion of Results
		situ grouting using the x-y positional system described in this report.
Noncritical test objective D-Determine time, equipment, and labor requirements for mobilization demobilization, and operations	Stage equipment, establish material laydown areas, equipment check-out	Vendor gained experience in mobilizing and demobilizing equipment which was factored into cost estimates made in this report.

Table 40. Data quality objectives compared to test results (field testing).

Data Quality Objective	Measurement	Discussion of Results
Test Objective 1-Estimate durability of grouted monolith	Rock Quality Designation/Water infiltration/monolith grab samples-leach testing etc.(see test plan)	Not obtained-monolith not completed
Test Objective 2-Evaluate hydraulic properties of the monolith	Hydraulic conductivity testing in special wells in the monolith	Not obtained-monolith not completed
Test Objective 4-Evaluate the chemical buffering qualities of the monolith	Measure eh and pH in leachate for ANS 16.1 leach testing	Not obtained-monolith not completed
Test Objective 5-Evaluate the physical stabilization of the waste site to control subsidence	Water infiltration, rock quality	Limited excavation showed solid monolith
Test Objective 6-Evaluate INEEL feasibility for in situ grouting process implementation	Pit construction and layout	Pit constructed in a typical manner to the INEEL SDA transuranic pits and trenches, weather structure installed and thrust blocks and associated cameras, and contamination control equipment installed on pit in the required time
	Stage equipment	Grouting equipment staged to allow safety of INEEL workers relative to the high pressure grouting equipment. All ancillary drill string shrouds laid out to allow easy access, high pressure pump near clean out pit with easy access for grout delivery tanks
	Parameter settings	Initial settings based on Implementability testing, some difficulty keeping the exhaust hoses for the thrust block open due to design issues; however problem not critical as negative pressure was maintained. Camera wells should have been deeper to allow better view of region under the thrust block.
	Time to grout	Grouting was taking too long relative to production rates required to remediate the SDA in a timely manner. Some of the delay is due to the inherent design of the thrust block sleeve system and some of the delay is due to inexperience.(see extensive

Table 40. (continued).

Data Quality Objective	Measurement	Discussion of Results
		discussion of this in the discussion of results section)
	Total volume of grout returns	Minimal grout returns observed in 12 holes grouted under the thrust block
	Heave observed	None observed in 12 holes of grouting
	Temperature of cure	Not obtained-grouting terminated after 12 holes
	Product costs	Grout costs for the test were considerably higher than for an actual application. GMENT-12 should cost \$2.55 per gal of liquid grout in an actual application
	Contamination control monitoring	Smears and air sampling did not interfere with operations
	Volume increase in the monolith	Not obtained-monolith not completed
Test Objective 7-Determine Contaminant release during in situ grouting	100 cm ² smears on top of thrust block	Twisted sleeve fell off drill string shroud when moving from hole 2 to hole 3 spilling potentially terbium-contaminated neat grout on top of the thrust block. Evaluation of samples showed slightly elevated levels on some smears-no airborne release to air monitors surrounding the pit.
Test Objective 8-Evaluate field implementability of the grout emplacement process for monolith design and application	Thrust block foundation	Pea gravel provided a good base for the metal thrust block
	Positioning thrust block	INEEL standard lifting and moving techniques utilized
	Parameter settings	Initial settings for first 12 holes based on implementability testing results and the results of the first few holes. Step time adjusted as need to achieve 60% void filling. This was accomplished using the Jean Lutz flow meter.
	Grouting Process	The grouting process was complicated by use of the glove-box contamination control system involving the thrust block, sleeves, and shrouds on the drill rig; however, the process worked as designed except for a spill of neat grout on the top surface of the thrust block which pointed to several design issues. The first issue is the need to account for a stagnant system between grout holes relative to allowing a trickle flow of grout. Without a trickle flow, the nozzles are prone to plugging using the GMENT-12 grout. The second issue was the need for a drill string drainage system to allow complete drainage of grout between holes. The third was the need for a engineered twist in the

Table 40. (continued).

Data Quality Objective	Measurement	Discussion of Results
		<p>shroud to maintain a space between the rotating drill steel and the inner shroud. Of particular importance was the lesson learned from the accident about the proper use of high pressure fitting, tie-downs, and hoses. Additionally, it was learned to employ automatic pressure relief for the high pressure pump during overpressurization events. Otherwise, the emplacement of grout in a buried transuranic waste environment is completely implementable. The thrust block concept should be particularly useful for small "hot spot" applications.</p>
	<p>Equipment Clean-Out(drill string, subassembly, grout supply system, pumps)</p>	<p>The drill string/shroud assembly removal process took on the order of 1 hour and is completely implementable. Although not demonstrated in the field test, cleaning the used shroud could easily be done in a partial glove-box environment for reuse. It is also anticipated that in actual practice, the plugged nozzles could be cleaned in a small portable glovebox assembly within the weather structure adjacent to the thrust block. It is possible that ice build up in the vortex mixing system could have caused plugging problems. The bottom line is that clean-up using a manifold to attach to the top of the drilling system once the shroud has been removed is completely implementable</p>
	<p>Volume of grout material</p>	<p>Even though the grout was mixed at an Idaho Falls Ready Mix plant 50 miles from the Cold Test Pit South, grout delivery to support the drilling/jet grouting process was accomplished. The grout was mixed in 800-gal batches with density and viscosity measurements essentially the same as in the laboratory bench studies. To avoid wastage of the grout and to allow better coordination between the grout batch plant and the jet grouting operation, it is recommended that the batch plant be located at the scene of jet grouting.</p>
	<p>Total Depth Measurements</p>	<p>Bottom of the pit, Elevation where grouting stopped, easily measured by using a painted mark on the top of the drill rig which gives a relative distance from the top surface of the thrust block. Time to drill recorded in the log books and was accomplished in a matter of minutes. However, there were multiple delays caused by nozzle plugging and time spent in rotopercussion trying to unplug the</p>

Table 40. (continued).

Data Quality Objective	Measurement	Discussion of Results
		nozzles. In addition, there were up to 20 minutes lost in each hole trying to drain the drill stem of grout to disallow any build-up of grout in the plastic sack formed by twisting off the plastic sleeve on the bottom of the drill stem.
	Parameter Settings(injection pressure, step distance, step rate, drill string rotation rate, total injection volume, nozzle size)	<p>The test used GMENT-12 grout which demanded using a 2.4-mm nozzle as per the implementability test.</p> <p>The pressure was as planned 6,000 psi and the step size was 5 cm with the step time varied depending upon the measured amount of grout as measured by the Jean Lutz. The desire was to achieve an overall void filling of 60% of the pit excavated volume; however, for edge holes, a lower amount of grout could be tolerated because of the predominant presence of low void soil. The string rotation rate was to get 2 revolutions of the drill stem per step thus ensuring complete coverage of grout in any 5 cm axial region.</p>
	Grout returns(total volume, per hole)	<p>In the 12 holes grouted during the field test, there were only minor returns observed; however, the cameras worked sufficiently to control the total grout return to a level such that the thrust block void space was not compromised. Therefore, use of the cameras within the thrust block worked as designed and is completely implementable.</p>
	Grout Specifications(viscosity, density, sheer strength)	<p>Field measurements made at the batch plant showed density and viscosity essentially identical as those in the laboratory. Limited Mud balance testing for density showed no change at the Cold Test Pit South after delivery. Initial batch of grout had multiple small debris that could have plugged nozzles but after double screening of the material at the batch plant, this problem was eliminated.</p> <p>The material appeared to be a mouse nest in either the vortex mixer, the “new” grout delivery trucks or in some part of the process at the batch plant. During one day of testing, there were multiple hours delay due to nozzle plugging events and overpressurization events that left the system in a stuck high pressure condition which resulted in the delivered grout going beyond the “pot” life of 4.5Hours. Use of a batch plant at the site of grouting would eliminate this timing problem.</p>

Table 40. (continued).

Data Quality Objective	Measurement	Discussion of Results
	Volume of rinse water	Not measured only 12 holes grouted
	Contamination Control System Evaluation(time to switch shroud assembly, time to apply plastic sleeves from thrust block etc.	The shroud assembly could be removed in approximately 1 hour; however, with practice, this time could be halved. The time to attach the plastic sleeves, grout a hole, allow the system to drain, twist-off the sleeve and cut the twist off and move the rig to a new hole, took nominally 1 hour; however by attaching the sleeves with only one band and using an automatic drain system to keep the nozzles open, this process could be reduced to a 30min time period. This compares to the estimated time to grout using the x-y positional system discussed in the appendix of under 10 min. per hole.
	Contamination Control System (100 cm ² smears of thrust block, drill string, shroud)	Thrust block smears showed elevated levels of terbium tracer following the spill in going from hole 2 to 3. No terbium was found on the outer shroud; however, the drill string and inner shroud showed terbium at the point where the inner shroud wore through due to twisting of the shroud during insertion. Samples above the seal on the shroud showed no terbium tracer indicating that the grease seals worked as designed. If the bag had not fallen on the top surface of the thrust block, there would not have been terbium present.
	Contamination Control(grout returns)	Only 1 in 12 holes showed a grout return with terbium above background (hole 12). This sample was barely above background and standard deviation. Minimal grout returns for the limited testing of 12 holes.
	Contamination Control (backgrounds-air, thrust block, and personnel monitors)	11 backgrounds taken for 7 high volume air samplers and multiple smears taken for top of thrust block, personnel monitors not taken. Adequate backgrounds taken for comparison to assess the implementability from a contamination control standpoint.
	Contamination Control-Air monitoring	During 2 days of grouting covering 12 holes no terbium tracer above background was found in the composited filters from the high-volume samplers suggesting that the in situ grouting process from a contamination spread standpoint is implementable.
	Camera Coverage under the thrust block	Cameras worked well in tracking the grout returns under the thrust block and in determining orientation of the drill string and nozzles during the grouting operation. To increase the view the camera Lexan

Table 40. (continued).

Data Quality Objective	Measurement	Discussion of Results
		well should be lowered to allow a more wide-angle view of all positions in the thrust block. Another possible solution (completely implementable) would be to install more wells in the thrust block to enhance the view.
	Relative humidity, pressure and temperature under the thrust block	Completely implementable and worked as planned. Only problem with the thrust block HEPA filtration system was in setting up the outlet flow flexible hose which collapsed when trying to establish a too low negative pressure; however by correct placement of the flexible hose a slight “hundredths of an inch” negative pressure was maintained during grouting.
	HEPA filter samples	Inconclusive in that there was no established background for HEPA filters. Pre-filler valves were higher than HEPA filler valves. Negative pressure was maintained under the thrust block; however, hose management needs redesign. Negative pressure under the thrust block did not puncture the plastic diaphragms under the thrust block for the ungrouted holes. Personnel monitors not evaluated in that enough holes were not grouted to warrant this action.
	Field-scale testing; tests/measurements postgrouting (volume increase, temperature of cure, excavation of the monolith, rock quality designation)	None made-testing postponed. Recommended that the pit be completed using the x-y positional system and a complete post grout test evaluation be performed as planned in Loomis 2000.
Test Objective 9-Evaluate effects of soil, organic sludge, and nitrate salt on grout properties	Grout/Interference Matrix Interaction-degree of void filling, degree of object bonding, encapsulation vs. permeation, extent of matrix distribution	Not made-testing postponed-recommended for future testing
Test Objective B-non critical-Evaluate effectiveness of grout microencapsulation in retaining VOCs Also Test Objectives 1 2 4 5 9 see above for description.	Grout integrity, set hardness, impeded curing, free water (surface and associated with source containers), Grout/ Interference Matrix Interaction, degree of void filling, degree of object bonding, encapsulation vs. permeation, extent of matrix distribution from source container, source container destruction, source container relocation/movement, extent of multiple source term interaction, soil inclusions mixing, void filling, fracture development, column development, water	Not made-testing postponed-recommended for future testing.

Table 40. (continued).

Data Quality Objective	Measurement	Discussion of Results
	infiltration by U.S. Bureau of Reclamation, monolith grab sample testing to duplicate bench studies (leach, hydraulic conductivity, etc.)	
Test Objective C-Evaluate volume, type and expected disposition of secondary waste	Rinse water evaluation by ICP-MS-qualitative observation of shroud ware	Water evaluation rinse water showed terbium at below detection limits. As described in text, inner shroud was cut by rubbing on drill string; however outer shroud was intact. Complete failure of the weldment of the shroud to the top bracket as described in text requires new type of weldment. Grease fittings appear to have worked in that no contamination above the grease seal in yet terbium contamination was found on the drill string.

In summary, all of the data quality objectives were met for the bench and implementability testing phases. Even with only limited testing in the field testing phase, many of the objectives associated with the field testing involving the thrust block and contamination control system were addressed.

The bench studies produced a data set for a wide variety of grouts that can be used to address the monolith durability questions and expected performance relative to reducing the migration of contaminants from the grouted site. These data quality objectives will be discussed in detail in Volume 2 of this report. By comparing the performance in laboratory studies for neat grouts as well as mixtures of neat grout and expected interferences, it was possible to down-select from six candidate grouts to three grouts for recommendation in the implementability studies. As part of that process, parameters affecting the implementability of those grouts for application in the jet grouting process were measured.

The implementability testing proved that the three candidate grouts could be mixed on site and jet grouted at 6,000 psi. These tests focused on implementability issues such as cleanup, mixing difficulties, grout returns, in situ temperature of set, capability to create a hydraulic conductivity well in the matrix with a polyethylene rod, nozzle plugging and grout pressurization issues. Although the three grouts were found to be implementable from a jet grouting standpoint, the U.S. Grout created a too-high grout return because of the lower density relative to the TECT HG and GMENT-12 grout. In addition, comparison of the mixing and clean-up properties between TECT HG and GMENT-12 along with the fact that monolith formation was similar, the GMENT-12 grout was chosen for the field testing. The fact that the system could be mobilized, configured for jet grouting and monoliths were formed contributed to the conclusion that the whole process was implementable at the INEEL Cold Test Pit South which is a main data quality objective.

Prior to performing the field test, special system check out testing was performed involving the integration of the thrust block/drill string shroud assembly with the jet grouting process. As a result of the special testing, it was concluded that the system could be mobilized and applied at the INEEL. During the field testing many of the objectives on jet grouting implementability in the field were assessed. Several areas were found lacking specifically the need for complete draining of the drill stem prior to moving the system to a new hole. This was simulated in the test by breaking the system at the high-pressure hose as it exited the weather structure; however, it was recognized that a automatic bleed of the system was required. By removing all neat grout in the drill stem, the problems with filling the bag (on the end of the drill stem formed by twisting and cutting the plastic sleeve) with draining grout will be eliminated.

Additionally, by using “hard piped” entrance and exit piping to the thrust block HEPA filtration system would eliminate the problems encountered with collapsing hoses. The shroud on the drill string required an “engineered” twist to avoid the inner shroud from contacting the rotating drill string and thus tearing the material.

In summary, the main data quality objective relating to implementability of the in situ grouting process using the thrust block contamination control system was demonstrated to be practical. Only minor design changes are required as discussed above. The overall grouting process is not as rapid (on a time per hole basis) compared to that expected using the alternative idea of the x-y positional system, which is discussed in the Appendix A of this report. However, the thrust block concept process could be applied for limited hot spots in buried waste regions. For instance, the thrust block concept could be used to grout a series of interconnected columns (say 10 hole columns) at various regions within a pit to support a cap and leave the thrust block in place. Another application would be to grout small very specific hot spots within a buried waste region. For this case, the relatively long time to grout a hole would not matter. The time issue only becomes important when grouting hundreds of thousands of holes over a 10-year period. Finally, to fully evaluate the missing data quality objectives (those relating to the characteristics of the emplaced monolith like void filling, and monolith durability) , would require completion of the grouting in the pit followed by hydraulic conductivity testing and excavation of the monolith with further chemical and physical testing of samples from the resultant monolith.

10. CONCLUSIONS AND RECOMMENDATIONS

The following conclusions are made relative to the in situ grouting technology:

- In situ grouting of buried transuranic waste using the thrust block concept is technically feasible at the INEEL with several modifications to the system. Modifications include providing a nozzle cleanout glovebox adjacent to the grouting area and developing a better pressure relief system to facilitate draining of fluid in the drill stem. In addition use of an additional plastic bag on the end of the taped plastic sleeve would avoid minor dripping of grout when moving the system. By using double screening in the grout preparation phase debris in the grout that could block nozzles can be avoided. Finally, modifications to the shroud assembly that would prevent wear on the inner shroud and disallow detachment at the upper bracket are required.
- Based on the quality of the monoliths formed in simulated buried waste pits during past testing and during the implementability and field testing phase of the current in situ grouting treatability study, it would be expected that the in situ grouting technology can be expected to fill voids in the waste and provide an excellent barrier to subsidence.
- A variety of grouting material are available for application to jet grouting. The current list includes TECT HG, U.S. Grout, and GMENT-12. With minor modifications, the paraffin based Waxfix and the Saltstone grout could most likely also be candidate grout materials. By reformulation of American Minerals, Inc.'s Enviro-Blend grout, it too could be considered a candidate grout.
- Bench studies of U.S. Grout, TECT HG, Enviro-Blend, and GMENT-12 show excellent retention of constituent elements aluminum, silicon, calcium, and the tracer strontium during ANS 16.1 leach testing.
- Bench studies suggest that U.S. Grout, TECT HG, and GMENT-12 show a strong tolerance to interferences commonly occurring within the transuranic buried waste at the INEEL including organic sludge (up to 9 wt% tolerance), soil (up to 50 wt% tolerance) and nitrate salts (up to 12 wt% tolerance).
- Bench studies of volatile organic retention show that there is only a few hundredths of a percent of source term lost per 10-day interval in special microencapsulation testing involving cured mixtures of neat grout and 9 wt% organic sludge (for U.S. Grout, TECT HG, and U.S. Grout).
- The contamination control features of thrust block/drill string shroud concept worked as planned. As expected, there was no terbium tracer spread to the high volume air monitors even though neat grout with potential terbium contamination was spilled onto the top surface of the thrust block when the sack containing grout drippings fell off the drill string stinger. In fact, ICP-MS of smears taken on the top surface of the thrust block following the clean-up of the spill showed terbium contamination; however, even with eventual extensive foot traffic and movement of the drill rig, there was no spread to the high volume filters. The idea is that the grout locks the tracer material up in larger less easily aerosolizable particles. It is speculated that if the bag had not dropped, there would only have been terbium tracer within the containment of the drill string shroud and under the negative pressure of the thrust block.
- Applying the lessons learned from the accident evaluation should ensure that an overpressurization event causing projectile motion of fittings does not happen on future grouting projects. It is not clear whether an ice mass blocked flow at the outlet of the pump and caused a sudden impulse in

pressure leading to the accident or rather, it was normal blockage at the nozzles that led to the overpressurization. By using proper pressure gauges and pump power pressure feedback deactivation technologies and further by using rated fittings, hoses, and whip checks, an accident of the magnitude suffered on the in situ grouting treatability study will not happen again.

- With grouting limited to two rows, a hard stand-alone monolith was created by injecting GMENT-12 grout. Although only a limited excavation was accomplished, the monolith was consistent with the grouting of two rows in the pit. A special nitrate drum with metal sides was embedded in the monolith and this drum was examined in detail. The drum had been penetrated by the drill steel and the voids in the drum had been filled with the grout. The drum was embedded in the monolith and the soil/grout matrix actually stuck to the side of the drum when excavated. All voids in the drum were filled neat grout while the interior appeared to be a low compressive strength mixture of grout and nitrate salts. Other waste forms examined in the pit included a grouted combustible drum in which a large void had been filled with grout and the large waste box at the bottom of the pit had large voids filled by grout.

The following recommendations are made based on the studies of the in situ grouting technology:

- There should be a tradeoff study comparing the thrust block concept and the x-y positional system remote grouting ideas. On paper, the x-y positional system answers all the problems encountered with the thrust block concept. With the x-y positional system, a trickle flow of grout can be allowed and there are no real limitations on grout returns which improves the chances of complete pit void filling (grout returns are allowed and even encouraged to ensure complete void filling). In addition, the x-y positional system has more flexibility when encountering large hard objects that might cause refusal of the drill bit. Finally, a cost comparison of the thrust block testings versus the x-y positional system show an approximate factor of 2.5 savings.
- If the tradeoff study shows that the x-y positional system is effective, then a system should be designed and tested with rare earth tracers in a pit similar to the in situ grouting pit at Cold Test Pit South. This study should focus on the implementability of the grouting delivery system but also should evaluate expected contamination spread if any within the grouting area. In this testing, all data quality objectives associated with monolith formation, hydraulic conductivity, and durability of the monolith should be completed as was planned for the subject in situ grouting treatability study.
- High-pressure jet grouting pumping equipment should include redundant pressure relief systems in the event of a stuck high-pressure event. In addition, to avoid these events (usually caused by nozzle blockage), a low-pressure gauge should be valved in to operate the pump during insertion of the drill string. During grouting, the low pressure gauge should be valved out and the high pressure gauge valved in. It is further recommended that the pumping equipment be located inside a heated weather structure to avoid potential ice build-up inside a pump system. Most importantly any high pressure equipment should be operated within the design range using easy to read gauges calibrated for the range of operation and the system should utilize only fittings, hoses, whip checks, and valves that are rated for the operating pressure expected in this case 400 bar (6,000 psi).
- In future excavations, the concept of using a quarry saw to cut the monolith may be desirable to avoid the collapse of the monolith due to the large stress caused by a backhoe bucket. In addition, use of the quarry saw will eliminate the excessive smearing of loose soil on the monolith that obscures the view.

11. REFERENCES

- Armstrong, A., et al. (2002), *DOE Waste Area Group 7 Operable Unit 7/13/14 Comprehensive Remedial Investigation Feasibility Study*, DOE/ID 10834, Rev. A, Feb 2, 2002, pp. 518; Kemper, W. D. (1986), *Methods of Soil Analysis, Part One Physical and Mineralogical Methods*, Chapter 43, "Soil Diffusivity," American Society of Agronomy, Madison Wisconsin, A. Klute, ed.
- ER-004-02, Logbook for the Field Testing Phase INEEL ER Optical Imaging System
- Grant, R., J. J. Jessmore, G. Loomis, J. Weidner (2000), *Test Plan for the Operable Unit 7-13/14 Bench-Testing In Situ Grouting Treatability Study*, INEEL/EXT-99-00914, Rev. 0, Bechtel BWXT Idaho, LLC, March 2000, 50 pp.
- Loomis, G. G. and Thompson, D. N. 1995. Innovative Grout/Retrieval Demonstration Final Report, INEL-94/001.
- Loomis, G. G., Meyer, L. C., Newton, G. J., Cronenberg, A. W. 1994. Lanthanide Oxides as Surrogates for Plutonium Oxides During Simulated Buried Transuranic Waste Retrieval, WM-94 Tucson, February 27-March 3, 1994:645-648.
- Loomis, G. G., Thompson, D. N., and Heiser, J. H. 1995. Innovative Subsurface Stabilization of Transuranic Pits and Trenches, INEL-95/0632.
- Loomis, G. G., Zdinak, A. P., and Bishop, C. W. 1997. Innovative Subsurface Stabilization Project—Final Report (Revision 1), INEL-96/0439.
- Loomis, G. G., Zdinak, A. P., Ewanic, M. A., and Jessmore, J. J. 1999. Acid Pit Stabilization Project (Vol. 1—Cold Testing, Vol. 2—Hot Testing), INEEL/EXT-98-00009.
- Miller et al., *Operable Unit 7-13/14 In Situ Grouting Treatability Studies Bench-Scale Testing*, INEEL/EXT-02-00851, July 2002.
- Shaw, Peter, July 2000, Surrogate Pits for the Operable Unit 7-13/14 In Situ Grouting and In Situ Vitrification Treatability Studies, INEEL/EXT-2000-00819, Engineering Design File ER-199.
- Thompson, D. N., et al. (1993), "Evaluation of the Contamination Control Unit During Simulated Transuranic Waste Retrieval," EGG-WTD-10973.

Appendix A

Preconceptual Design for In Situ Grouting

Appendix A

Preconceptual Design for In Situ Grouting

Introduction

The concept of creating a solid monolith within the buried transuranic waste by filling void space with grout materials using jet grouting was originated at the INEEL. In a series of EM-50 sponsored research projects, the technology was developed and culminated in a 1997 hot CERCLA treatability study in the INEEL Subsurface Disposal Area Acid Pit. Currently, the technology is part of treatability studies for the INEEL WAG 7-13/14 (INEEL SDA transuranic PITS and TRENCHES). The technology involves drilling into the waste and jet grouting specially formulated grouts at nominally 6,000 psi such that interstitial clay soil is pulverized and incorporated with the grout into the voids in the waste seam. The result is a solid monolith with low hydraulic conductivity and by using special additives to the grouts, a certain degree of chemical fixation of contaminants can be obtained. The grouts considered for application at the INEEL SDA all have natural analogs, which have been shown to be durable for geological times. The past work in INEEL jet grouting has developed a detailed design to mitigate migration of plutonium fines during the grouting process, which involves a complicated thrust block, and drill string shroud assembly. While considered safe and effective, the design is fairly complicated and involves difficult operations. Because of this an alternative idea has been developed at the INEEL involving a more straightforward approach. What follows are preconceptual design features of a novel application of the jet grouting process for creating a final disposal scenario for the INEEL buried transuranic waste.

Design Features

The design involves using a remotely operated bridge crane mounted jet grouting drill string assembly to deliver the grout with total x,y, z control. The overall idea is to create a total monolith out of the waste, side and bottom burdens, and the overburden material. The main departure from the past designs is that some grout returns will be allowed to the surface to facilitate grouting soil side, bottom and over-burden soils. This is accomplished by performing the whole operation in a weather structure with flexible inner liner under negative pressure. While the weather structure is costly, it is relatively straightforward to design and build and allows a very simplified operation of the grouting process. By using a bridge crane mounted system, access to all points within a pit is assured. For instance if a certain hole shows refusal of the drill steel, the bridge crane assembly can position the drill a few inches away and perform the drilling/grouting operation. By suspending the drill system considerably above the top surface, the need to control grout returns diminishes and the risk of overfilling the thrust block used in the original concept is eliminated. What follows are details of the grouting system.

Grouting Rig-Bridge Crane/Concrete Side Walls

Construction of the system would first involve placing a concrete containing wall just outside the boundaries of the waste pit. This concrete wall also acts as a support structure for the bridge crane as shown in Figure 1. Depending upon support requirements this wall could be constructed of driven "H" piles or slurry walls depending upon characterization of the suspected clean sideburden soils. The wall extends above the surface of the overburden and allows an ample space to contain grout returns and to also allow burial of the inner flexible shroud in the weather structure at the completion of grouting. The drill mast and associated hydraulic tubing for rotopercussion drilling and jet grouting are placed on a special platform on the bridge crane that allows exact x,y,z positioning for the sub assembly of the drill

rig. Figure 1 also shows a top view of the weather structure and the relative position of a RadCon support building which allows personnel entry for manned maintenance. The high-pressure injection pump, all hydraulic motors, and associated grout receiving hopper are also shown as being external to the grouting operation.

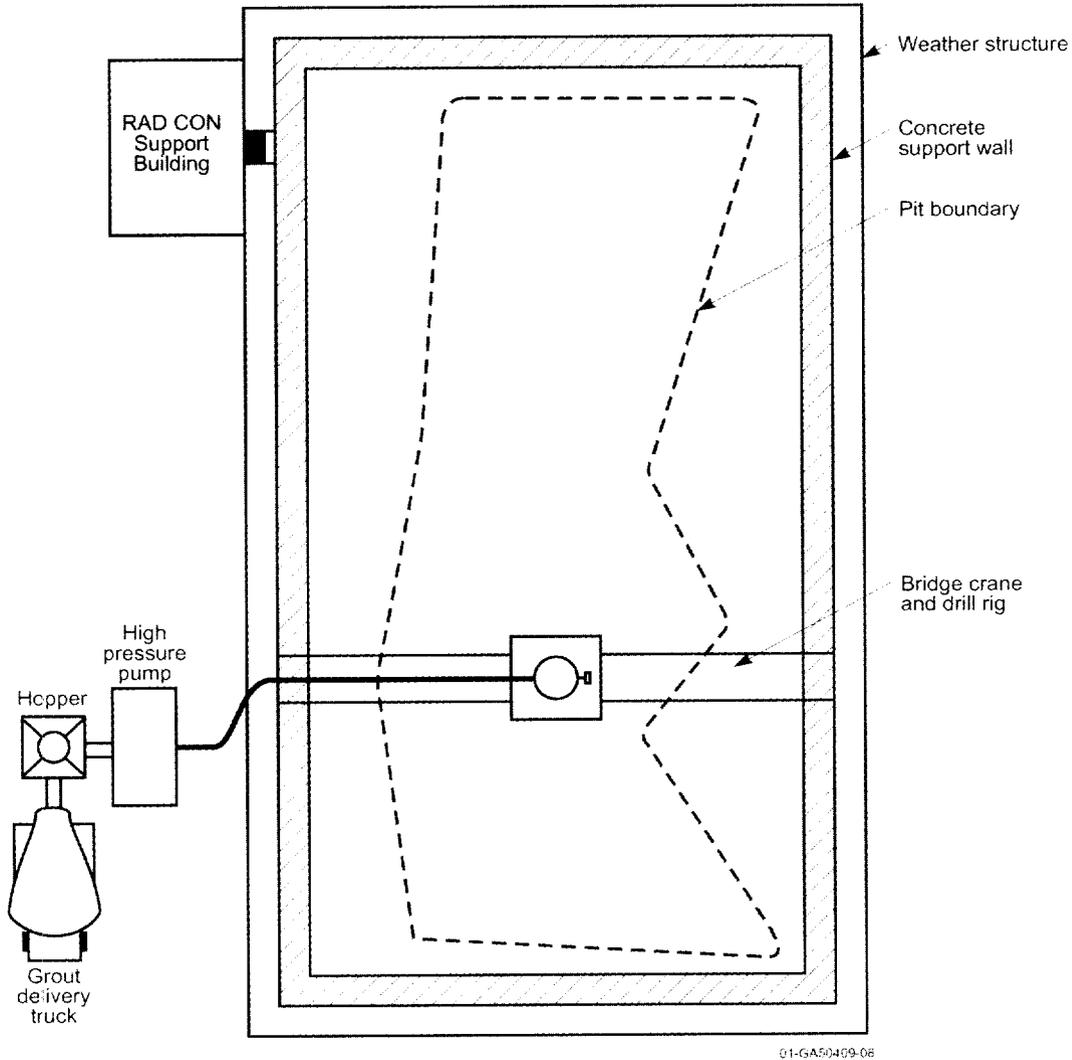


Figure 1. Top View Grouting Process.

Weather Structure/RADCON Building

The weather structure is assumed to be a negative pressure building with a TBD designation relative to status as a DOE nuclear materials handling facility. It is assumed as in all past SDA related projects that this weather structure will be designated and defined through negotiation with the agencies and regulators. Regardless it will be used to house the grouting operation allowing year-long grouting. It

is also assumed that there will be an inner flexible “plastomer” wall that is considered disposable and the outer building is rigid “Butler Building” type of construction. Pit-9-Phase II has developed adequate requirements for such a structure; however, the inner flexible disposable inner sheath would require fire resistance materials and minimum volume for disposal. It is intended that when grouting is completed that the inner sheath is placed in the space at the top of the pit and covered with a final grout cap. Figure 2 shows the conceptual operation in a side view with the HEPA ventilation system and the inner flexible shroud material.

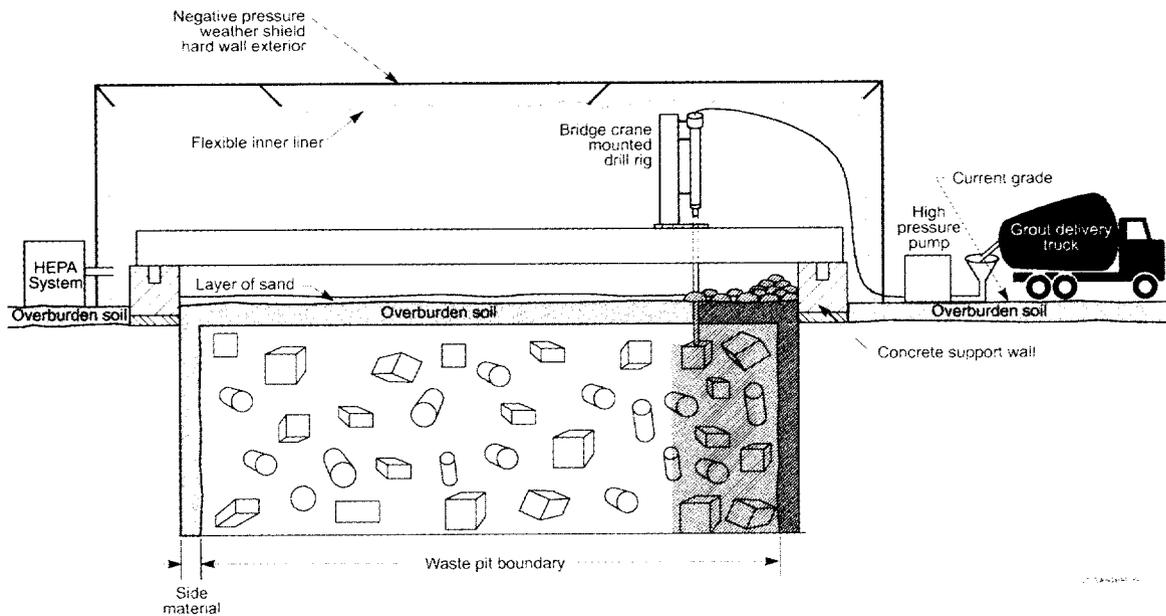


Figure 2. End View Grouting Operation.

This figure also shows how the grouting and hydraulic hoses enter the weather structure. On both Figure 1 and 2, only one drill/grouting assembly is shown on a single bridge crane. However, to expedite operations it may be desirable to have two grouting operations going at the same time or a separate rig in reserve in the event of injection nozzle plugging or other unforeseen events requiring operations shutdown for maintenance.

Details of Grouting

For this grouting concept the main departure from past operations is the inclusion of the top overburden in the grout monolith. To make a solid monolith out of the top overburden would require at least a 35 wt% grout 65 wt% soil mixture and accomplishing this task will create considerable grout returns. From a contamination control standpoint, this should present no problems in that the finely divided plutonium particulate will be incorporated into the liquidous grout/soil material. In addition, the top overburden material is essentially free of contaminants to start the operation. Therefore, grouting the top material is not expected to create a contamination spread problem only a fairly substantial amount of

grout returns which can easily be handled by controlling the space between the top of the overburden and the top of the “H” piles or concrete support walls.

Figure 3 shows details of the grouting operation including a layer of clean sand on top of the overburden to act as a containment for the grout stream as the very top positions of the overburden are grouted jet grouted under high pressure.

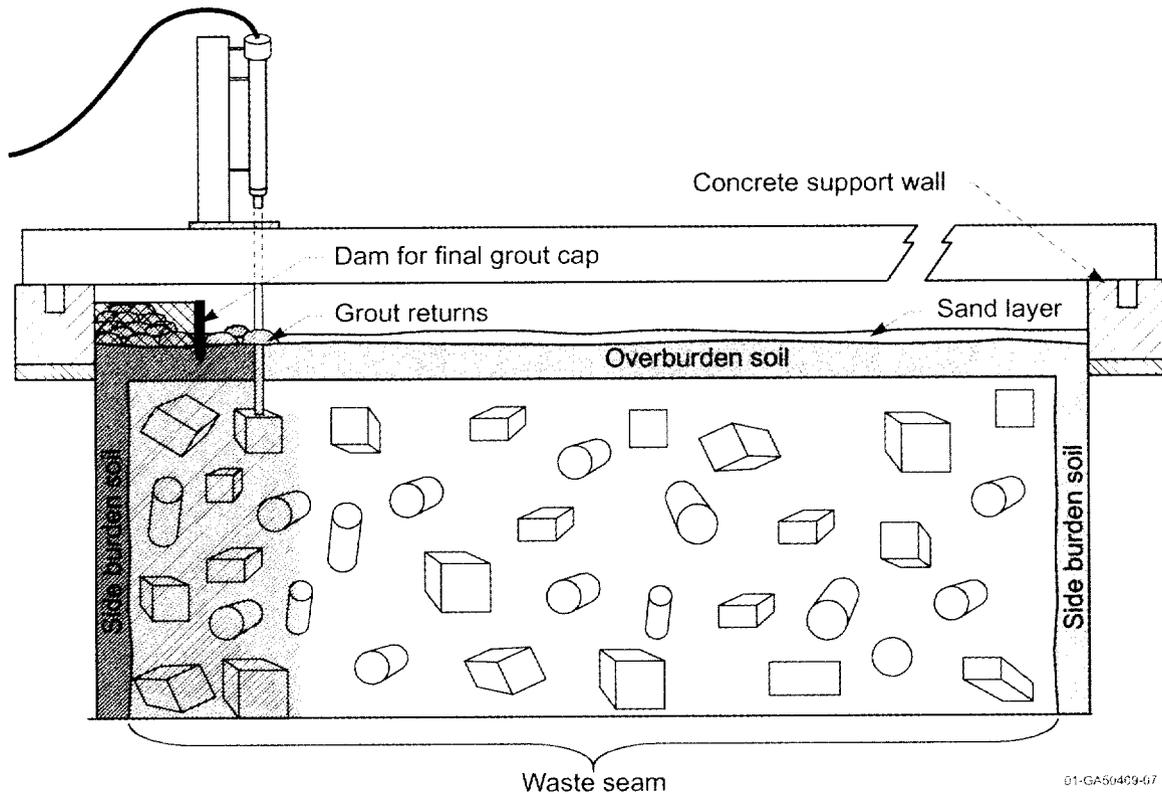


Figure 3. Detail of Grouting Side View.

A sand layer above the overburden allows grouting to extend the monolith to the very top layer using a relatively low pressure (100 psi) without a violent spray associated with 6,000 psi jet grouting. Sand allows easy penetration of the grout and column formation under much lower pressures than that required for the tightly packed silty clay materials in the overburden. During the grouting operation, a region that has been completed can be isolated using a solid cofferdam block to allow partial filling of a just grouted region. By covering the grout returns in these regions with a neat grout, contamination spread via solidification and aerosolization are eliminated allowing a clean inner working area. During grouting operations, it will be initially assumed that maintenance will be performed using manned entry in bubble suits; however, an aggressive filter, smear and grout return sampling campaign will be performed using

radiochemistry to determine loose surface and aerosolized spread of the plutonium oxide particulate which may allow manned entry in less restrictive personnel protective equipment.

Grouting will be accomplished identical to past grouting operations in that the drill stem is driven into the waste and when inserted to refusal in the basalt, the high-pressure pump is started and the rotating drill string is withdrawn in discrete steps. The other grouting variables are the time spent on a step and the number of revolutions of the drill string per step. If refusal is encountered on the way down (encountering heavy metal etc.) the drill string can be withdrawn and moved to several different positions near the refusal hole until penetration can occur. In this manner, “shadowing” effects can be eliminated.

Advantages of this grouting technique are that difficult materials like low-void organic sludges can be thoroughly mixed with grout without fear of excessive grout returns. While the operation will still be monitored with remote TV cameras, the amount of returns are not critical because ample space is provided by using the “wall” concept.

Final Disposal Cap

Following completion of grouting, the inner shroud assembly will be pulled into the remaining space provided by the wall and covered with a final pour of grout. To the extent possible, the drill string assembly will have been decontaminated prior to placing the shroud in the space. It is also possible that the drill string will simply also be disposed in the space provided by the wall prior to a final grout pour. In any case used or plugged drill steel will definitely be disposed of in the final pour. Following the final pour, the entire inner surface of the weather structure should be isolated from the contaminants and the weather structure can be removed for use on the next pit. Once the building has been removed, a final soil freeze cap will be placed to prevent freeze thaw cycles from degrading the monolith as shown in Figure 4.

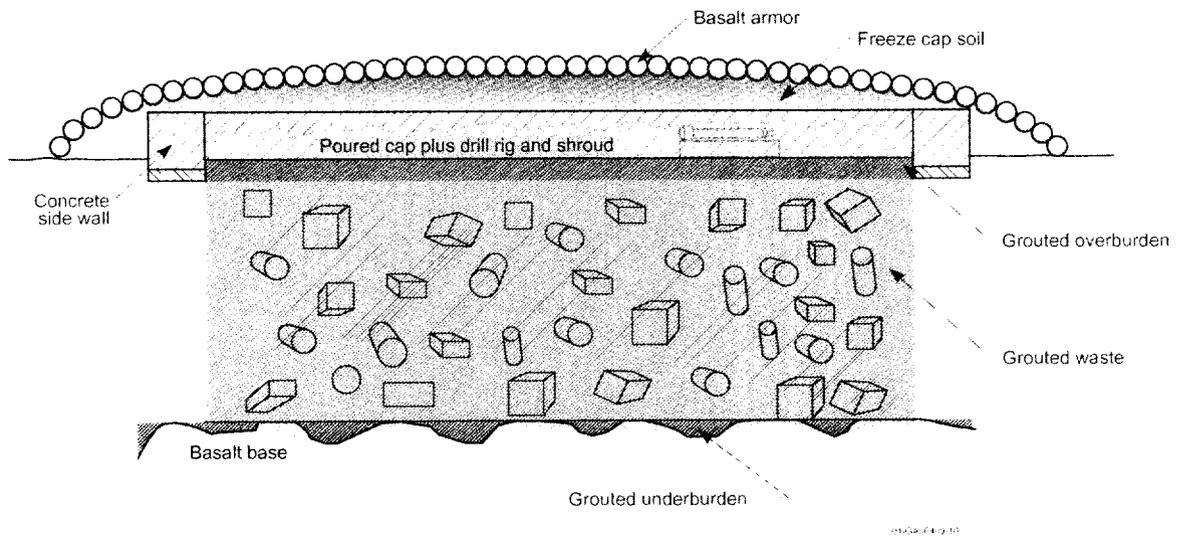


Figure 4. Monolith with Final Cap.

This freeze cap will then be armored with 3-4 ft native basaltic cobble to prevent wind and water erosion of the cap (note: it is assumed that this basaltic cobble cap will be a soil collection zone that will eventually self vegetate with native plants). There will be no special monitoring with an individual pit rather monitoring for migration of contaminants will be part of the overall site-monitoring program.

Performance Standard

A performance standard for this type of operation would be to deliver on a pit wide basis nominally a volume of grout equal to 60% of the volume of the pit. On an average basis, this would ensure complete void filling within the waste seam. An additional performance standard would be to create a grouted overburden/sand region of nominally 35 wt% grout.

Grouting Schedule

It is estimated that the design could support grouting and placing a final cap for 9 acres in 6 years as follows: Using the x-y positional system in the single grout delivery system it is estimated that using a double grouting shift with a back shift for maintenance 64 holes per day can be grouted. It is estimated that grouting on a 20 in. triangular pitch matrix would involve up to 22,000 insertions per acre or about 343 days of operation, which is basically an 18-month operation with contingency. It is assumed that a grout batch plant would be built adjacent to the INEEL SDA and this plant would feed three systems operating simultaneously such that each system would grout 3 –1 acre sites each. It is assumed that the outer weather shield will be dismantled and placed on the next available pit. Allowing down time for moving between sites, the process could be accomplished in a 6-year timeframe including final freeze caps and basaltic cobble installation. This allows for slightly less than a year for initial set up and moving from pit to pit and 1 year for dismantlement of the weather structures and placement of the soil cap and cobble. It is assumed that at least a 2-year period will be required to permit and plan such a task and this is in addition to the 6 years for the actual process.

Preliminary Cost Estimate

- It is estimated that the weather structure system design/fabrication and construction including inner flexible disposable shroud, concrete retaining walls and total management and planning and waste management of the HEPA filtration systems would be \$25M each. Since three weather structures will be used the total cost would be \$75M.
- Grouting systems with bridge crane and controls with special control room in the RADCON building would be on the order of \$3M for each pit (the drilling assembly is considered disposed of within the void created by the wall. However, the system control apparatus can be reused but this is offset by the need for new instrumentation as the project unfolds therefore it is assumed that the full price will be used for each pit. For 9 pits this would cost \$27M.
- Batch Plant-\$5M
- Planning for the whole operation would take 2 years of negotiations with the regulatory agencies and DOE as well as a complete internal design and ES&H RADCON review. This would involve approximately 20 people for 2 years or approximately 40 man-years or \$6M.
- Operations would involve a staff of 30 plus nine shift supervisors x 6 years x \$150,000/person for a total of \$35.1M

- Assuming that the lowest cost grout that made the implementability testing criteria during the current in situ grouting treatability study is used at \$2/gal and further assuming 60% void filling would result in approximately 2M gal/acre x 9 acres x \$2/gal = \$36M.
- Final cap pour would involve 400,000 gal per acre or 3.6M gal per 9 acres @ \$2/gal would be \$7.2M
- Final soil and Basaltic Cover would cost \$5M.

Totals for 9 acres in 6 years:

- Planning/permitting-\$6M
- Weather structure and construction of walls-\$75M
- Grouting Systems-\$27M
- Batch Plant-\$5M
- Operations-\$35.1M
- Grout-\$36M
- Final Cap Pour-\$7.2M
- Final Soil/Basaltic cover-\$5M
- Total for 9 acres=\$196.3M

(If \$5/gal grout is used the total is; \$250.3M and if \$8/gal grout is used the price is \$304.3M.)

Appendix B

Data for Interference Tolerance Testing

Appendix B

Data for Interference Tolerance Testing

Table 1. Individual compressive strength test results in psi for the interference tolerance testing of neat grout specimens and specimens containing the INEEL soil interference at various loadings.

Specimen	Interference Type	Interference Percentage	Grout Product				
			C75	E	S	T	U
			Modified Tank Closure	Enviro-Blend	Salt Stone	TECT HG	U.S. Grout
Specimen A	None		7,502	147	1,619	6,232	2,355
Specimen B	None		8,909	160	1,605	6,378	2,643
Specimen C	None		6,505	142	693	6,349	2,748
Specimen A	INEEL Soil	12	5,734	61	1,407	3,759	3,980
Specimen B	INEEL Soil	12	5,145	59	1,167	4,227	3,803
Specimen C	INEEL Soil	12	6,774	65	1,202	4,464	3,904
Specimen A	INEEL Soil	25	5,876	25	919	3,501	2,995
Specimen B	INEEL Soil	25	5,855	23	933	3,762	3,159
Specimen C	INEEL Soil	25	6,413	29	877	3,698	3,139
Specimen A	INEEL Soil	50	2,722	41	1,351	1,884	1,186
Specimen B	INEEL Soil	50	2,263	45	1,386	1,927	1,421
Specimen C	INEEL Soil	50	2,602	43	1,216	1,962	1,228
Specimen A	INEEL Soil	75			403		757
Specimen B	INEEL Soil	75			382		835
Specimen C	INEEL Soil	75			424		823

Table 4. Individual compressive strength test results in psi for the interference tolerance testing of specimens containing the nitrate salt interference at various loadings.

Specimen	Interference Type	Interference Percentage	Grout Product				
			C75	E	S	T	U
			Modified Tank Closure	Enviro-Blend	Salt Stone	TECT HG	U.S. Grout
Specimen A	Nitrate Salts	12	1,906	36	771	3,224	5,298
Specimen B	Nitrate Salts	12	2,906	43	615	3,254	4,617
Specimen C	Nitrate Salts	12	4,702	37	714		4,490
Specimen A	Nitrate Salts	25	2,948	3	385	1,198	1,306
Specimen B	Nitrate Salts	25	2,298	4	424	1,184	1,420
Specimen C	Nitrate Salts	25	3,408	3	400	1,196	1,423
Specimen A	Nitrate Salts	50	3		2		1,819
Specimen B	Nitrate Salts	50	3		2		1,765
Specimen C	Nitrate Salts	50	2		1		1,857
Specimen A	Nitrate Salts	75	98	12	3		873
Specimen B	Nitrate Salts	75	102	12	3		866
Specimen C	Nitrate Salts	75	113	11	4		868

Table 5. Individual compressive strength test results in psi for the interference tolerance testing of specimens containing the organic sludge interference at various loadings.

Specimen	Interference Type	Interference Percentage	Grout Product				
			C75	E	S	T	U
			Modified Tank Closure	Enviro-Blend	Salt Stone	TECT HG	U.S. Grout
Specimen A	Organic Sludge	3	7,460	128	1,386	4,230	3,202
Specimen B	Organic Sludge	3	6,456	138	1,237	4,266	3,084
Specimen C	Organic Sludge	3	8,131	133	1,202	4,391	3,542
Specimen A	Organic Sludge	5	5,077	136	905	3,764	3,010
Specimen B	Organic Sludge	5	6,788	135	1,117	3,664	2,736
Specimen C	Organic Sludge	5	6,434	125	1,202	3,690	2,887
Specimen A	Organic Sludge	7	6,463	98	1,110	2,805	2,501
Specimen B	Organic Sludge	7	5,897	107	693	2,827	2,746
Specimen C	Organic Sludge	7	6,286	102	1,153	2,828	2,685
Specimen A	Organic Sludge	9	6,123	104	1,054	2,586	3,161
Specimen B	Organic Sludge	9	6,194	105	933	2,650	3,047
Specimen C	Organic Sludge	9	5,932	107	1,075		3,201
Specimen A	Organic Sludge	12		105	955	2,349	
Specimen B	Organic Sludge	12		126	820	2,308	
Specimen C	Organic Sludge	12		118	997	2,383	
Specimen A	Organic Sludge	25			615	204	
Specimen B	Organic Sludge	25			339		
Specimen C	Organic Sludge	25			566		
Specimen A	Organic Sludge	50		53		6	
Specimen B	Organic Sludge	50		44		7	
Specimen C	Organic Sludge	50		58			

Appendix C

Neat Grout ANS 16.1 Individual Sample Data

Appendix C

Neat Grout ANS 16.1 Individual Sample Data

Table 1. U Grout replicate A neat grout American Nuclear Society (ANS) 16.1 data.

Time (d)	Sr (mg/L)	Al (mg/L)	Ca (mg/L)	Si (mg/L)	NO ₃ ⁻ (mg/L)
0.083	0.100	0.334	18.971	0.800	0.180
0.292	0.055	0.320	10.865	0.749	0.235
1.000	0.105	0.602	13.455	1.824	0.255
2.000	0.109	0.589	21.745	2.354	0.310
3.000	0.082	0.472	17.634	2.126	0.265
4.000	0.083	0.484	16.978	2.144	0.280
5.000	0.055	0.394	13.629	2.054	0.230
19.000	0.167	1.853	23.673	11.719	2.090
47.000	0.082	1.558	9.313	13.261	1.590
90.000	0.073	1.331	6.659	15.115	1.110

Time (d)	Sr	Al	Ca	Si	NO ₃ ⁻
0.083	1.38E-10	8.97E-12	1.28E-09	2.74E-11	4.22E-10
0.292	5.48E-11	1.07E-11	5.50E-10	3.13E-11	9.32E-10
1.000	5.97E-11	1.13E-11	2.51E-10	5.57E-11	3.26E-10
2.000	7.87E-11	1.33E-11	8.06E-10	1.13E-10	5.95E-10
3.000	7.59E-11	1.46E-11	8.98E-10	1.57E-10	7.39E-10
4.000	1.09E-10	2.15E-11	1.18E-09	2.24E-10	1.16E-09
5.000	6.18E-11	1.84E-11	9.78E-10	2.67E-10	1.01E-09
19.000	7.05E-12	5.00E-12	3.64E-11	1.07E-10	1.04E-09
47.000	1.23E-12	2.58E-12	4.05E-12	9.90E-11	4.30E-10
90.000	8.78E-13	1.70E-12	1.89E-12	1.16E-10	1.89E-10

Time (d)	PH	eH (mV)
0.083	10.8	368.2
0.292	11.0	183.6
1.000	10.6	176.5
2.000	10.8	217.9
3.000	10.3	213.9
4.000	9.9	227.3
5.000	10.8	187.5
19.000	11.0	128.1
47.000	11.1	380.1
90.000	11.1	389.0

Table 2. U Grout replicate B neat grout ANS 16.1 data.

Time (d)	Sr (mg/L)	Al (mg/L)	Ca (mg/L)	Si (mg/L)	NO ₃ ⁻ (mg/L)
0.083	0.046	0.238	6.622	0.048	0.180
0.292	0.055	0.324	9.998	0.704	0.270
1.000	0.118	0.612	14.661	1.924	0.255
2.000	0.118	0.589	25.172	2.461	0.290
3.000	0.091	0.492	19.872	2.179	0.240
4.000	0.082	0.456	17.230	2.133	0.280
5.000	0.056	0.401	15.345	2.161	0.230
19.000	0.155	1.859	21.558	11.016	1.890
47.000	0.082	1.549	9.683	13.534	1.500
90.000	0.073	1.376	11.107	16.710	1.110

Time (d)	Sr	Al	Ca	Si	NO ₃ ⁻
0.083	2.94E-11	4.58E-12	1.57E-10	9.89E-14	4.22E-10
0.292	5.48E-11	1.10E-11	4.67E-10	2.77E-11	1.23E-09
1.000	7.50E-11	1.17E-11	2.98E-10	6.18E-11	3.26E-10
2.000	9.24E-11	1.33E-11	1.08E-09	1.25E-10	5.22E-10
3.000	9.29E-11	1.58E-11	1.14E-09	1.64E-10	6.09E-10
4.000	1.07E-10	1.92E-11	1.21E-09	2.21E-10	1.16E-09
5.000	6.41E-11	1.90E-11	1.24E-09	2.93E-10	1.01E-09
19.000	6.09E-12	5.07E-12	3.02E-11	9.46E-11	8.41E-10
47.000	1.23E-12	2.54E-12	4.40E-12	1.03E-10	3.85E-10
90.000	8.78E-13	1.79E-12	5.21E-12	1.41E-10	1.89E-10

Time (d)	PH	eH (mV)
0.083	11.1	355.0
0.292	10.9	181.0
1.000	10.8	178.0
2.000	10.9	216.0
3.000	10.3	203.0
4.000	10.8	212.0
5.000	10.8	193.0
19.000	11.2	134.0
47.000	11.1	366.0
90.000	11.1	391.0

Table 3. U Grout replicate C neat grout ANS 16.1 data.

Time (d)	Sr (mg/L)	Al (mg/L)	Ca (mg/L)	Si (mg/L)	NO ₃ ⁻ (mg/L)
0.083	0.036	0.236	7.334	0.129	0.170
0.292	0.055	0.329	11.274	0.746	0.225
1.000	0.100	0.621	13.808	1.869	0.250
2.000	1.092	0.593	25.606	2.556	0.290
3.000	0.082	0.484	19.060	2.132	0.260
4.000	0.046	0.423	5.390	2.031	0.270
5.000	0.065	0.391	13.301	2.123	0.235
19.000	0.155	1.867	25.424	11.452	2.090
47.000	0.091	1.557	14.040	15.135	1.490
90.000	0.082	1.378	10.326	15.808	1.010

Time (d)	De (cm ² /s)				
	Sr	Al	Ca	Si	NO ₃ ⁻
0.083	1.80E-11	4.48E-12	1.93E-10	7.12E-13	3.75E-10
0.292	5.48E-11	1.14E-11	5.91E-10	3.10E-11	8.59E-10
1.000	5.37E-11	1.20E-11	2.64E-10	5.77E-11	3.14E-10
2.000	7.87E-09	1.35E-11	1.12E-09	1.33E-10	5.22E-10
3.000	7.59E-11	1.53E-11	1.05E-09	1.57E-10	7.13E-10
4.000	3.36E-11	1.65E-11	1.18E-10	2.01E-10	1.08E-09
5.000	8.65E-11	1.81E-11	9.30E-10	2.84E-10	1.05E-09
19.000	6.09E-12	5.13E-12	4.20E-11	1.02E-10	1.04E-09
47.000	1.50E-12	2.58E-12	9.26E-12	1.29E-10	3.80E-10
90.000	1.11E-12	1.82E-12	4.49E-12	1.27E-10	1.58E-10

Time (d)	PH	eH (mV)
0.083	10.8	365.0
0.292	10.9	172.0
1.000	10.8	175.0
2.000	10.8	209.0
3.000	10.4	202.0
4.000	10.5	237.0
5.000	10.6	203.0
19.000	11.0	130.0
47.000	11.2	375.0
90.000	11.1	388.0

Table 4. T Grout replicate A neat grout ANS 16.1 data.

Time (d)	Sr (mg/L)	Al (mg/L)	Ca (mg/L)	Si (mg/L)	NO ₃ ⁻ (mg/L)
0.083	0.073	0.028	14.097	0.625	0.019
0.292	0.086	0.051	15.697	0.489	0.019
1.000	0.182	0.174	36.784	1.421	0.028
2.000	0.187	0.220	47.850	1.782	0.038
3.000	0.147	0.186	40.863	1.722	0.038
4.000	0.100	0.184	16.329	1.676	0.029
5.000	0.118	0.209	30.554	1.925	0.019
19.000	0.975	0.611	208.154	3.825	1.010
47.000	0.564	0.639	95.820	4.554	1.010
90.000	0.664	0.757	82.308	4.463	0.820

Time (d)	De (cm ² /s)				
	Sr	Al	Ca	Si	NO ₃ ⁻
0.083	4.33E-11	4.22E-14	4.89E-11	5.04E-12	2.75E-12
0.292	7.82E-11	1.83E-13	7.93E-11	4.01E-12	3.58E-12
1.000	1.04E-10	6.29E-13	1.30E-10	1.01E-11	2.31E-12
2.000	1.35E-10	1.25E-12	2.69E-10	1.96E-11	5.22E-12
3.000	1.43E-10	1.52E-12	3.35E-10	3.10E-11	8.87E-12
4.000	9.26E-11	2.07E-12	7.51E-11	4.14E-11	7.28E-12
5.000	1.66E-10	3.44E-12	3.39E-10	7.02E-11	4.04E-12
19.000	1.40E-10	3.65E-13	1.94E-10	3.41E-12	1.40E-10
47.000	3.40E-11	2.89E-13	2.98E-11	3.52E-12	1.01E-10
90.000	4.23E-11	3.65E-13	1.98E-11	3.05E-12	6.03E-11

Time (d)	PH	eH (mV)
0.083	11.0	329.0
0.292	11.0	146.0
1.000	11.0	121.0
2.000	10.9	154.0
3.000	10.3	158.0
4.000	10.8	166.0
5.000	11.1	131.0
19.000	11.1	66.0
47.000	11.0	367.0
90.000	11.4	346.0

Table 5. T Grout replicate B neat grout ANS 16.1 data.

Time (d)	Sr (mg/L)	Al (mg/L)	Ca (mg/L)	Si (mg/L)	NO ₃ ⁻ (mg/L)
0.083	0.073	0.027	15.806	0.733	0.019
0.292	0.073	0.050	5.825	0.470	0.029
1.000	0.191	0.169	38.283	1.211	0.028
2.000	0.191	0.210	49.536	1.503	0.018
3.000	0.146	0.191	37.910	1.548	0.038
4.000	0.082	0.174	10.897	1.539	0.029
5.000	0.127	0.182	30.473	1.656	0.039
19.000	0.866	0.593	160.838	3.359	0.910
47.000	0.592	0.629	95.092	3.999	1.010
90.000	0.592	0.793	52.029	4.718	0.820

Time (d)	De (cm ² /s)				
	Sr	Al	Ca	Si	NO ₃ ⁻
0.083	4.33E-11	3.91E-14	6.16E-11	6.92E-12	2.75E-12
0.292	5.64E-11	1.76E-13	1.09E-11	3.72E-12	8.31E-12
1.000	1.15E-10	5.97E-13	1.41E-10	7.35E-12	2.31E-12
2.000	1.42E-10	1.13E-12	2.89E-10	1.39E-11	1.17E-12
3.000	1.41E-10	1.59E-12	2.88E-10	2.51E-11	8.87E-12
4.000	6.24E-11	1.85E-12	3.35E-11	3.49E-11	7.28E-12
5.000	1.93E-10	2.63E-12	3.35E-10	5.20E-11	1.69E-11
19.000	1.11E-10	3.45E-13	1.16E-10	2.66E-12	1.14E-10
47.000	3.75E-11	2.80E-13	2.93E-11	2.70E-12	1.01E-10
90.000	3.37E-11	4.01E-13	7.94E-12	3.38E-12	6.03E-11

Time (d)	pH	eH (mV)
0.083	11.0	324.0
0.292	10.5	141.0
1.000	11.0	124.0
2.000	11.1	165.0
3.000	10.6	150.0
4.000	9.6	190.0
5.000	11.1	193.0
19.000	11.1	83.0
47.000	11.1	379.0
90.000	11.4	373.0

Table 6. T Grout replicate C neat grout ANS 16.1 data.

Time (d)	Sr (mg/L)	Al (mg/L)	Ca (mg/L)	Si (mg/L)	NO ₃ - (mg/L)
0.083	0.073	0.023	15.676	0.760	0.019
0.292	0.105	0.059	23.057	0.474	0.009
1.000	0.182	0.164	34.892	1.160	0.028
2.000	0.191	0.201	48.875	1.393	0.028
3.000	0.164	0.219	37.748	1.548	0.038
4.000	0.137	0.200	32.921	1.474	0.029
5.000	0.109	0.183	28.913	1.565	0.028
19.000	0.556	0.582	92.503	3.068	1.010
47.000	0.601	0.630	96.322	3.726	1.110
90.000	0.601	0.730	76.777	4.263	1.020

Time (d)	De (cm ² /s)				
	Sr	Al	Ca	Si	NO ₃ -
0.083	4.330E-11	2.860E-14	6.060E-11	7.440E-12	2.750E-12
0.292	1.170E-10	2.450E-13	1.710E-10	3.770E-12	7.990E-13
1.000	1.040E-10	5.570E-13	1.170E-10	6.750E-12	2.310E-12
2.000	1.420E-10	1.040E-12	2.820E-10	1.200E-11	2.840E-12
3.000	1.770E-10	2.090E-12	2.840E-10	2.510E-11	8.870E-12
4.000	1.730E-10	2.450E-12	3.060E-10	3.200E-11	7.280E-12
5.000	1.420E-10	2.670E-12	3.020E-10	4.660E-11	8.750E-12
19.000	4.560E-11	3.310E-13	3.830E-11	2.210E-12	1.400E-10
47.000	3.850E-11	2.810E-13	3.010E-11	2.350E-12	1.230E-10
90.000	3.470E-11	3.400E-13	1.720E-11	2.770E-12	9.330E-11

Time (d)	pH	eH (mV)
0.083	11.0	322.0
0.292	11.1	129.0
1.000	11.0	133.0
2.000	11.2	145.0
3.000	10.5	143.0
4.000	11.0	153.0
5.000	11.1	126.0
19.000	11.1	83.0
47.000	11.1	380.0
90.000	11.3	361.0

Table 7. E Grout replicate A neat grout ANS 16.1 data.

Time (d)	Sr (mg/L)	Al (mg/L)	Ca (mg/L)	Si (mg/L)	NO ₃ ⁻ (mg/L)
0.083	0.020	0.027	1.103	1.138	0.385
0.292	0.005	0.005	0.759	0.024	0.415
1.000	0.005	0.010	1.358	0.034	0.480
2.000	0.001	0.018	1.190	0.052	0.450
3.000	0.001	0.018	1.386	0.047	0.482
4.000	0.001	0.019	1.289	0.029	0.500
5.000	0.001	0.009	1.459	0.021	0.444
19.000	0.010	0.027	3.362	0.056	2.990
47.000	0.018	0.036	4.967	0.094	4.190
90.000	0.027	0.035	6.276	0.103	2.090

Time (d)	De (cm ² /s)				
	Sr	Al	Ca	Si	NO ₃ ⁻
0.083	4.90E-12	1.31E-13	2.82E-10	1.53E-11	1.69E-09
0.292	3.99E-13	5.88E-15	1.74E-10	8.87E-15	2.56E-09
1.000	1.19E-13	7.05E-15	1.65E-10	5.27E-15	1.02E-09
2.000	5.80E-15	2.79E-14	1.56E-10	1.52E-14	1.10E-09
3.000	9.85E-15	4.74E-14	3.60E-10	2.12E-14	2.15E-09
4.000	1.39E-14	7.46E-14	4.38E-10	1.13E-14	3.25E-09
5.000	1.79E-14	2.15E-14	7.21E-10	7.67E-15	3.30E-09
19.000	2.21E-14	2.39E-15	4.76E-11	6.74E-16	1.85E-09
47.000	5.21E-14	3.07E-15	7.50E-11	1.37E-15	2.63E-09
90.000	1.05E-13	2.62E-15	1.08E-10	1.48E-15	5.92E-10

Time (d)	pH	eH (mV)
0.083	10.2	269.0
0.292	9.6	174.0
1.000	9.8	176.0
2.000	10.3	193.0
3.000	9.8	212.0
4.000	10.8	214.0
5.000	8.7	207.0
19.000	10.2	122.0
47.000	10.7	339.0
90.000	10.7	360.0

Table 8. E Grout replicate B neat grout ANS 16.1 data.

Time (d)	Sr (mg/L)	Al (mg/L)	Ca (mg/L)	Si (mg/L)	NO ₃ ⁻ (mg/L)
0.083	0.010	0.113	0.964	1.150	0.395
0.292	0.005	0.005	0.732	0.023	0.405
1.000	0.005	0.005	1.313	0.022	0.450
2.000	0.001	0.009	1.338	0.046	0.460
3.000	0.010	0.001	1.094	0.037	0.482
4.000	0.010	0.009	1.044	0.048	0.520
5.000	0.100	0.009	0.985	0.003	0.414
19.000	0.010	0.001	3.252	0.030	2.790
47.000	0.009	0.001	4.379	0.029	3.890
90.000	0.018	0.001	5.226	0.029	1.790

Time (d)	De (cm ² /s)				
	Sr	Al	Ca	Si	NO ₃ ⁻
0.083	1.21E-12	2.31E-12	2.15E-10	1.56E-11	1.78E-09
0.292	3.99E-13	5.88E-15	1.61E-10	8.15E-15	2.43E-09
1.000	1.19E-13	1.75E-15	1.55E-10	2.22E-15	8.95E-10
2.000	5.80E-15	6.98E-15	1.97E-10	1.20E-14	1.15E-09
3.000	9.85E-13	1.47E-16	2.26E-10	1.32E-14	2.15E-09
4.000	1.39E-12	1.67E-14	2.86E-10	3.10E-14	3.52E-09
5.000	1.79E-10	2.15E-14	3.30E-10	1.56E-16	2.87E-09
19.000	2.21E-14	3.30E-18	4.44E-11	1.93E-16	1.61E-09
47.000	1.30E-14	2.39E-18	5.84E-11	1.30E-16	2.27E-09
90.000	4.69E-14	2.15E-18	7.48E-11	1.17E-16	4.34E-10

Time (d)	pH	eH (mV)
0.083	10.2	266.0
0.292	9.8	172.0
1.000	9.8	175.0
2.000	10.3	189.0
3.000	9.8	208.0
4.000	10.3	226.0
5.000	10.1	177.0
19.000	10.2	123.0
47.000	10.6	336.0
90.000	10.6	375.0

Table 9. E Grout replicate C neat grout ANS 16.1 data.

Time (d)	Sr (mg/L)	Al (mg/L)	Ca (mg/L)	Si (mg/L)	NO ₃ ⁻ (mg/L)
0.083	0.019	0.007	1.072	1.170	0.400
0.292	0.005	0.009	0.548	0.034	0.412
1.000	0.005	0.010	1.303	0.025	0.460
2.000	0.001	0.072	1.335	0.164	0.450
3.000	0.010	0.010	1.031	0.151	0.498
4.000	0.010	0.010	0.925	0.003	0.500
5.000	0.100	0.019	0.967	0.012	0.444
19.000	0.010	0.001	3.434	0.048	2.890
47.000	0.009	0.001	4.261	0.029	4.090
90.000	0.027	0.009	5.344	0.020	2.090

Time (d)	De (cm ² /s)				
	Sr	Al	Ca	Si	NO ₃ ⁻
0.083	4.39E-12	8.84E-15	2.64E-10	1.62E-11	1.82E-09
0.292	3.99E-13	1.90E-14	9.05E-11	1.77E-14	2.52E-09
1.000	1.19E-13	7.05E-15	1.52E-10	2.86E-15	9.35E-10
2.000	5.80E-15	4.47E-13	1.97E-10	1.52E-13	1.10E-09
3.000	9.85E-13	1.47E-14	2.01E-10	2.18E-13	2.29E-09
4.000	1.39E-12	2.07E-14	2.28E-10	1.21E-16	3.25E-09
5.000	1.79E-10	9.62E-14	3.21E-10	2.50E-15	3.30E-09
19.000	2.21E-14	3.30E-18	4.96E-11	4.94E-16	1.73E-09
47.000	1.30E-14	2.39E-18	5.53E-11	1.30E-16	2.51E-09
90.000	1.05E-13	1.73E-16	7.83E-11	5.59E-17	5.92E-10

Time (d)	pH	eH (mV)
0.083	10.2	276.0
0.292	9.9	172.0
1.000	9.6	185.0
2.000	10.4	145.0
3.000	9.6	210.0
4.000	10.8	202.0
5.000	10.3	171.0
19.000	10.3	123.0
47.000	10.8	332.0
90.000	10.7	378.0

Table 10. C75 Grout replicate A neat grout ANS 16.1 data.

Time (d)	Sr (mg/L)	Al (mg/L)	Ca (mg/L)	Si (mg/L)	NO ₃ ⁻ (mg/L)
0.083	0.040	0.010	2.910	0.070	0.047
0.292	0.020	0.010	2.280	0.020	0.038
1.000	0.110	0.080	7.510	0.450	0.038
2.000	0.110	0.100	11.050	0.600	0.029
3.000	0.120	0.130	12.190	0.820	0.048
4.000	0.100	0.140	12.480	0.850	0.038
5.000	0.130	0.190	14.050	1.320	0.047
19.000	0.910	0.950	68.500	8.300	0.820
47.000	0.640	0.730	43.640	10.020	1.010
90.000	0.500	0.600	30.000	6.020	0.590

Time (d)	De (cm ² /s)				
	Sr	Al	Ca	Si	NO ₃ ⁻
0.083	2.53E-11	1.17E-14	5.59E-12	4.21E-13	3.26E-11
0.292	8.24E-12	1.52E-14	4.46E-12	4.46E-14	2.77E-11
1.000	7.39E-11	2.89E-13	1.45E-11	6.75E-12	8.25E-12
2.000	9.10E-11	5.58E-13	3.87E-11	1.48E-11	5.92E-12
3.000	1.85E-10	1.59E-12	7.98E-11	4.69E-11	2.76E-11
4.000	1.82E-10	2.60E-12	1.18E-10	7.10E-11	2.43E-11
5.000	3.94E-10	6.21E-12	1.93E-10	2.19E-10	4.80E-11
19.000	2.38E-10	1.91E-12	5.65E-11	1.08E-10	1.80E-10
47.000	8.51E-11	8.17E-13	1.66E-11	1.14E-10	1.99E-10
90.000	1.02E-10	1.08E-12	1.54E-11	8.07E-11	1.33E-10

Time (d)	pH	eH (mV)
0.083	10.7	310.0
0.292	11.0	235.0
1.000	10.9	185.0
2.000	11.6	213.0
3.000	11.1	190.0
4.000	11.0	199.0
5.000	10.9	203.0
19.000	10.6	210.0
47.000	10.8	292.0
90.000	10.9	301.0

Table 11. C75 Grout replicate B neat grout ANS 16.1 data.

Time (d)	Sr (mg/L)	Al (mg/L)	Ca (mg/L)	Si (mg/L)	NO ₃ ⁻ (mg/L)
0.083	0.040	0.020	4.200	0.100	0.037
0.292	0.020	0.040	1.490	0.020	0.047
1.000	0.110	0.090	10.270	0.550	0.038
2.000	0.110	0.110	11.470	0.660	0.028
3.000	0.100	0.130	11.960	0.720	0.028
4.000	0.130	0.190	15.290	1.160	0.037
5.000	0.140	0.220	15.200	1.400	0.047
19.000	0.980	0.920	70.350	8.200	0.920
47.000	0.620	0.810	42.630	9.670	1.110
90.000	0.450	0.580	31.120	6.360	0.820

Time (d)	De (cm ² /s)				
	Sr	Al	Ca	Si	NO ₃ ⁻
0.083	2.53E-11	4.67E-14	1.17E-11	8.60E-13	2.02E-11
0.292	8.24E-12	2.43E-13	1.90E-12	4.46E-14	4.25E-11
1.000	7.39E-11	3.66E-13	2.71E-11	1.01E-11	8.25E-12
2.000	9.10E-11	6.74E-13	4.17E-11	1.79E-11	5.53E-12
3.000	1.29E-10	1.59E-12	7.69E-11	3.62E-11	9.39E-12
4.000	3.06E-10	4.82E-12	1.76E-10	1.33E-10	2.30E-11
5.000	4.58E-10	8.30E-12	2.27E-10	2.46E-10	4.80E-11
19.000	2.75E-10	1.79E-12	5.96E-11	1.05E-10	2.25E-10
47.000	8.00E-11	1.00E-12	1.58E-11	1.06E-10	2.39E-10
90.000	8.29E-11	1.01E-12	1.66E-11	9.00E-11	2.57E-10

Time (d)	pH	eH (mV)
0.083	10.7	311.0
0.292	10.7	237.0
1.000	11.2	172.0
2.000	10.5	198.0
3.000	10.9	192.0
4.000	10.4	195.0
5.000	10.8	206.0
19.000	11.2	198.0
47.000	10.9	284.0
90.000	10.9	312.0

Table 12. C75 Grout replicate C neat grout ANS 16.1 data.

Time (d)	Sr (mg/L)	Al (mg/L)	Ca (mg/L)	Si (mg/L)	NO ₃ ⁻ (mg/L)
0.083	0.070	0.020	6.330	0.150	0.047
0.292	0.030	0.010	3.440	0.050	0.038
1.000	0.090	0.050	7.770	0.320	0.047
2.000	0.140	0.130	13.460	0.830	0.028
3.000	0.170	0.200	17.930	1.270	0.028
4.000	0.130	0.170	15.890	1.080	0.038
5.000	0.110	0.140	12.630	1.020	0.047
19.000	0.950	0.940	66.750	7.880	0.820
47.000	0.630	0.760	44.110	9.740	1.010
90.000	0.470	0.550	29.160	6.130	0.910

Time (d)	Sr	Al	Ca	Si	NO ₃ ⁻
0.083	7.75E-11	4.67E-14	2.66E-11	1.93E-12	3.26E-11
0.292	1.86E-11	1.52E-14	1.02E-11	2.81E-13	2.77E-11
1.000	4.99E-11	1.13E-13	1.55E-11	3.41E-12	1.27E-11
2.000	1.49E-10	9.38E-13	5.69E-11	2.83E-11	5.53E-12
3.000	3.71E-10	3.79E-12	1.72E-10	1.12E-10	9.39E-12
4.000	3.06E-10	3.87E-12	1.91E-10	1.15E-10	2.43E-11
5.000	2.80E-10	3.35E-12	1.55E-10	1.32E-10	4.80E-11
19.000	5.19E-10	3.72E-12	1.07E-10	1.93E-10	3.59E-10
47.000	8.26E-11	8.85E-13	1.70E-11	1.07E-10	1.99E-10
90.000	9.06E-11	9.13E-13	1.46E-11	8.39E-11	3.14E-10

Time (d)	pH	eH (mV)
0.083	10.5	313.0
0.292	10.9	229.0
1.000	10.8	180.0
2.000	10.3	200.0
3.000	10.9	186.0
4.000	10.7	189.0
5.000	11.0	214.0
19.000	10.6	223.0
47.000	10.6	262.0
90.000	10.8	296.0

Table 13. S Grout replicate A neat grout ANS 16.1 data.

Time (d)	Sr (mg/L)	Al (mg/L)	Ca (mg/L)	Si (mg/L)	NO ₃ ⁻ (mg/L)
0.083	0.010	0.040	0.060	0.910	0.038
0.292	0.160	0.120	2.720	0.220	0.028
1.000	0.170	0.340	10.260	1.090	0.038
2.000	0.110	0.200	6.910	0.720	0.028
3.000	0.160	0.540	12.400	1.980	0.019
4.000	0.100	0.380	10.390	1.630	0.028
5.000	0.090	0.290	8.470	1.450	0.010
19.000	0.680	1.510	46.430	7.630	0.820
47.000	0.340	1.140	13.110	7.480	1.400
90.000	0.170	0.590	7.500	4.400	0.720

Time (d)	De (cm ² /s)				
	Sr	Al	Ca	Si	NO ₃ ⁻
0.083	1.14E-12	2.40E-14	6.66E-15	1.21E-10	1.56E-11
0.292	3.87E-10	2.81E-13	1.79E-11	9.32E-12	1.10E-11
1.000	1.30E-10	6.72E-13	7.50E-11	6.83E-11	6.04E-12
2.000	6.62E-11	2.86E-13	4.21E-11	3.65E-11	4.04E-12
3.000	2.41E-10	3.53E-12	2.32E-10	4.70E-10	3.14E-12
4.000	1.31E-10	2.45E-12	2.28E-10	4.47E-10	9.67E-12
5.000	1.38E-10	1.86E-12	1.96E-10	4.53E-10	1.58E-12
19.000	9.71E-11	6.21E-13	7.25E-11	1.56E-10	1.32E-10
47.000	1.75E-11	2.55E-13	4.20E-12	1.08E-10	2.78E-10
90.000	8.66E-12	1.34E-13	2.69E-12	7.38E-11	1.44E-10

Time (d)	pH	eH (mV)
0.083	10.4	300.0
0.292	10.6	222.0
1.000	10.1	202.0
2.000	10.3	203.0
3.000	11.0	200.0
4.000	9.8	205.0
5.000	10.5	212.0
19.000	10.8	226.0
47.000	10.5	205.0
90.000	10.9	224.0

Table 14. S Grout replicate B neat grout ANS 16.1 data.

Time (d)	Sr (mg/L)	Al (mg/L)	Ca (mg/L)	Si (mg/L)	NO ₃ ⁻ (mg/L)
0.083	0.060	0.030	2.850	0.120	0.038
0.292	0.020	0.020	1.290	0.030	0.028
1.000	0.110	0.011	6.100	1.090	0.037
2.000	0.130	0.140	8.250	0.640	0.028
3.000	0.200	0.440	14.550	1.930	0.019
4.000	0.120	0.250	10.470	1.160	0.028
5.000	0.090	0.200	7.450	1.030	0.010
19.000	0.690	1.150	41.390	7.740	1.020
47.000	0.340	1.230	12.100	6.960	1.590
90.000	0.180	0.570	7.600	4.500	0.820

Time (d)	De (cm ² /s)				
	Sr	Al	Ca	Si	NO ₃ ⁻
0.083	4.16E-11	1.33E-14	1.50E-11	2.12E-12	1.56E-11
0.292	6.03E-12	7.80E-15	4.01E-12	1.72E-13	1.10E-11
1.000	5.37E-11	7.00E-16	2.67E-11	6.83E-11	5.72E-12
2.000	9.38E-11	1.40E-13	6.03E-11	2.88E-11	4.04E-12
3.000	3.75E-10	2.35E-12	3.18E-10	4.45E-10	3.14E-12
4.000	1.91E-10	1.07E-12	2.31E-10	2.28E-10	9.67E-12
5.000	1.38E-10	8.80E-13	1.51E-10	2.30E-10	1.58E-12
19.000	1.00E-10	3.60E-13	5.76E-11	1.60E-10	2.04E-10
47.000	1.75E-11	2.97E-13	3.57E-12	9.37E-11	3.57E-10
90.000	9.71E-12	1.26E-13	2.77E-12	7.71E-11	1.87E-10

Time (d)	pH	eH (mV)
0.083	10.2	301.0
0.292	11.0	205.0
1.000	10.0	228.0
2.000	10.1	198.0
3.000	10.5	210.0
4.000	10.0	201.0
5.000	10.7	199.0
19.000	10.9	203.0
47.000	10.6	223.0
90.000	10.8	232.0

Table 15. S Grout replicate C neat grout ANS 16.1 data.

Time (d)	Sr (mg/L)	Al (mg/L)	Ca (mg/L)	Si (mg/L)	NO ₃ ⁻ (mg/L)
0.083	0.080	0.060	3.360	0.190	0.037
0.292	0.060	0.010	1.660	0.040	0.028
1.000	0.140	0.340	7.510	0.460	0.038
2.000	0.250	0.440	15.740	1.830	0.028
3.000	0.130	0.220	9.710	1.020	0.019
4.000	0.150	0.380	12.580	1.790	0.028
5.000	0.130	0.340	10.460	1.640	0.019
19.000	0.680	1.440	46.140	7.580	1.020
47.000	0.330	1.240	11.960	7.070	1.500
90.000	0.160	0.560	7.400	4.600	0.820

Time (d)	De (cm ² /s)				
	Sr	Al	Ca	Si	NO ₃ ⁻
0.083	7.38E-11	5.39E-14	2.09E-11	5.34E-12	1.47E-11
0.292	5.42E-11	1.94E-15	6.64E-12	3.07E-13	1.10E-11
1.000	8.83E-11	6.72E-13	4.05E-11	1.21E-11	6.04E-12
2.000	3.44E-10	1.38E-12	2.19E-10	2.37E-10	4.04E-12
3.000	1.59E-10	5.87E-13	1.41E-10	1.25E-10	3.14E-12
4.000	2.98E-10	2.45E-12	3.34E-10	5.38E-10	9.67E-12
5.000	2.89E-10	2.55E-12	2.98E-10	5.82E-10	5.70E-12
19.000	9.71E-11	5.63E-13	7.17E-11	1.54E-10	2.04E-10
47.000	1.65E-11	3.02E-13	3.48E-12	9.68E-11	3.16E-10
90.000	7.68E-12	1.21E-13	2.62E-12	8.07E-11	1.87E-10

Time (d)	pH	eH (mV)
0.083	10.6	297.0
0.292	10.1	216.0
1.000	9.8	232.0
2.000	10.2	192.0
3.000	10.9	206.0
4.000	9.5	195.0
5.000	10.2	211.0
19.000	11.0	207.0
47.000	10.6	197.0
90.000	10.9	211.0

Appendix D

Cement Chemistry and Durability

Appendix D

Cement Chemistry and Durability

Introduction

The purpose of this review is to describe the chemical properties of cementitious grout systems, discuss their expected change with time, and use this information to estimate the solubility limits of contaminants of potential concern found in the Idaho National Engineering and Environmental Laboratory (INEEL) Subsurface Disposal Area (SDA).

The in situ grouting technology is a method to stabilize and encapsulate buried waste such as that found at the SDA. Many different grout materials may be used for this application and may have a very broad range of compositions and properties. Examples include grout materials based on silicone, or phosphate, or iron oxide-sulfate, or paraffin or others. The specific grout material would be selected to meet the requirements of a specific application. Cementitious grout materials and their derivatives are discussed in the following paragraphs. They include a very broad range of materials having a very broad range of properties. They share the common characteristic of belonging within the same chemical family as the well known Portland cements and they are often a derivative of one of the Portland cements. The in situ grouting application mixes the anhydrous cementitious grout material with water and injects this mixture into the waste site at high pressure. The result is a hydrous grout material in intimate contact with the waste materials and whose chemical properties may affect the buried waste components.

The chemical properties of the grout material may affect, and be affected, by the chemical properties of the waste site ground water and waste materials. The acid-base character (pH) and oxidation-reduction potential (eH) are two chemical properties, which are particularly important for estimating the behavior of grout materials in the waste site chemical environment. Changes in pH and/or eH can affect the dissolution/precipitation of mineral material and the dissolution/evolution of gasses and also the adsorption/desorption of aqueous species.

PH is defined as the negative logarithm of the hydrogen ion activity and is a measure of the acid versus base properties of an aqueous system. The pH can affect the solubility of the grout and waste materials by altering the chemical speciation of a particular material in aqueous solution. The eH is the electrical potential required for moving electron(s) between oxidized and reduced species in an aqueous solution and is expressed in volts. eH is important for estimating the behavior of elements, which can exist in more than one oxidation state, such as technetium, chromium, plutonium, neptunium, and americium. Elements such as technetium and chromium are very insoluble in reducing conditions, but become very soluble in a more oxidized environment. Some elements can exist in as many as four oxidation states. Each oxidation state has a different solubility because the oxidation state (and pH) affects the speciation of the element in aqueous solution.

Chemical Properties of Cement

Cement grout is an engineered material, which usually has an anhydrous bulk composition of about 60 to 65 percent lime (CaO) and 21 to 24 percent silica (SiO₂) with less than about 15 percent total of alumina (Al₂O₃), iron oxide (Fe₂O₃), magnesia (MgO) and sulphate (SO₄). Several variations of the cement compositions have been developed for certain applications, for example sulfate resistant varieties, quick set varieties, expanding varieties for demolition application, varieties for oil field applications and many others. The composition may also be modified by adding various substances, both organic and

inorganic, to optimize a particular set of properties for various applications. Inorganic materials used to modify the composition include fly ashes, silica fumes, blast furnace slags, and various natural pozzolans.

After one year at ambient temperatures, a typical Portland cement material will be made up of 95 to 98 percent of hydrated compounds and will, based on engineering experience, remain unchanged within the next 100 to 200 years (Atkins and Glasser, 1992) to perhaps thousands of years as found in ancient cements (Atkins et al, 1991). The grout after set and cure will consist of a liquid and a solid material. The liquid is an aqueous phase consisting of water and dissolved species. The water is located in the pore space. The pore space makes up about 20 to 30 volume percent of the set material and has a pore size generally <2µm, both pore volume and size depends primarily on the initial water-cement ratio. The porosity generally decreases with age (Atkins and Glasser, 1992). The solid material is composed primarily of cement matrix gel (referred to in the cement literature as "CSH"), a hydrated, amorphous material composed of lime (CaO), and silica (SiO₂) as well as water (H₂O). Additional phases may include lesser amounts of portlandite (Ca(OH)₂) and smaller amounts of other phases such as ettringite [(Ca₃Al(OH)₆*12H₂O)]₂(SO₄)₃*2H₂O, and hydrogarnet (Ca₃Al₂(OH)₁₂-Ca₃Al₂Si(OH)₈ and others (Atkins and Glasser, 1992). In the cases where fly ashes, silica fumes, or blast furnace slags are added to Portland cement, the amount of portlandite is reduced or eliminated by chemical reaction during the set and cure process and other phases, such as gehlinit hydrate (Ca₂AlSiO₄(OH)₃ and others, may be produced.

PH, Acid-Base Properties

The cement materials are somewhat soluble in water and control the pH of the water in the intergranular space within the waste form monolith. The most soluble materials produce the pH of the intergranular solution at a given time. The pH will change with time, becoming lower in successive steps, as each of the pH controlling phases is removed in turn by some processes such as dissolution or chemical reaction. In the case of Portland cement, small amounts of sodium and/or potassium hydroxide may cause the initial pH values to be very high, in excess of 13. These hydroxides are very water soluble, therefore the pH drops to lower values as they dissolve and are leached from the system. The portlandite, Ca(OH)₂, component of Portland cement maintains the pH of the intergranular solution at about 12.5 as long as any portlandite remains in the cement matrix. If portlandite is depleted or is initially not present as is the case in many blended grouts, dissolution of the cement matrix gel, CSH, controls the pH of the intergranular solution. As the CSH ages and changes composition slightly, the pH may decrease to about eleven. (Abrojano and Johnson, 1990) or 10.5 (Krupka and Serne 1998) The pH will remain at these values as long as CSH remains in the waste form matrix. Cement grouts "buffer" the pH for long periods of time because CSH is the dominant material, greater than about seventy percent of the total cementitious material. The pH will remain approximately constant as long as a portion of the CSH remains in chemical contact with the remainder of the system. If the cement matrix gel is totally removed or isolated by some process, residual phases or reaction products, particularly calcite, or the ambient environment will control the pH of the system. In the case of the SDA, ground water pH is about 7.2 at present, (Hull and Pace 2000) and is controlled by chemical reactions among calcite (CaCO₃) and carbon dioxide (CO₂) and ground water

The cement matrix gel may be removed from the system by several mechanisms. These include simple dissolution, crystallization and chemical reaction.

Dissolution is unlikely to remove significant quantities of the cement matrix because the results of American Nuclear Society (ANS)/ANSI 16.1 leach test show (see Section 3.6 in the body of the report) that several tens of thousands of years are required to remove one percent of the major components, given the water infiltration rate (8.5 cm/year) at the SDA. The ANS/ANSI 16.1 leach tests provide conservative estimates because the procedure uses distilled water and frequent leachate replacement. Similar

conclusions were reached by Alkorn et al, (1989) and also Alcorn et al (1990) who showed that Portland type-V grout waste repository seals, 0.5 m thick, would have worst case performance life time of several tens of thousands of years.

Crystallization of cement matrix gel would be unlikely to significantly affect pH values in time periods less than several thousand years. Crystallization of cement matrix gel would cause it to become a crystalline material. and would therefore have different properties. The cement matrix gel is an amorphous to slightly ordered material capable of showing a diffuse, poorly defined x-ray diffraction pattern similar to the mineral tobermorite. The cement matrix gel is thermodynamically unstable with respect to well crystallized materials, such as tobermorite, which have a similar bulk composition. The pH produced by a semicrystalline tobermorite is 11 (Atkins et al, 1990). If the matrix crystallizes, the pH will be somewhat lower. Experimental studies measuring pH versus time have shown that both Portland type V cement and type V cement modified with blast furnace slag or fly ash require about 500,000 to 1,000,000 years for the pH to decline to 10 (Atkinson, et al 1990).

The cement matrix gel can also be affected by reaction with other chemical species within the waste site environment such as sulfate (SO₄) and carbon dioxide (CO₂). In this case the pH controlling phases are removed from the system by chemical reaction. The rate of these degradation reactions is controlled by the rate of diffusion of sulfate, carbon dioxide and related species into the cement matrix from the surrounding environment. Potential sulfate-cement reaction products include gypsum (CaSO₄) and ettringite Ca₆Al₂(SO₄)₃(OH)₁₂*26H₂O. A minor amount of gypsum is an additive to certain grout materials and minor ettringite is a common cement phase. Typical SDA ground water does not have a high sulfate content and is not saturated in gypsum, (Hull and Pace 2000). Compared to typical grout materials the chemical potential of sulphate in SDA ground water is not high and is not expected to have a significant affect on in situ grouting grout materials. Carbon dioxide is an important component in the SDA geochemical system and locally comprises up to ten percent of the soil gas. In the case of the in situ grouting materials, the diffusion rate of carbon dioxide and related species, as well as sulfate, will be no greater than the rate of diffusion of the nitrate measured using laboratory in situ grouting samples and the ANS/ANSI 16.1 diffusion measurement procedure. Computer model estimates of the rate of carbon dioxide penetration of cement waste form materials indicate about 7 cm of the outer repository wall could be penetrated in 300 years (Keum et al 1997), assuming a CO₂ aqueous source saturated with calcite and an effective diffusion coefficient of 4.1×10^{-4} m²/year. The measured effective diffusion coefficients for nitrate in the in situ grouting grout materials are about 1.2×10^{-6} m²/year or about 100 times smaller than that used in the computer simulation model. The SDA ground water is saturated in calcite at a pH of about 7.2 (Hull and Pace 2000). The 7.2 pH is the limiting value in the case of complete alteration of the cement matrix to calcite and silica (opal)

eH

The oxidation state of in situ grouting grout materials control the eH environment within the intergranular pore solutions within the in situ grouting monolith in a fashion similar to the pH (Atkins and Glasser, 1992). Portland cement and similar cementitious materials are manufactured by heating, in air, mixtures of calcite, clay and other materials to temperatures somewhat above the beginning of melting of the calcined ingredients. Air is “oxidizing” compared to many environments and the relatively oxidizing character of air present in the high temperature kilns during the cement manufacturing processes is inherited by the finished cement product. eH measurements of typical Portland cements range from 0 to about 100 mV (Atkins and Glasser 1992). Blast furnace slags have an oxidation-reduction character exactly opposite that of Portland cement. Blast furnace slag is a by-product of the iron and steel manufacturing processes. Like Portland cement, iron and steel are also produced at high temperature, above the beginning of melting of the oxide as well as metallic constituents. Unlike Portland cement, the manufacture of iron and steel produces very reducing conditions, much more so than is found in most

environments. Blast furnace slag is a glassy material containing one to two percent of dissolved sulphur and also iron and manganese (Atkins and Glasser,1992), all of which are in a chemically reduced form. The reduced chemical species impose and maintain the very strongly reducing conditions of the original iron and steel making process when used as a hydraulic cement material. The eH of the intergranular pore fluid in blast furnace slag cements is typically about -300 mV. (Atkins and Glasser,1992) The oxidizing capacity of a grout material to control eH can be measured by an electro-titration method (Atkins and Glasser,1992). The development of the eH value of grout materials produced by blends between Portland cement and blast furnace slag is time dependent, with lesser quantities of slag requiring longer time periods to produce the low eH, reducing conditions. For example, cement-blast furnace slag blends containing more than 70% slag produced reducing conditions within one month where as the data suggested that a 50% blend would probably require more than 18 months. The time dependence of the eH reduction is thought to be due to the slow reaction rate of blast furnace slag. (Atkins and Glasser,1992) In the natural environment, the grout materials would become oxidized over time and eventually lose their eH controlling properties. There are virtually no quantitative data to estimate the time period that grout materials would control the eH of their intergranular pore solutions. The oxidation rate would probably be comparable to the rate of diffusions of oxidizing chemical species into the treated waste material.

In Situ Grouting Bench Test Results

The in situ grouting bench tests have measured the pH, eH and many other properties of five potential grout candidates and the affect on the in situ grouting properties when mixed with nitrate salts (12 weight percent), SDA soils (fifty weight percent) and simulated series 743 organic sludge from the Rocky Flats Plant (nine weight percent). The results are presented in detail in Appendix C and Appendix D and discussed in Section ---of the Final Report. The grout materials include:

TECT

TECT is a pozzolanic cementitious grout with proprietary additives (Grant et al. 2000). The average pH of five through forty three day ANS/ANDI 16.1 leach periods is 11.2 (Appendix C). Similar measurements with added nitrate salts: 11.7, with added organic sludge: 11.6, with added SDA soil:11.6 (Appendix D). The average eH values are 241 mV (Appendix C) for the neat material and is virtually constant for all interference mixtures at 410 mV.

U.S. Grout (Ultra Fine Grout)

U.S. Fine grout (American Petroleum Institute [API] Type H) is a pozzolanic material (Grant et al. 2000). The average pH of five through forty three day ANS/ANDI 16.1 leach periods is 11 (Appendix C). Similar measurements with added nitrate salts: 11.3, with added organic sludge: 11.5, with added SDA soil:11.6 (Appendix D). The average eH values are 241 mV (Appendix C) for the neat material. It is virtually constant for all interference mixtures at 410 mV.

Enviro-Blend

Enviro-Blend is a proprietary cementitious grout containing phosphorous. The average pH of five through forty three day ANS/ANDI 16.1 leach periods is 10.3 (Appendix C). Similar measurements with added nitrate salts: 10.8, with added organic sludge: 10.2, with added SDA soil:10.8 (Appendix D). The average eH values are 254 mV (Appendix C) for the neat material. eH is virtually constant for all interference mixtures at 408 mV.

GMENT-12

GMENT-12 is a derivative of the Tank Closure Grout (Westinghouse Savannah River Company [WSRC] 1997) developed at the Savannah River site to stabilize waste remnants in storage tanks. It is formulated with over 50 weight percent Type V cement (ASTM C150), about nine weight percent ground blast furnace slag and lesser silica fume and thirty percent water plus various additives. (Grant et al. 2000). The average pH of five through forty three day ANS/ANDI 16.1 leach periods is 10.8 (Appendix C). Similar measurements with added nitrate salts: 11.4, with added organic sludge: 11.6, with added SDA soil: 11.0 (Appendix D). The average eH values are 247 mV (Appendix C) for the neat material. eH is virtually constant for all interference mixtures at 410 mV.

Salt Stone

Salt Stone was developed at the Savanna River Site to stabilize nitrate salt waste streams and associated radioactive contaminants. (WSRC 1992 and 1994) It is formulated from a mixture of Class F fly ash and grade 120 blast furnace slag in equal proportions together with 3.3 weight percent Portland Type II cement (ASTM C150) and 41.3 weight percent water (Grant et al. 2000). The average pH of five through forty three day ANS/ANDI 16.1 leach periods is 10.7 (Appendix C). Similar measurements with added nitrate salts: 10.9, with added organic sludge: 10.5, with added SDA soil: 11.3 (Appendix D). The average eH values are 212 mV (Appendix C) for the neat material. eH is virtually constant for all interference mixtures at 411 mV.

Recommended pH and eH Values

A single recommended pH value for contaminate solubility estimates is eleven. The results of the bench testing indicate that all the tested grout formulations behave similar to blended cements and have pH values less than 12.5, indicating an absence of the phase portlandite. TECT, GMENT-12, SALT TONE, and U.S. Grout are very similar to one another and have pH values in the range 10.7 to 11.7 including neat grout samples as well as the mixtures of grout and interference material. Of these, GMENT-12 and Saltstone may have systematically slightly lower pH values by about 0.4 units, but the variation in the data is too great to demonstrate this conclusively. The pH of neat Enviro-Blend and mixtures of this grout with interference materials range from 10.3 (neat) to 10.8 (INEL soil and nitrate salts). These values are about 0.8 units less than TECT and U.S. Grout and are greater than the scatter in the data.

The single value recommended for modeling purposes is pH 11, a reasonable representative of the grout formulations being considered and consistent with the long-term pH boundary of 11 (Atkins and Glasser, 1992) or 10.5 (Krupka and Serne 1998) imposed by cement matrix gel on the intergranular matrix pore solutions.

Three eH values are suggested for contaminant solubility estimates. These are: -300 mV, a representative value for blast furnace slag (Atkins and Glasser, 1992); 0 mV, a representative value for Portland cements and similar grout materials (Atkins and Glasser, 1992); and 500 mV, a representative value for SDA ground water (Eric Miller, personal communication, 2002). The blast furnace slag represents the long term eH boundary for reducing materials such as SALT STONE and GMENT-12. The eH value for Portland cement is a reasonable estimate for grout formulations which do not contain chemically reducing materials such as sulfur and/or ferrous iron.

The measured eH data of the grout formulations and their mixture with interference materials are difficult to interpret. It is suggested that they not be used for contaminant solubility estimates. The average values for all measurement of neat grout samples are virtually identical at 241 to 254 mV, except

for SALT STONE, which is 212 mV and is not significantly different from the other samples. Individual measurements for a given sample may vary by up to 100 mV. The eH values for all grout-interference mixtures is very constant at about 410 mV with very little scatter in the data. All the measured eH values are very oxidizing compared to most environments and above the values expected by pure Portland cement (0 to 100 mV, Atkins and Glasser 1992). Saltstone and GMENT-12 both contain blast furnace slag and are potentially very reducing. Blast furnace slag typically has eH values of about -300 mV although several months may be needed for the necessary chemical reactions to take place, (Atkins and Glasser 1992). The eH measurements were made on the leachate from the ANS/ANSI16.1 leach tests using the ASTM 1498-93 standard procedure. The leachate itself has very little capacity to preserve the eH of the intergranular pore solutions. Other factors, such as oxygen from air, may have changed the apparent eH value. The eH imposed by the neat grouts is significantly less than the same grouts mixed with interference materials, about 210 mV for neat grouts versus about 410 mV for grouts mixed with interference materials. It is suggested that some common factor, such as air entrainment during blending of the cement- interference mixture samples, together with very slow chemical reaction rates in the grouts containing reducing materials may have resulted in little or no eH reaction and reduction during the sample leach period. Given the uncertainty in the measured eH data, three eH values are given for the contaminant solubility estimates to provide a reasonable set of values for comparison

References

- Alkorn, S.R., J.Meyers, M.A.Gardiner, and C.A. Givens (1989) *Chemical Modeling of Cementitious Grout Materials Alteration in HLW Repositories Waste Management 1989* Vol. 1 High-Level Waste and General Interest pg279-286
- Alkorn, S.R., WE Coons, and MA Gardner (1990) *Estimation of longevity of Portland Cement Grout Using Chemical Modeling Techniques* Mat Res Soc sym Proc Vol 176 pg 165-173
- ASTM (1999) *Annual Book of ASTM Standards American Society for Testing Materials* West Conshohocken, Pennsylvania
- Atkins, M., F.P.Glasser, and L.P.Moroni (1990) *The Long-Term Properties of Cement and Concretes Scientific Basis for Nuclear waste Management XIV*, Nov.26-29 Boston Massachusetts Materials Research Society Symposium Proceedings Vol 212 pgs373-386
- Atkins, M., F.Glasser, A.Kindness, D.Bennet, A.Dawes, D.Read (1991) *A Thermodynamic model for Blended Cements DOE Report No. DoE/HMIP/RR/005* DOE Reference: PECD/7/9/503 120 pgs.
- Atkins, M. and F.P. Glasser, (1992) *Application of Portland cement-based Materials to Radioactive waste Immobilization Waste Management* Vol. 12 pgs 105-131
- Atkinson, Alan, Niccola M.Everitt and Richard M. Guppy (1988) *Time Dependence of pH in a Cementitious Repository Scientific Basis for Nuclear waste Management XII*, Oct 10-13 Berlin Germany Materials Research Society Symposium Proceedings Vol 127 pg 439-446
- Grant, R., J. J. Jessmore, G. Loomis, J. Weidner (2000), *Test Plan for the Operable Unit 7-13/14 Bench-Testing In Situ Grouting Treatability Study*, INEEL/EXT-99-00914, Rev. 0, Bechtel BWXT Idaho, LLC, March 2000, 50 pp.
- Krupka, K.M., and R.J. Serne (1998) *Effects on Radionuclide Concentrations by Cement/Ground-water Interactions in Support of Performance Assessment of Low-Level Radioactive waste Disposal Facilities NUREG/CR-6377 PNNL-11408* Pacific Northwest Laboratory, 118 pp.

Keum, D.K. W.J. Cho and P.S.Hahn (1997) *Evaluation of Concrete Degradation Under Disposal Environment Journal of the Korean Nuclear Society* V29, No.3 pp260-26

WSRC, (1992) *Radiological Performance Assessment Z-area Salt Stone Disposal Facility, WSRC-RP-92-1360*, Westinghouse Savannah River Company, Aiken, South Caroline

WSRC, (1994) *Radiological Performance Assessment E-area Vaults Disposal Facility, WSRC-RP-94-218*, Westinghouse Savannah River Company, Aiken, South Caroline

WSRC, (1997) *Tank Closure Reducing Grout, WSRC-TR-97-0102*, Westinghouse Savannah River Company, Aiken, South Carolina.

Appendix E

Grout with Interferences ANS 16.1 Individual Sample Data

Appendix E

Grout with Interferences ANS 16.1 Individual Sample Data

Table 1. U Grout with 9% Organic Sludge - Strontium American Nuclear Society (ANS) 16.1 Data.

Time (d)	Sr (mg/L)			Leach Index (LI)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	0.010	0.010	0.010	11.8	11.8	11.8
0.292	0.010	0.010	0.020	11.7	11.7	11.1
1.000	0.080	0.080	0.110	10.4	10.4	10.2
2.000	0.110	0.090	0.120	10.1	10.3	10.0
3.000	0.110	0.130	0.120	9.8	9.7	9.8
4.000	0.060	0.050	0.060	10.2	10.4	10.2
5.000	0.050	0.040	0.040	10.3	10.5	10.5
19.000	0.230	0.270	0.260	10.9	10.7	10.7
47.000	0.150	0.160	0.140	11.4	11.3	11.4
90.000	0.190	0.150	0.130	11.2	11.4	11.5

Time (d)	De (cm ² /s)			eH (mV)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	1.46E-12	1.46E-12	1.46E-12	323.0	322.1	398.0
0.292	1.90E-12	1.90E-12	7.62E-12	382.1	375.5	384.2
1.000	3.61E-11	3.61E-11	6.83E-11	402.4	395.0	388.5
2.000	8.41E-11	5.58E-11	9.96E-11	403.0	421.0	401.2
3.000	1.43E-10	2.01E-10	1.69E-10	412.3	422.0	412.0
4.000	5.99E-11	4.16E-11	5.99E-11	416.8	404.1	420.1
5.000	5.35E-11	3.42E-11	3.42E-11	411.0	398.0	412.0
19.000	1.40E-11	1.92E-11	1.79E-11	412.0	403.0	421.0
47.000	4.30E-12	4.88E-12	3.76E-12	412.0	423.0	432.0
90.000	6.21E-12	3.87E-12	2.93E-12	414.8	412.6	421.5

Time (d)	pH			eH (mV)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	10.3	10.2	10.3	323.0	322.1	398.0
0.292	10.6	10.5	10.6	382.1	375.5	384.2
1.000	11.4	11.2	11.2	402.4	395.0	388.5
2.000	11.2	11.0	11.8	403.0	421.0	401.2
3.000	11.0	11.1	11.0	412.3	422.0	412.0
4.000	11.1	11.1	11.1	416.8	404.1	420.1
5.000	11.0	11.0	10.9	411.0	398.0	412.0
19.000	11.6	11.6	11.3	412.0	403.0	421.0
47.000	11.3	10.2	10.9	412.0	423.0	432.0
90.000	11.7	11.8	11.7	414.8	412.6	421.5

Table 2. U Grout with 12% Nitrate Salt - Strontium ANS 16.1 Data.

Time (d)	Sr (mg/L)			Leach Index (LI)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	0.010	0.010	0.010	11.9	11.9	11.9
0.292	0.010	0.010	0.010	11.8	11.8	11.8
1.000	0.020	0.020	0.020	12.3	12.3	12.3
2.000	0.040	0.020	0.030	11.0	11.6	11.2
3.000	0.050	0.020	0.040	10.6	11.4	10.8
4.000	0.020	0.010	0.020	11.2	11.8	11.2
5.000	0.030	0.010	0.010	10.7	11.7	11.7
19.000	0.130	0.120	0.130	11.4	11.4	11.4
47.000	0.080	0.060	0.070	11.9	12.2	12.1
90.000	0.060	0.140	0.130	12.2	11.5	11.6

Time (d)	De (cm ² /s)			eH (mV)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	1.36E-12	1.36E-12	1.36E-12	398.0	376.2	388.1
0.292	1.77E-12	1.77E-12	1.77E-12	376.4	398.4	396.5
1.000	5.27E-13	5.27E-13	5.27E-13	398.2	402.6	429.8
2.000	1.03E-11	2.60E-12	5.81E-12	411.0	403.0	422.6
3.000	2.75E-11	4.41E-12	1.76E-11	399.0	400.2	412.0
4.000	6.21E-12	1.55E-12	6.21E-12	406.9	403.2	403.5
5.000	1.79E-11	2.00E-12	2.00E-12	409.0	411.0	423.0
19.000	4.14E-12	3.57E-12	4.14E-12	399.2	410.0	412.0
47.000	1.14E-12	6.42E-13	8.74E-13	405.0	413.8	399.8
90.000	5.78E-13	3.13E-12	2.69E-12	407.2	405.2	408.3

Time (d)	pH			eH (mV)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	10.0	10.3	10.2	398.0	376.2	388.1
0.292	8.9	10.0	10.1	376.4	398.4	396.5
1.000	10.3	10.4	9.8	398.2	402.6	429.8
2.000	11.0	11.1	11.1	411.0	403.0	422.6
3.000	10.8	10.8	10.6	399.0	400.2	412.0
4.000	10.9	10.8	11.2	406.9	403.2	403.5
5.000	11.0	11.1	11.0	409.0	411.0	423.0
19.000	11.3	11.4	11.3	399.2	410.0	412.0
47.000	11.5	11.4	11.6	405.0	413.8	399.8
90.000	11.4	11.6	11.5	407.2	405.2	408.3

Table 3. U Grout with 50% INEEL Soil - Strontium ANS 16.1 Data.

Time (d)	Sr (mg/L)		
	Sample 1	Sample 2	Sample 3
0.083	0.010	0.010	0.010
0.292	0.010	0.010	0.010
1.000	0.040	0.070	0.060
2.000	0.080	0.080	0.070
3.000	0.040	0.070	0.100
4.000	0.030	0.050	0.050
5.000	0.020	0.040	0.020
19.000	0.080	0.050	0.120
47.000	0.050	0.070	0.080
90.000	0.060	0.060	0.020

Time (d)	De (cm ² /s)			Leach Index (LI)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	1.13E-12	1.13E-12	1.13E-12	11.9	11.9	11.9
0.292	1.48E-12	1.48E-12	1.48E-12	11.8	11.8	11.8
1.000	7.03E-12	2.16E-11	1.58E-11	11.2	10.7	10.8
2.000	3.47E-11	3.47E-11	2.66E-11	10.5	10.5	10.6
3.000	1.47E-11	4.51E-11	9.21E-11	10.8	10.3	10.0
4.000	1.17E-11	3.24E-11	3.24E-11	10.9	10.5	10.5
5.000	6.67E-12	2.67E-11	6.67E-12	11.2	10.6	11.2
19.000	1.32E-12	5.16E-13	2.95E-12	11.9	12.3	11.5
47.000	3.73E-13	7.31E-13	9.56E-13	12.4	12.1	12.0
90.000	4.83E-13	4.83E-13	5.37E-14	12.3	12.3	13.3

Time (d)	pH			eH (mV)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	10.5	10.2	10.1	388.5	389.2	366.1
0.292	10.3	10.2	10.2	376.0	402.1	398.5
1.000	11.0	11.2	11.2	412.0	413.0	411.5
2.000	11.1	11.2	11.2	400.0	412.0	405.3
3.000	10.2	10.1	9.8	398.2	411.0	416.0
4.000	11.0	11.0	11.0	399.5	399.5	423.1
5.000	11.3	11.4	11.3	402.6	407.8	407.4
19.000	11.3	11.4	11.5	399.0	413.0	412.0
47.000	11.7	11.5	11.6	423.0	413.0	401.6
90.000	11.6	11.5	11.8	412.0	412.8	415.9

Table 4. T Grout with 9% Organic Sludge - Strontium ANS 16.1 Data.

Time (d)	Sr (mg/L)			Leach Index (LI)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	0.040	0.040	0.030	10.9	10.9	11.1
0.292	0.010	0.020	0.010	12.0	11.4	12.0
1.000	0.080	0.170	0.060	10.7	10.0	10.9
2.000	0.080	0.140	0.090	10.6	10.1	10.5
3.000	0.100	0.100	0.080	10.2	10.2	10.4
4.000	0.060	0.140	0.070	10.5	9.7	10.3
5.000	0.060	0.110	0.040	10.3	9.8	10.7
19.000	0.690	0.750	0.730	10.1	10.1	10.1
47.000	0.710	0.680	0.770	10.2	10.3	10.2
90.000	1.350	1.450	1.390	9.7	9.7	9.7

Time (d)	De (cm ² /s)			eH (mV)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	1.37E-11	1.37E-11	7.67E-12	398.0	376.2	347.8
0.292	1.12E-12	4.46E-12	1.12E-12	382.1	376.5	386.2
1.000	2.13E-11	9.59E-11	1.20E-11	403.0	421.6	388.5
2.000	2.62E-11	8.00E-11	3.31E-11	400.2	412.0	401.2
3.000	6.95E-11	6.95E-11	4.45E-11	412.3	422.0	412.0
4.000	3.52E-11	1.91E-10	4.78E-11	421.0	404.1	416.2
5.000	4.53E-11	1.52E-10	2.02E-11	411.0	426.0	412.0
19.000	7.42E-11	8.74E-11	8.29E-11	409.0	412.0	421.0
47.000	5.67E-11	5.20E-11	6.68E-11	399.8	416.3	405.0
90.000	1.85E-10	2.12E-10	1.95E-10	407.8	412.7	415.6

Time (d)	pH			eH (mV)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	10.5	10.5	10.5	398.0	376.2	347.8
0.292	10.1	10.2	10.2	382.1	376.5	386.2
1.000	11.1	11.0	11.1	403.0	421.6	388.5
2.000	11.1	11.1	11.3	400.2	412.0	401.2
3.000	11.0	11.0	10.8	412.3	422.0	412.0
4.000	10.9	10.9	10.9	421.0	404.1	416.2
5.000	11.0	11.5	11.4	411.0	426.0	412.0
19.000	11.8	11.8	11.8	409.0	412.0	421.0
47.000	11.6	11.7	11.5	399.8	416.3	405.0
90.000	11.9	11.8	11.9	407.8	412.7	415.6

Table 5. T Grout with 12% Nitrate Salt - Strontium ANS 16.1 Data.

Time (d)	Sr (mg/L)			Leach Index (LI)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	0.020	0.020	0.010	11.5	11.5	12.1
0.292	0.010	0.020	0.020	12.0	11.4	11.4
1.000	0.050	0.040	0.110	11.1	11.3	10.4
2.000	0.070	0.080	0.160	10.6	10.1	10.5
3.000	0.100	0.100	0.100	10.2	10.2	10.2
4.000	0.050	0.070	0.120	10.5	9.7	10.3
5.000	0.080	0.040	0.110	10.1	10.7	9.8
19.000	0.880	0.210	1.180	9.9	11.2	9.7
47.000	0.600	0.900	0.800	10.4	10.0	10.1
90.000	0.330	0.340	0.710	11.0	10.9	10.3

Time (d)	De (cm ² /s)			pH		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	3.42E-12	3.42E-12	8.56E-13	10.2	10.2	10.6
0.292	1.12E-12	4.46E-12	4.46E-12	10.5	10.5	10.4
1.000	8.28E-12	5.31E-12	4.01E-11	11.0	11.2	11.2
2.000	2.62E-11	8.00E-11	3.31E-11	11.1	11.0	11.1
3.000	6.95E-11	6.95E-11	6.95E-11	11.4	11.2	11.3
4.000	3.52E-11	1.91E-10	4.78E-11	11.2	11.0	11.1
5.000	8.06E-11	2.02E-11	1.52E-10	11.6	11.4	11.5
19.000	1.21E-10	6.90E-12	2.17E-10	12.0	12.0	11.7
47.000	4.05E-11	9.11E-11	7.21E-11	11.9	11.5	11.6
90.000	1.10E-11	1.17E-11	5.11E-11	11.9	11.7	11.9

Time (d)	eH (mV)		
	Sample 1	Sample 2	Sample 3
0.083	398.0	376.2	347.8
0.292	376.6	398.1	396.4
1.000	398.2	402.6	441.2
2.000	411.0	393.0	396.2
3.000	399.0	403.0	421.2
4.000	407.1	402.3	403.5
5.000	410.5	411.0	423.0
19.000	399.2	412.0	403.0
47.000	400.0	401.0	396.0
90.000	205.8	412.7	412.9

Table 6. T Grout with 50% INEEL Soil - Strontium ANS 16.1 Data.

Time (d)	Sr (mg/L)			Leach Index (LI)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	0.020	0.020	0.030	11.5	11.5	11.1
0.292	0.010	0.020	0.020	12.0	11.4	11.4
1.000	0.070	0.050	0.080	10.8	11.1	10.7
2.000	0.110	0.070	0.120	10.7	10.6	10.0
3.000	0.840	1.020	1.030	8.3	8.1	8.1
4.000	0.060	0.040	0.070	10.6	10.3	9.9
5.000	0.050	0.030	0.050	10.5	10.9	10.5
19.000	0.500	0.420	0.560	10.4	10.6	10.3
47.000	0.490	0.510	0.400	10.6	10.5	10.7
90.000	0.570	0.440	0.190	10.5	10.7	11.4

Time (d)	De (cm ² /s)			pH		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	3.42E-12	3.42E-12	7.67E-12	10.2	10.2	10.1
0.292	1.12E-12	4.46E-12	4.46E-12	10.2	10.5	10.4
1.000	1.62E-11	8.28E-12	2.13E-11	11.1	11.1	11.0
2.000	2.00E-11	2.62E-11	1.04E-10	11.4	11.4	11.4
3.000	4.90E-09	7.22E-09	7.37E-09	11.0	11.0	11.0
4.000	2.44E-11	4.78E-11	1.41E-10	10.8	11.0	10.9
5.000	3.14E-11	1.13E-11	3.14E-11	11.0	11.3	11.4
19.000	3.88E-11	2.74E-11	4.88E-11	11.9	11.8	11.6
47.000	2.71E-11	2.93E-11	1.80E-11	11.6	11.8	11.5
90.000	3.29E-11	1.96E-11	3.64E-12	11.8	11.5	11.7

Time (d)	eH (mV)		
	Sample 1	Sample 2	Sample 3
0.083	366.5	381.2	376.5
0.292	376.0	402.2	398.5
1.000	401.6	423.0	412.6
2.000	399.8	403.6	402.0
3.000	398.2	411.0	416.0
4.000	400.0	399.5	423.1
5.000	402.6	402.8	407.8
19.000	415.6	411.2	411.0
47.000	415.6	423.5	412.8
90.000	421.5	413.7	412.0

Table 7. E Grout with 9% Organic Sludge - Strontium ANS 16.1 Data.

Time (d)	Sr (mg/L)		
	Sample 1	Sample 2	Sample 3
0.083	0.010	0.010	0.010
0.292	0.010	0.010	0.010
1.000	0.010	0.010	0.010
2.000	0.010	0.010	0.010
3.000	0.010	0.020	0.020
4.000	0.010	0.020	0.030
5.000	0.010	0.010	0.010
19.000	0.010	0.010	0.010
47.000	0.100	0.120	0.110
90.000	0.030	0.020	0.020

Time (d)	De (cm ² /s)			Leach Index (LI)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	1.31E-12	1.31E-12	1.31E-12	11.9	11.9	11.9
0.292	1.71E-12	1.71E-12	1.71E-12	11.8	11.8	11.8
1.000	5.08E-13	5.08E-13	5.08E-13	12.3	12.3	12.3
2.000	6.26E-13	6.26E-13	6.26E-13	12.2	12.2	12.2
3.000	1.06E-12	4.29E-12	4.29E-12	12.0	11.4	11.4
4.000	1.50E-12	6.04E-12	1.35E-11	11.8	11.2	10.9
5.000	1.93E-12	1.93E-12	1.93E-12	11.7	11.7	11.7
19.000	2.38E-14	2.38E-14	2.38E-14	13.6	13.6	13.6
47.000	1.72E-12	2.50E-12	2.10E-12	11.8	11.6	11.7
90.000	1.40E-13	6.26E-14	6.26E-14	12.9	13.2	13.2

Time (d)	pH			eH (mV)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	7.9	8.8	8.8	325.6	333.8	356.4
0.292	8.5	7.8	7.7	366.5	378.6	398.6
1.000	9.5	8.4	9.8	398.0	376.2	388.1
2.000	9.9	9.9	9.7	376.4	398.4	396.5
3.000	10.5	10.5	10.4	398.2	402.6	441.2
4.000	10.2	10.3	10.4	411.0	403.0	421.6
5.000	10.0	10.1	10.1	399.2	409.0	412.0
19.000	9.7	10.5	10.0	397.0	399.8	416.3
47.000	10.1	10.3	10.2	407.1	402.3	403.5
90.000	10.2	10.7	10.5	406.4	412.6	417.8

Table 8. E Grout with 12% Nitrate Salt - Strontium ANS 16.1 Data.

Time (d)	Sr (mg/L)			Leach Index (LI)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	0.020	0.010	0.010	11.3	11.9	11.9
0.292	0.010	0.010	0.010	11.8	11.8	11.8
1.000	0.010	0.010	0.010	12.3	12.3	12.3
2.000	0.010	0.010	0.010	12.2	12.2	12.2
3.000	0.010	0.010	0.010	12.0	12.0	12.0
4.000	0.020	0.010	0.020	11.9	11.3	10.9
5.000	0.020	0.020	0.010	11.2	11.2	11.8
19.000	0.010	0.010	0.010	13.7	13.7	13.7
47.000	0.120	0.140	0.150	11.6	11.5	11.5
90.000	0.010	0.010	0.010	13.9	13.9	13.9

Time (d)	De (cm ² /s)			eH (mV)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	4.76E-12	1.19E-12	1.19E-12	382.1	376.5	386.2
0.292	1.55E-12	1.55E-12	1.55E-12	393.0	396.2	388.5
1.000	4.62E-13	4.62E-13	4.62E-13	403.0	421.2	401.2
2.000	5.69E-13	5.69E-13	5.69E-13	412.3	422.0	412.0
3.000	9.66E-13	9.66E-13	9.66E-13	402.4	402.7	414.0
4.000	1.36E-12	5.44E-12	1.22E-11	412.0	403.0	421.0
5.000	7.01E-12	7.01E-12	1.75E-12	401.0	396.0	405.0
19.000	2.17E-14	2.17E-14	2.17E-14	421.0	404.1	416.2
47.000	2.24E-12	3.08E-12	3.52E-12	411.0	426.0	412.0
90.000	1.41E-14	1.41E-14	1.41E-14	411.6	412.3	411.9

Time (d)	pH			eH (mV)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	7.6	7.6	8.0	382.1	376.5	386.2
0.292	8.7	8.5	8.0	393.0	396.2	388.5
1.000	8.9	7.7	9.2	403.0	421.2	401.2
2.000	9.2	9.1	9.6	412.3	422.0	412.0
3.000	9.6	10.0	10.0	402.4	402.7	414.0
4.000	10.1	10.2	10.4	412.0	403.0	421.0
5.000	10.6	10.6	10.3	401.0	396.0	405.0
19.000	11.0	11.0	11.1	421.0	404.1	416.2
47.000	10.8	10.6	10.9	411.0	426.0	412.0
90.000	10.6	10.8	10.7	411.6	412.3	411.9

Table 9. E Grout with 50% INEEL Soil - Strontium ANS 16.1 Data.

Time (d)	Sr (mg/L)		
	Sample 1	Sample 2	Sample 3
0.083	0.010	0.010	0.010
0.292	0.010	0.010	0.010
1.000	0.010	0.010	0.010
2.000	0.010	0.010	0.010
3.000	0.010	0.010	0.010
4.000	0.020	0.020	0.010
5.000	0.010	0.010	0.010
19.000	0.010	0.010	0.010
47.000	0.010	0.010	0.010
90.000	0.010	0.010	0.010

Time (d)	De (cm ² /s)			Leach Index (LI)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	9.72E-13	9.72E-13	9.72E-13	12.0	12.0	12.0
0.292	1.27E-12	1.27E-12	1.27E-12	11.9	11.9	11.9
1.000	3.77E-13	3.77E-13	3.77E-13	12.4	12.4	12.4
2.000	4.65E-13	4.65E-13	4.65E-13	12.3	12.3	12.3
3.000	7.90E-13	7.90E-13	7.90E-13	12.1	12.1	12.1
4.000	4.42E-12	4.42E-12	1.11E-12	11.4	11.4	12.0
5.000	1.43E-12	1.43E-12	1.43E-12	11.8	11.8	11.8
19.000	1.77E-14	1.77E-14	1.77E-14	13.8	13.8	13.8
47.000	1.28E-14	1.28E-14	1.28E-14	13.9	13.9	13.9
90.000	1.15E-14	1.15E-14	1.15E-14	13.9	13.9	13.9

Time (d)	pH			eH (mV)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	10.5	9.4	9.9	398.2	389.0	376.6
0.292	10.0	10.0	9.9	399.8	389.2	364.1
1.000	9.8	10.0	10.2	416.8	403.6	405.5
2.000	10.0	10.4	10.3	376.0	402.1	398.5
3.000	10.4	10.6	10.6	415.6	411.2	411.0
4.000	10.7	10.5	10.6	402.6	402.6	407.5
5.000	10.6	10.6	10.6	412.0	413.0	411.5
19.000	11.0	11.0	11.1	417.3	413.0	412.0
47.000	10.9	10.8	10.8	409.0	411.0	423.0
90.000	10.8	10.9	10.8	411.7	412.7	416.0

Table 10. C75 Grout with 9% Organic Sludge - Strontium ANS 16.1 Data.

Time (d)	Sr (mg/L)			Leach Index (LI)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	0.020	0.030	0.010			
0.292	0.030	0.020	0.020			
1.000	0.080	0.050	0.090			
2.000	0.180	0.160	0.150			
3.000	0.100	0.120	0.170			
4.000	0.070	0.140	0.140			
5.000	0.070	0.050	0.060			
19.000	0.660	0.680	0.670			
47.000	0.310	0.320	0.330			
90.000	0.440	0.420	0.290			

Time (d)	De (cm ² /s)			Leach Index (LI)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	6.64E-12	1.49E-11	1.65E-12	11.2	10.8	11.8
0.292	1.94E-11	8.66E-12	8.66E-12	10.7	11.1	11.1
1.000	4.12E-11	1.61E-11	5.18E-11	10.4	10.8	10.3
2.000	2.57E-10	2.04E-10	1.79E-10	9.6	9.7	9.7
3.000	1.34E-10	1.93E-10	3.90E-10	9.9	9.7	9.4
4.000	9.29E-11	3.73E-10	3.73E-10	10.0	9.4	9.4
5.000	1.20E-10	6.10E-11	8.78E-11	9.9	10.2	10.1
19.000	1.31E-10	1.40E-10	1.36E-10	9.9	9.9	9.9
47.000	2.09E-11	2.23E-11	2.37E-11	10.7	10.7	10.6
90.000	3.81E-11	3.47E-11	1.65E-11	10.4	10.5	10.8

Time (d)	pH			eH (mV)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	9.7	9.6	9.6	336.5	354.8	346.7
0.292	10.0	10.3	10.2	398.6	377.5	386.0
1.000	10.7	10.7	10.7	402.5	396.2	402.3
2.000	10.8	10.9	10.9	412.0	421.2	401.2
3.000	10.6	10.5	10.6	407.8	422.0	412.0
4.000	11.8	11.8	11.6	421.0	404.1	415.8
5.000	11.6	11.4	11.9	404.6	426.0	412.0
19.000	11.5	11.4	11.7	413.6	403.0	422.6
47.000	11.4	11.4	11.6	405.0	413.0	402.8
90.000	11.6	11.7	11.6	404.6	405.8	416.7

Table 11. C75 Grout with 12% Nitrate Salt - Strontium ANS 16.1 Data.

Time (d)	Sr (mg/L)		
	Sample 1	Sample 2	Sample 3
0.083	0.010	0.010	0.010
0.292	0.020	0.020	0.010
1.000	0.040	0.040	0.040
2.000	0.050	0.060	0.060
3.000	0.027	0.030	0.040
4.000	0.040	0.020	0.030
5.000	0.030	0.030	0.040
19.000	0.080	0.290	0.290
47.000	0.160	0.180	0.200
90.000	0.320	0.280	0.280

Time (d)	De (cm ² /s)			Leach Index (LI)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	1.54E-12	1.54E-12	1.54E-12	11.8	11.8	11.8
0.292	8.10E-12	8.10E-12	2.01E-12	11.1	11.1	11.7
1.000	9.60E-12	9.60E-12	9.60E-12	11.0	11.0	11.0
2.000	1.85E-11	2.66E-11	2.66E-11	10.7	10.6	10.6
3.000	9.16E-12	1.13E-11	2.01E-11	11.0	10.9	10.7
4.000	8.66E-11	3.47E-10	3.47E-10	10.1	9.5	9.5
5.000	2.05E-11	2.05E-11	3.64E-11	10.7	10.7	10.4
19.000	1.80E-12	2.36E-11	2.36E-11	11.7	10.6	10.6
47.000	5.21E-12	6.57E-12	8.17E-12	11.3	11.2	11.1
90.000	1.88E-11	1.44E-11	1.44E-11	10.7	10.8	10.8

Time (d)	pH			eH (mV)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	9.9	10.0	9.9	398.0	376.2	347.8
0.292	9.8	9.6	9.8	382.1	375.5	384.2
1.000	11.8	11.7	11.7	398.2	402.6	441.2
2.000	10.9	10.8	10.9	411.0	403.7	421.6
3.000	11.0	11.0	11.3	399.0	400.8	412.0
4.000	10.7	10.5	10.6	407.1	402.9	403.9
5.000	11.2	11.2	11.1	409.0	411.0	423.0
19.000	11.5	11.6	11.5	399.2	409.0	412.0
47.000	11.4	11.2	11.4	412.0	403.0	421.0
90.000	11.5	11.5	11.8	404.9	415.6	411.8

Table 12. C75 Grout with 50% INEEL Soil - Strontium ANS 16.1 Data.

Time (d)	Sr (mg/L)			Leach Index (LI)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	0.020	0.020	0.030			
0.292	0.020	0.020	0.030			
1.000	0.110	0.110	0.080			
2.000	0.100	0.120	0.080			
3.000	0.100	0.100	0.140			
4.000	0.050	0.080	0.040			
5.000	0.040	0.060	0.050			
19.000	0.630	0.630	0.190			
47.000	0.430	0.320	0.300			
90.000	0.330	0.330	0.330			

Time (d)	De (cm ² /s)			Leach Index (LI)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	5.00E-12	5.00E-12	1.12E-11	11.3	11.3	11.0
0.292	6.52E-12	6.52E-12	1.46E-11	11.2	11.2	10.8
1.000	5.87E-11	5.87E-11	3.09E-11	10.2	10.2	10.5
2.000	5.91E-11	8.54E-11	3.80E-11	10.2	10.1	10.4
3.000	1.00E-10	1.00E-10	1.98E-10	10.0	10.0	9.7
4.000	2.27E-11	5.71E-12	1.28E-11	10.6	11.2	10.9
5.000	2.93E-11	6.60E-11	4.57E-11	10.5	10.2	10.3
19.000	8.99E-11	8.99E-11	8.16E-12	10.0	10.0	11.1
47.000	3.02E-11	1.68E-11	1.47E-11	10.5	10.8	10.8
90.000	1.60E-11	1.60E-11	1.60E-11	10.8	10.8	10.8

Time (d)	pH			eH (mV)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	9.9	10.0	10.0	376.1	376.2	388.1
0.292	9.9	10.2	10.2	376.0	412.8	398.5
1.000	12.0	12.0	12.0	412.0	413.0	421.0
2.000	10.7	10.6	10.6	400.6	403.6	402.0
3.000	10.7	10.6	10.5	398.2	411.0	417.8
4.000	10.7	10.6	10.6	399.8	413.2	422.4
5.000	10.9	11.0	11.0	402.6	402.6	407.4
19.000	11.7	11.6	11.6	417.9	411.2	411.0
47.000	11.0	11.4	11.3	415.8	412.6	399.5
90.000	11.3	11.4	11.5	421.0	413.0	415.0

Table 13. S Grout with 9% Organic Sludge - Strontium ANS 16.1 Data.

Time (d)	Sr (mg/L)		
	Sample 1	Sample 2	Sample 3
0.083	0.040	0.060	0.020
0.292	0.050	0.050	0.070
1.000	0.130	0.130	0.080
2.000	0.150	0.120	0.110
3.000	0.120	0.110	0.100
4.000	0.080	0.090	0.080
5.000	0.070	0.050	0.060
19.000	0.390	0.320	0.440
47.000	0.250	0.220	0.260
90.000	0.340	0.340	0.390

Time (d)	De (cm ² /s)			Leach Index (LI)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	1.96E-11	4.41E-11	4.90E-12	10.7	10.4	11.3
0.292	3.99E-11	3.99E-11	7.82E-11	10.4	10.4	10.1
1.000	7.97E-11	7.97E-11	3.04E-11	10.1	10.1	10.5
2.000	1.31E-10	8.41E-11	7.11E-11	9.9	10.1	10.1
3.000	1.43E-10	1.21E-10	9.85E-11	9.8	9.9	10.0
4.000	8.94E-11	1.13E-10	8.94E-11	10.0	9.9	10.0
5.000	8.82E-11	4.50E-11	6.49E-11	10.1	10.3	10.2
19.000	3.38E-11	2.28E-11	4.31E-11	10.5	10.6	10.4
47.000	1.01E-11	7.82E-12	1.09E-11	11.0	11.1	11.0
90.000	1.68E-11	1.68E-11	2.20E-11	10.8	10.8	10.7

Time (d)	pH			eH (mV)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	9.0	9.1	8.9	345.0	340.0	378.7
0.292	8.9	8.9	9.0	382.6	377.0	393.0
1.000	10.6	10.5	9.9	393.0	396.2	388.5
2.000	10.8	10.8	10.8	403.0	413.0	403.6
3.000	10.7	10.8	10.6	411.0	405.8	412.0
4.000	10.4	10.4	10.3	422.0	406.7	416.3
5.000	10.3	10.2	10.3	411.0	423.0	412.0
19.000	10.9	10.2	10.2	412.0	404.6	421.0
47.000	10.6	10.4	10.1	401.0	401.4	405.9
90.000	10.8	10.9	10.8	401.5	412.6	414.8

Table 14. S Grout with 12% Nitrate Salt - Strontium ANS 16.1 Data.

Time (d)	Sr (mg/L)			Leach Index (LI)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	0.020	0.020	0.020	11.3	11.3	11.3
0.292	0.070	0.040	0.060	10.1	10.6	10.3
1.000	0.140	0.080	0.070	10.1	10.5	10.7
2.000	0.190	0.110	0.120	9.9	10.1	10.2
3.000	0.100	0.130	0.120	10.0	9.8	9.9
4.000	0.100	0.100	0.090	9.9	9.9	10.0
5.000	0.080	0.060	0.050	10.0	10.2	10.4
19.000	0.500	0.440	0.210	10.3	10.4	11.0
47.000	0.380	0.300	0.310	10.7	10.9	10.8
90.000	0.420	0.450	0.500	10.6	10.6	10.5

Time (d)	De (cm ² /s)			pH		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	4.58E-12	4.58E-12	4.58E-12	9.6	9.7	9.6
0.292	7.29E-11	2.37E-11	5.35E-11	10.0	10.0	9.8
1.000	8.70E-11	2.83E-11	2.17E-11	10.5	10.5	10.6
2.000	1.23E-10	7.87E-11	6.62E-11	10.5	10.5	10.2
3.000	9.25E-11	1.57E-10	1.34E-10	10.6	10.7	10.7
4.000	1.30E-10	1.30E-10	1.05E-10	10.8	10.8	10.7
5.000	1.07E-10	6.04E-11	4.19E-11	10.5	10.5	10.5
19.000	5.18E-11	4.01E-11	9.18E-12	11.4	11.6	11.5
47.000	2.16E-11	1.35E-11	1.44E-11	10.6	10.7	10.5
90.000	2.38E-11	2.73E-11	3.37E-11	10.9	10.9	10.9

Time (d)	eH (mV)		
	Sample 1	Sample 2	Sample 3
0.083	402.0	370.6	399.0
0.292	378.5	399.7	396.8
1.000	400.2	404.9	433.2
2.000	411.5	402.5	421.6
3.000	399.0	400.2	412.0
4.000	407.1	416.4	403.5
5.000	409.8	411.0	402.3
19.000	402.6	409.9	412.0
47.000	412.6	419.7	422.0
90.000	403.2	405.1	412.4

Table 15. S Grout with 50% INEEL Soil - Strontium ANS 16.1 Data.

Time (d)	Sr (mg/L)		
	Sample 1	Sample 2	Sample 3
0.083	0.050	0.050	0.070
0.292	0.030	0.020	0.020
1.000	0.070	0.060	0.080
2.000	0.070	0.060	0.070
3.000	0.100	0.110	0.120
4.000	0.100	0.100	0.100
5.000	0.080	0.070	0.070
19.000	0.480	0.460	0.490
47.000	0.260	0.280	0.280
90.000	0.180	0.410	0.360

Time (d)	De (cm ² /s)			Leach Index (LI)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	2.45E-11	2.45E-11	4.82E-11	10.6	10.6	10.3
0.292	1.15E-11	5.11E-12	5.11E-12	10.9	11.3	11.3
1.000	1.87E-11	1.37E-11	2.44E-11	10.7	10.9	10.6
2.000	1.69E-10	5.69E-11	6.74E-11	9.8	10.2	10.2
3.000	7.98E-11	9.66E-11	1.14E-10	10.1	10.0	9.9
4.000	1.12E-10	1.12E-10	1.12E-10	10.0	10.0	10.0
5.000	9.24E-11	7.09E-11	7.09E-11	10.0	10.1	10.1
19.000	4.12E-11	3.78E-11	4.29E-11	10.4	10.4	10.4
47.000	8.74E-12	1.01E-11	1.01E-11	11.1	11.0	11.0
90.000	3.78E-12	1.96E-11	1.51E-11	11.4	10.7	10.8

Time (d)	pH			eH (mV)		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
0.083	10.0	9.9	10.0	376.7	398.7	366.8
0.292	10.0	9.8	10.0	376.0	403.0	399.0
1.000	10.7	10.6	10.4	415.0	403.6	404.5
2.000	10.6	10.7	10.5	377.0	402.1	402.0
3.000	10.9	11.1	11.1	416.5	411.2	411.0
4.000	10.8	10.9	10.9	403.5	412.0	407.5
5.000	10.7	10.7	10.7	412.0	402.7	413.0
19.000	11.7	11.7	11.6	418.0	414.0	419.0
47.000	11.3	11.2	11.4	409.0	411.0	416.9
90.000	11.3	11.6	11.7	406.5	404.3	408.4

Appendix F

Carbon Additive to Reduce Migration of VOCs

Appendix F

Carbon Additive to Reduce Migration of VOCs

Scope and Objectives

This report addresses the feasibility of using powdered-activated carbon (PAC) as an additive to a barrier grout wall to reduce the migration of carbon tetrachloride (CCl₄), perchloroethylene (PCE), trichloroethane (TCA), and trichloroethylene (TCE). The report addresses the following four topics:

- PAC potential to effectively remove CT, PCE, TCA, and TCE volatile organic compounds (VOCs) from the vapor phase
- Postulated effect of PAC as a grout additive
- Postulated grout PAC concentration needed to accomplish positive effects
- Anticipated long-term waste form stability.

PAC Potential to Effectively Remove CT, PCE, TCA, and TCE Contaminants from the Vapor Phase

The potential effectiveness for activated carbon to treat vapor contaminated with CT, PCE, TCA, and TCE is a complicated undertaking and requires (at a minimum) examination of:

- Each component vapor phase and water vapor concentration.
- The equilibrium adsorption relationship for each material in the vapor phase and activated carbon (called an adsorption isotherm).
- A multicomponent model to accurately predict how the components interact and the expected sorption capacity of each compound at equilibrium.
- A dynamic model to relate mass transfer from the diffusing gas into the carbon, used to determine the time needed to remove the vapors and how much contact is needed with the activated carbon.

To assess PAC potential, the adsorptive capacity for the contaminant on activated carbon could be compared to components that are currently removed from the gas phase with carbon. Nyer et al. (1996) present carbon adsorption capacity information for five compounds that are effectively treated:

Table 1. Adsorption Capacity (pounds compound per 100 pounds activated carbon).

Compound	At 10 ppm _v	At 100 ppm _v
Benzene	13	19
Carbon Tetrachloride	20	33
Methylene Chloride	1.3	2.7
Toluene	21	27
Trichloroethylene	19	33

Benzene and toluene are components of petroleum products; they are commonly removed from the gas phase with activated carbon as a result of remediation efforts. This comparative information indicates that at a concentration of 10-ppm_v, CT and TCE adsorption is similar to toluene and considerably greater than for benzene. At 100-ppm_v, CT and TCE adsorb better than both toluene and benzene. These data indicate that powdered activated carbon potential for removing CT and TCE is quite good.

Information is needed for PCE and TCA to compare with the other compounds. Isotherm data were not available for all the components on the same activated carbon, but information is available for TCE and PCE on BPL activated carbon (Crittenden, et al., 1989). They demonstrate an isotherm of a single component that covers a wide range of equilibrium concentrations can be used to define the activated carbon adsorption performance (toluene was used in their study). This isotherm can be used to predict the gas phase adsorption of TCE and PCE using the Dubinin-Radushkevich (D-R) equation [see appendix for the equation and brief discussion]. Using the D-R equation, the TCE and PCE isotherms of Crittenden et al (1989), and the molecular weights, liquid densities, and vapor pressures of TCE, PCE, CT and TCA, the adsorption isotherms for CT and TCA were predicted.

The experimental PCE and TCE isotherms indicate that PCE is considerably more adsorbable than TCE. Since TCE is effectively removed from the gas phase, PCE should have an even greater adsorption capacity on activated carbon. The projected isotherms for CT and TCA are nearly identical, with both projecting greater ability to sorb than TCE. From Table 1, CT was projected to sorb slightly better than TCE at the lower concentration and about the same at the higher concentration—the same trends can be observed with the projected isotherms. These data all indicate that activated carbon adsorption can effectively remove all four volatile organic contaminants from the vapor phase.

Multi-component adsorption equilibrium modeling and the dynamic mass transfer modeling required to evaluate CT, PCE, TCA, and TCE mixtures is beyond the scope of this project, largely because of the extensive computer programming and modeling requirements. Literature information can be used to assist in determining how multi-component equilibrium adsorption will be affected by additional components and with water vapor present. When both TCE and PCE are present (Crittenden, et al., 1989), the presence of the other lowers the equilibrium loading of the other component on the adsorbent. The presence of TCE reduces the amount of PCE the carbon can adsorb and the PCE reduces the amount of TCE adsorbed. Since the PCE is the more strongly adsorbed component, the amount of TCE adsorbed would be more adversely affected by PCE than the PCE adsorption when the vapor phase concentrations are the same. Water vapor has the same negative effect on sorption as does a competing organic compound (Nyer et al. 1996 and Crittenden et al. 1989). When the gas phase concentration of the TCE in these studies was high (> 4,000 ppm_v), the reduction in amount adsorbed was small; when the TCE concentration was small, an 85% reduction was observed.

Literature information provides insight into the dynamic sorption of multiple contaminants in an adsorption column treating air stripper off-gas (Mueller and DiToro, 1993). Considering the breakthrough curves of CT, PCE, TCA, and TCE, TCA exited the column slightly ahead of CT, which was immediately followed by TCE. PCE broke through the adsorber last. Using the isotherms above, the compound with the greatest amount adsorbed at a particular concentration (PCE) is expected to breakthrough last. TCE was expected to breakthrough the adsorber first. Mass transfer must also play an important role in the process. TCE diffusivity in the gas phase ($8.3 \times 10^{-2} \text{ cm}^2/\text{s}$) is greater than that for TCA and CT (both are $8.0 \times 10^{-2} \text{ cm}^2/\text{s}$) and is likely to cause it to adsorb faster on the carbon. Since TCA and CT sorption is not substantially greater than that of TCE, that energy of sorption difference may not have been substantial enough to displace the TCE. An additional complicating factor is the difference in gas concentrations of the different species. Even though PCE diffusivity is smaller than the other components, its adsorption equilibrium capacity is considerably greater than the other components. This substantial difference in adsorption strength is the main reason its breakthrough is last.

Except for the last component in the gas stream to exit the adsorption column, PCE in this case, each component is displaced from the activated carbon. Immediately after component breakthrough, the effluent concentration continues its breakthrough and the effluent concentration is greater than the influent concentration. To prevent these higher concentrations in the effluent than in the influent, adsorber operation would have to be terminated prior to the weakest materials breaking through the adsorber. Dynamic modeling and the pilot study demonstrate that complete removal, within detection limits, is possible with TCA likely to breakthrough the column first. Estimates of the adsorption cycle run time are very important for the control of treated gas concentrations.

Postulated Effect of PAC as a Grout Additive

The literature is sparse on the addition of materials to prevent the gas phase migration of organic compounds by sorption. The literature relates to contaminant sorption from the aqueous phase, or more accurately to the prevention of leaching from the solid into the aqueous phase. Organophilic clays and activated carbon have been tested. The activated carbon results are discussed to indicate its potential as an additive to the grout, keeping in mind that liquid phase application was tested and not the gas phase.

One study evaluated PAC for the adsorption of phenol, aniline and naphthalene (Hebatpuria, et al.; 1999a & b). In their research, sand was contaminated with phenol and allowed to age, and then PAC was added as a percentage of the sand weight, and the cement and water added to this mix. Leaching tests were performed on the solidified/stabilized soil using both the Toxicity Characteristic Leaching Procedure (TCLP) and the American Nuclear Society (ANS) 16.1 test. PAC dramatically reduced the leaching of phenol and aniline (Hebatpuria, et al.; 1999b), indicating significant potential in reducing leached contaminant concentrations to acceptable concentrations. The inability of PAC to reduce naphthalene leaching is a concern. It should be recognized that naphthalene did not leach significantly during the TCLP test (sample without PAC leached 1.3% of the naphthalene, whereas for phenol and aniline the values were 65% and 26% respectively); PAC didn't dramatically reduce the amount of leaching that occurred. A more detailed study of phenol adsorption determined that phenol sorption appeared irreversible (Hebatpuria, et al.; 1999a). Phenol desorption is a concern, since mixing cement with activated carbon increases the pH of the mix to above 12; phenol has a pK_a of 10, so at this pH the phenol becomes phenate anion which is substantially less adsorbable. Mixing the phenol contaminated sand, water, and cement simultaneously did not adversely affect adsorption, so the adsorption process is rapid and isn't adversely affected by the pH swing during the hydration of the cement. Changes in crystallization of the cement mixture was noted when PAC was present, probably from an accelerated hydration of the cement and less formation of $Ca(OH)_2$ gels.

PAC was added to sand and the mixture evaluated as a permeable-barrier media to remove benzene from groundwater (Rael et al. 1995). Several sorptive additives were evaluated, with PAC performing best and therefore studied more extensively. A column test with an empty-bed-contact-time of 350 minutes and 3% PAC removed 40-mg/L of benzene to less than its detection limit, with the PAC use rate slightly better than predicted in adsorption isotherm tests. This study does indicate that a classic breakthrough curve does develop and nearly theoretical carbon use is obtained with a fluid flowing through a permeable barrier.

Postulated Grout PAC Concentration Needed to Accomplish Positive Effects

The PAC concentration to be added will depend upon:

- VOC concentrations in the vapor phase,
- mass of waste material encapsulated by the grout,

- propensity of the VOCs to sorb onto the PAC,
- rate at which the each VOC diffuses into the barrier,
- ability of each VOC to sorb onto grout, soil and other materials present in the waste cell,
- ability of other reactions to degrade and destroy each VOC,
- presence of water within the cell and its affect on vapor phase concentration and adsorption to PAC.

Other than the mass of the material trapped by the barrier grout wall and the ability of the VOCs to sorb, the other factors require assumptions that could vary by at least an order of magnitude. In an effort to estimate the barrier ability to sorb VOCs, the following assumptions were made:

- VOC concentration in the vapor phase can be calculated:
 - At equilibrium
 - Without water present
 - Using the fugacity approach (Mackay, 1979)
- All VOC movement results from a concentration gradient from the vapor phase concentration calculated in the waste cell; these concentrations at the barrier wall are assumed to remain constant. [In other words, within the main cell, diffusion is not restricted and the vapor phase concentration will be immediately replaced as the VOCs diffuse into the barrier wall and is sorbed in the PAC. Actually the contaminant concentration adjacent to the barrier would decrease; additional time would be required to replace the VOCs, and therefore this estimate is conservative.]
- The sorption mass transfer zone is 5 cm with the concentration decreasing from the main cell VOC concentration to zero using Fick's law of diffusion.
- VOC diffusivity is substantially less than the gas phase diffusivity. TCE diffusivity in soil with a porosity of 0.29 was measured to be $2.5 \times 10^{-4} \text{ cm}^2/\text{s}$ while the gas phase diffusivity is $8.3 \times 10^{-2} \text{ cm}^2/\text{s}$ (Hutter et al. 1992). Other VOC diffusivity values will be adjusted proportionately to the TCE values.
- The interior surface of the PAC is not blocked by the grout; i.e. all the adsorptive capacity of the PAC can be used.
- Internal diffusion is much faster than the diffusion of VOCs to the external PAC surface; therefore, the PAC reaches equilibrium rapidly.
- Maximum amount of PAC is $0.053\text{-g}/\text{cm}^3$ in the barrier wall; the value could be greater but this in line with the values used by Hebatpuria et al. (1999 a,b).
- VOCs do not sorb on nonPAC materials; even though VOCs are likely to sorb on the other materials, therefore this will overestimate the quantity of material going to the barrier wall.
- No VOC destruction mechanisms are present.

- Barrier thickness is 3-ft.

Fugacity calculations using assumption (1) resulted in the following equilibrium gas phase concentrations:

Compound	Concentration g/L
CT	0.490
PCE	0.023
TCA	0.001
TCE	0.081

The projected vapor phase concentration of CT is greatest, followed by TCE; PCE is significant and TCA is small. The flux of each component into the barrier was determined based on Table 2 and assumptions (2), (3), and (4). The total flux is estimated at 2.9×10^{-4} g/day/cm².

Multi-component modeling of the equilibrium situation is beyond the scope of this report and because the uncertainties are not likely to aid the estimates. TCE adsorbs the least of the compounds and CT was found to breakthrough a column treating air stripper off-gas before CT (Mueller and DiToro, 1993). The solid phase loading at these high influent concentrations is about 0.6-gVOC/gPAC based on the D-R equation and assumptions (5) and (6). This high loading is near the maximum amount that can be sorbed on the PAC and therefore would depend upon the PAC.

Using this information and assumptions (7) through (10), the barrier would be expected to last 30 years before VOCs started to breakthrough the barrier. This may not be satisfactory, but many assumptions could easily be varied by an order-of-magnitude.

- For instance, instead of using pure VOCs in equilibrium with the gas phase to determine the concentrations in Table 2, the gas phase concentrations would be reduced by a factor greater than 10 by including water. The reduced VOC transport rate would be countered with a reduced equilibrium concentration on the PAC resulting from lower VOC concentrations (about a 20% reduction in capacity) and water vapor competing for space. It is unlikely that these two considerations would reduce PAC capacity for the VOCs by 90%; therefore the life expectancy of the PAC barrier would increase.
- The VOC diffusion rate within the barrier and within the main cell could be substantially smaller. This could result from a smaller solid phase porosity or VOC sorption on the solid matrix—the overall effect would reduce the VOC transport to the PAC barrier.

Anticipated Long-Term PAC Stability in Grout

The PAC and sorbed VOCs should be quite stable, with chemical degradation and/or biological activity having little effect on the PAC. Reactions could cause VOC degradation, but they would not be expected to occur either. VOC desorption could occur if a high concentration of a competing organic compound were exposed to the barrier or if the barrier temperature were increased substantially; again, this would not be expected.

The main concern would be the constant influx of VOCs from the main cell. Once the PAC capacity is completely exhausted with the weakest adsorbing contaminant, TCE, the continuing inflow of

the mixture of contaminants would permit PCE to displace TCE. High TCE concentrations could then diffuse from the barrier to the surrounding area, with all but the most strongly adsorbed material eventually being displaced from the adsorbent.

Summary

Addition of PAC to the exterior barrier confining the bulk of the waste could reduce the target VOC concentrations to very low concentrations. Using several conservative assumptions, this barrier is expected to be effective for approximately 30 years. After that time, the weakest adsorbed VOCs could be displaced by a more strongly adsorbed VOCs, and the displaced VOCs would enter the vapor phase outside the cell. While the 30-year life may not appear good, there are two main reasons to think this may be underestimated by a factor of 10 to 100:

- The equilibrium vapor phase concentrations are very high. Including water in the estimates would reduce these values by at least an order of magnitude. Sorption of the VOCs to the solid matrix within the cell could also reduce these concentrations by an order of magnitude. Both these effects would drastically reduce the amount of VOCs being transported to the barrier and the PAC. Vapor phase VOC concentrations need to be determined for the main cell design.
- Effective diffusivities of the VOCs in the main cell and within the barrier may greatly reduce the transport of VOCs. The values used were deduced from gas phase values and correspond to transport in soils with 29% porosity. If the main cell and the barrier porosities are smaller and if the materials are retarded in their movement by constant sorption/desorption on the solid matrix, the amount of VOCs transported to the barrier and the PAC would be substantially reduced. Effective VOC diffusivities need to be experimentally determined for the main cell and barrier wall materials.

There is one main reason why the estimate could be optimistic: The matrix surrounding the PAC could block access to the activated carbon microspore surface area and prevent sorption from occurring. This would drastically reduce the sorptive capacity of the PAC and prevent VOC sorption. PAC needs to be imbedded into the barrier matrix and adsorption equilibrium studies determined.

Dubinin-Radushkevich (D-R) Equation

$$q = \left[\frac{W_o \rho_1}{MW \cdot 10^{-6}} \right] \exp \left[\frac{-B}{\beta^2} \left(RT \ln \frac{P_s}{P} \right)^2 \right]$$

Where:

q = the solid phase concentration of the VOCs ($\mu\text{mol/g}$ carbon);

W_o = the maximum adsorption space of the adsorbent (cm^3/g);

B = the microporosity constant ($\text{mol}^2/\text{cal}^2$);

ρ_1 = liquid density of the pure VOCs (g/cm^3);

MW = the VOC molecular weight;

β = the affinity coefficient of the VOCs (dimensionless);

P_s = VOC vapor pressure (mmHg);

P = partial pressure of VOCs (mmHg);

R = gas law constant (1.986-cal/mol/°K); and

T = the temperature (°K).

This equation can be used to estimate an adsorption isotherm for a compound adsorbed onto an adsorbent from the gas phase. To use this equation, an adsorption isotherm has to be determined over a wide range of concentrations for a single chemical – referred to as the characteristic curve for that particular adsorbent. The characteristic curve and the reference compound molecular weight, liquid density, and vapor pressure are used to define the W_o and B . The β is an additional correction factor needed to predict another compound isotherm (for the reference compound β is 1). β depends on a compound molar volume, parachor, or polarizability relative to that of the reference compound; while all are claimed to work, different situations result in one working better for certain families of compounds. The D-R equation was used to predict the CT and TCA adsorption data, with both the TCE and PCE data used to generate the characteristic curve with molar volumes used to calculate the β .

References

- Crittenden, J.C.; et al.; "Predicting Gas-Phase Adsorption Equilibria of Volatile Organics and Humidity," *Journ. Environ. Engr*, 115, 3, 560-573, 1989
- Hebatpuria, V.M.; et al.; "Immobilization of Phenol in Cement-based Solidified/stabilized Hazardous Wastes Using Regenerated Activated Carbon: Leaching Studies," *Journ. Hazardous Mat'ls*, B70, 117-138, 1999a
- Hebatpuria, V.M.; et al.; "Leaching Behavior of Selected Aromatics in Cement-based Solidification/Stabilization under Different Leaching Tests," *Environ. Engr. Sci.*, 16, 6, 451-463, 1999b
- Hutter, G.M.; G.R. Brenniman; R.J. Andersen; "Measurement of the Apparent Diffusion Coefficient of Trichloroethylene in Soil," *Water Environ. Res.*, 64, 1, 69-77, 1992
- Mackay, D.; "Finding Fugacity Feasible," *Environ. Sci. & Technol.*, 13, 10, 1218-1223, 1979
- Mueller, J.A.; D.M. DiToro; "Multicomponent Adsorption of Volatile Organic Chemicals from Air Stripper Offgas," *Water Environ. Res.*, 65, 1, 15-25, 1993
- Nyer, E.K.; et al.; "Air Treatment for In Situ Technologies," *In Situ Treatment Technology*, CRC Press Inc., Boca Raton, FL, pp. 227-231, 1996
- Rael, J.; S. Shelton; R. Dayaye; "Permeable Barriers to Remove Benzene: Candidate Media Evaluation," *Journ. Environ. Engr*, 121, 5, pp. 411-415, 1995
- Rho, H.; "Decomposition of Hazardous Organic Materials in the Solidification/Stabilization Process Using Catalytic-Activated Carbon," *Waste Management*, 21, 343-356, 2000.

Appendix G

Evaluation of Void Space in a Pit

Appendix G

Evaluation of Void Space in a Pit

Based on a rough order of magnitude for estimating purposes, the total available voids in the pit are estimated by analysis. For the organic drums there is only about 15% voids, for the inorganic there could be as high as 33% voids, for the nitrate salts 33%, for the boxes 70-80%, for the combustible drums 40-70% voids, and for the surrounding soils 40-50% voids(including bridging effects). Now looking at the test plan (Grant) table2 "volume fractions of buried transuranic waste" the combustible drums are about .536, the organic sludges are about .059, and nitrate is .043 and the inorganic is .124, and the boxes full of asphalt/metal/cinder blocks and wood are about .238. If the soil is about 50% and the waste about 50% then the available voids can be estimated as follows (the volume percent of the pit times the volume percent of the waste times the estimated void volume in the waste or soil type).

Estimated void volume where V is the volume of the pit:

- Soil $.5 \times .5 = .25V$
- organic $.059 \times .5 \times .15 = .004V$
- inorganic $.124 \times .5 \times .33 = .020V$
- nitrate $.043 \times .5 \times .33 = .007V$
- combustibles $.5 \times .5 \times .7 = .17V$
- other boxes etc. $.5 \times .2 \times .7 = .07V$
- Total voids = $0.521V$

Now, allowing for some grout returns (look at the Loomis 1996 data for TECT), the amount of return is 50 gal for a pit, which is on a total pit volume basis: $(11/18) \times 1,615$ gal or $.05V$. Since most of these returns are neat grout, this $.05V$ can be added to the $.521V$ above to give a total expected grout injected of $0.571V$.

Now, finally, accounting for some compaction of soil around the pit due to the grouting action (conveniently say $.029V$) we get $0.6V$. In other words, 60% of the volume of the pit is the amount of voids expected in the pit.

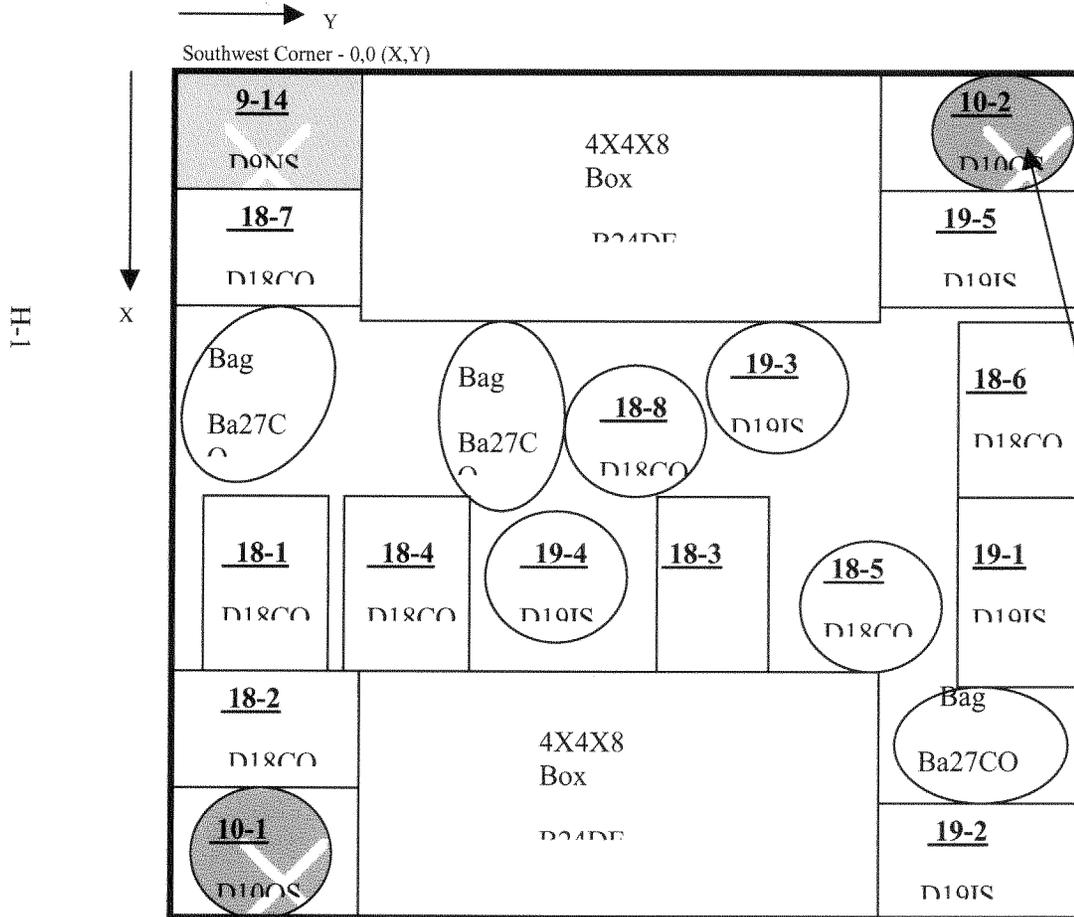
Appendix H

Grouting Pit Construction Details

Appendix H

Grouting Pit Construction Details

As-Built for LAYER 1, 0-2 FT., Includes
Numbers for Surrogate Waste Forms



DRUMS

D18CO =8

D19IS = 5

D10OS= 2, Metal

D9NS= 1, Metal

Scale 1 in. = 2.5 ft

CO-Combustibles

IS-Inorganic Sludge

OS- Organic Sludge

NS- Nitrate Salt

DE-Debris

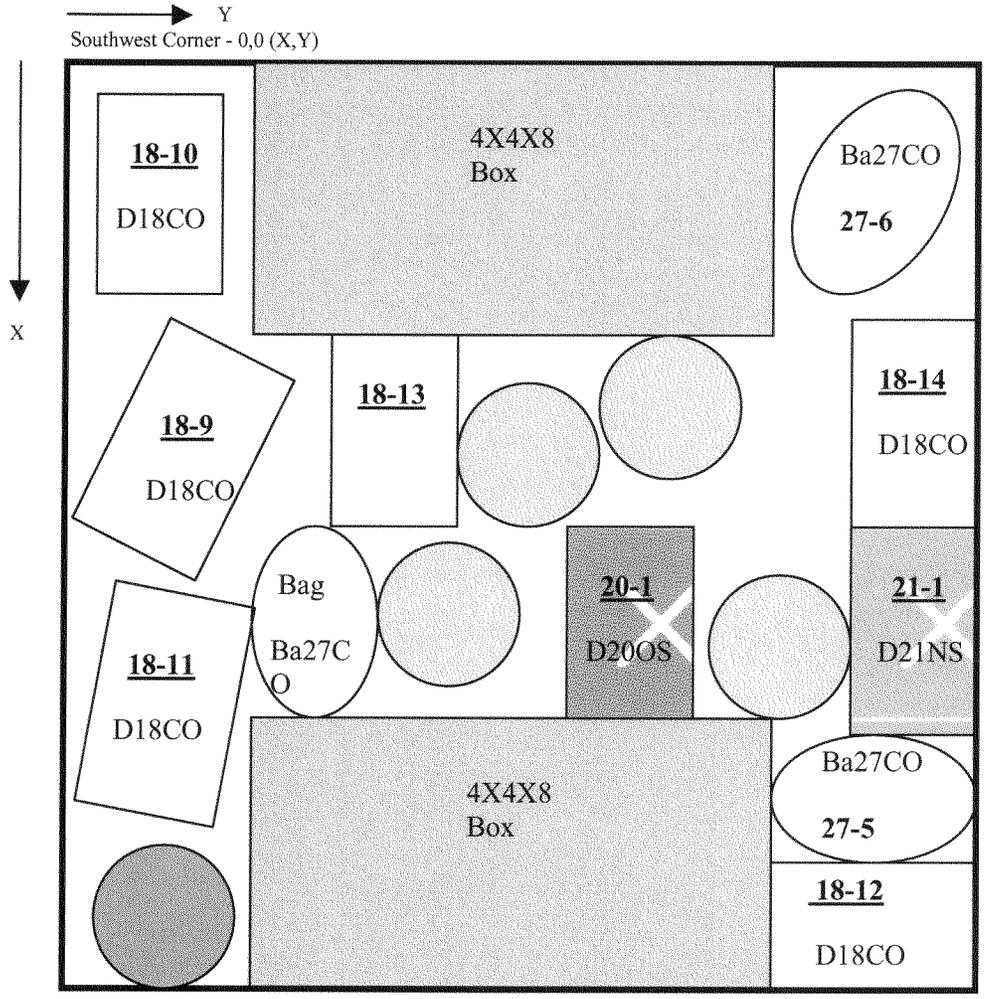
Drum 10-2 was installed in a Vertical Position instead of horizontal

As Built for LAYER 2, 0-4 FT., Includes Numbers for Surrogate Waste Forms



- DRUMS
 D18CO =6
 D20OS= 1
 D21NS= 1

Scale 1 in. = 2.5 ft

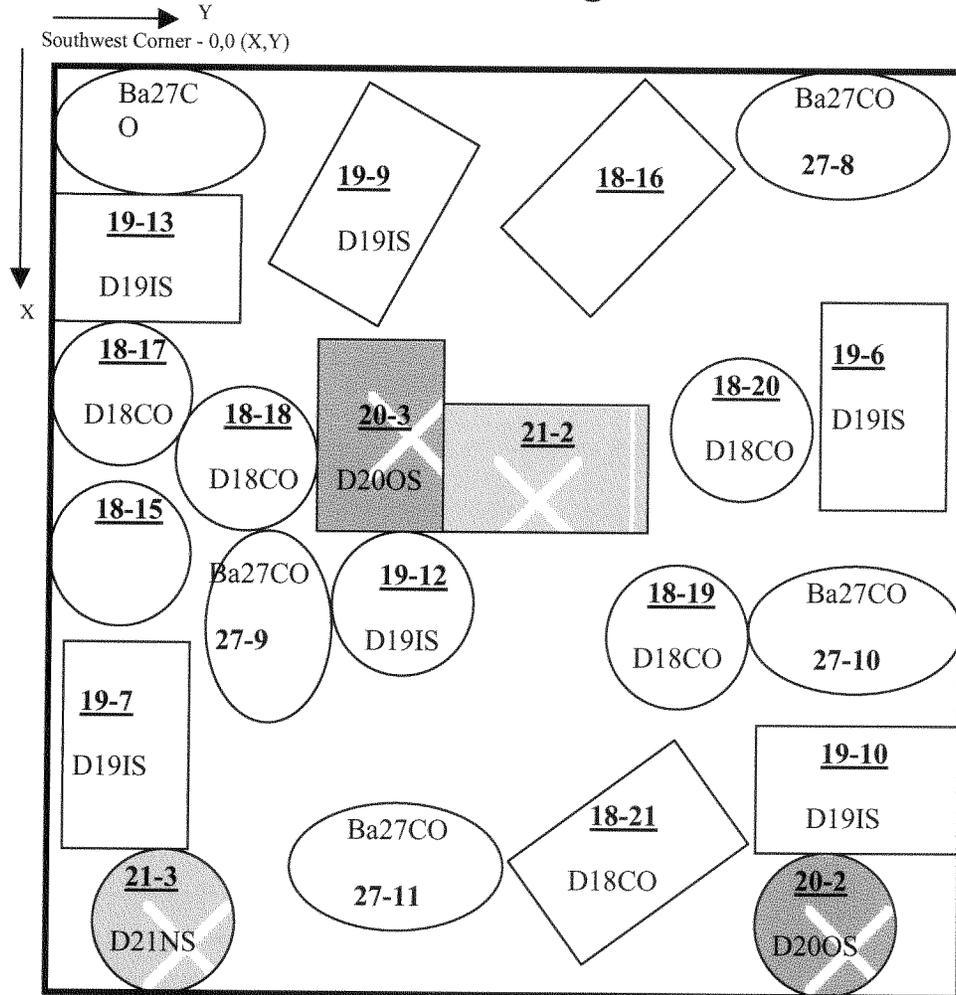


H-2

As-Built for LAYER 3, 0-6 FT., Includes Numbers for Surrogate Waste Forms



North



DRUMS

D18CO = 7

D19IS = 6

D20OS = 2

D21NS = 2

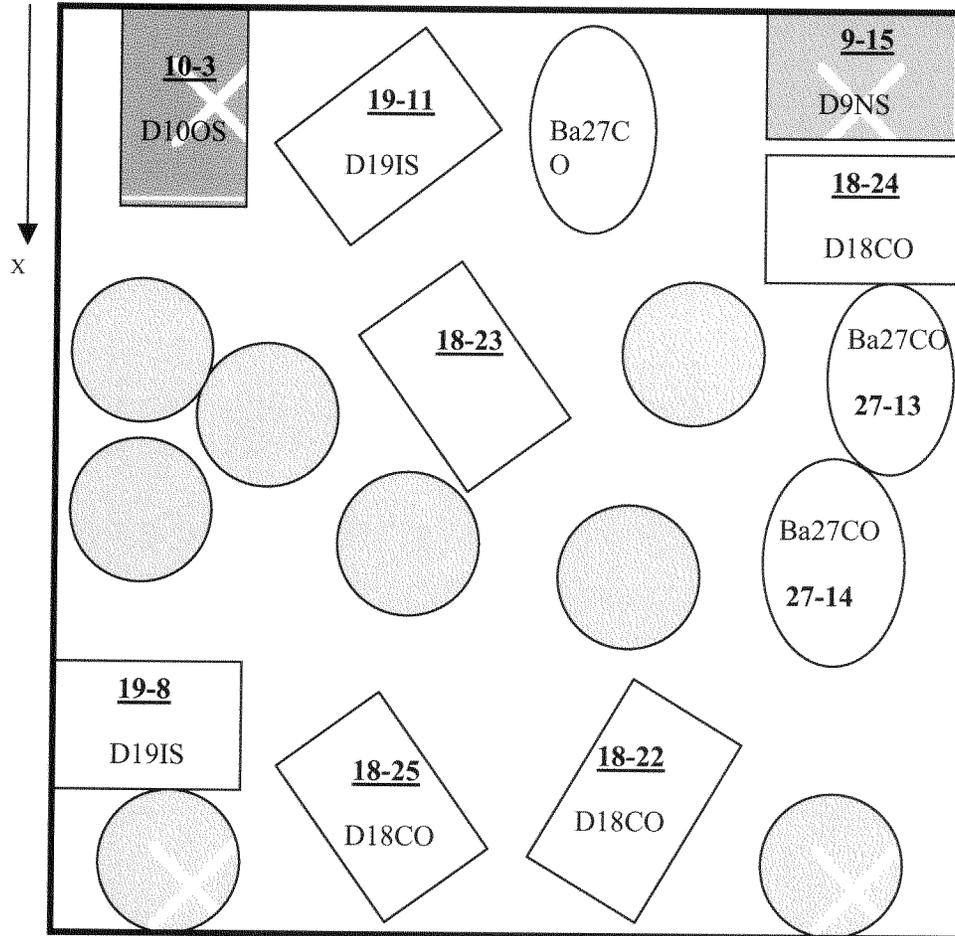
Scale 1 in. = 2.5 ft

As-Built for LAYER 4, 0-8 FT., Includes Numbers for Surrogate Waste Forms



North

→ Y
Southwest Corner - 0,0 (X,Y)



DRUMS

D18CO =4

D19IS = 2

D10OS= 1, Metal

D9NS= 1, Metal

Scale 1 in. = 2.5 ft

Appendix I

Conceptual Design and Cost Estimate

Appendix I

Conceptual Design and Cost Estimate

Introduction

This appendix presents an innovative conceptual approach to retrieving waste buried in transuranic pits and trenches at the Idaho National Engineering and Environmental Laboratory (INEEL) Radioactive Waste Management Complex (RWMC) Subsurface Disposal Area (SDA). In addition, the appendix gives a rough-order-of-magnitude cost estimate for applying the design in the SDA transuranic pits and trenches. The overall concept is to first grout the buried transuranic waste using the INEEL jet-grouting process to form a monolith, then retrieve the monolith, taking advantage of the agglomeration of contaminants caused by grouting.

Efforts in the 1970s to retrieve transuranic waste focused on contamination control problems as the most challenging aspect of the retrieval effort. These retrieval efforts included the early waste retrieval (EWR) in SDA Pits 1, 2, and 3,¹ and the initial drum retrieval (IDR) effort at SDA Pits 11 and 12.²

The EWR project used an archeological approach to retrieval using a small backhoe and personnel were in totally enclosed bubble suits. Many examples of plutonium spread were encountered and only hundreds of drums were removed.

The IDR project retrieved recently interred drums without spreading airborne contamination within a weather shield. During the IDR project, a backhoe was used to probe SDA Pits 6, 9, and 10 for possible applications of the open-air IDR technology. However, the plutonium/americium contamination on the backhoe was too high (up to E6 counts per minute) to effectively retrieve the waste with the open-air IDR approach.

During the late 1980s, a Pit-9 retrieval project was planned, and a preliminary design was produced that involved full-pit retrieval using double containment retrieval enclosures, remote operation of a variety of large excavating equipment, and an elaborate contamination control strategy.³ The project involved retrieving and packaging the waste and storing the waste in Resource Conservation and Recovery Act (RCRA)-approved containment buildings. The project was stopped when the SDA transuranic pits and trenches were placed on the National Priorities List as part of the Comprehensive, Environmental Response, Compensation, and Liability Act (CERCLA) which demanded examining a wide variety of technologies prior to applying retrieval as the remediation of choice.

During the 1990s, a series of transuranic waste retrieval projects was examined as developmental research under the Department of Energy Technology Development program. Both conventional and innovative transuranic waste retrieval concepts were examined, including basic retrieval with heavy equipment,⁴ cryogenic retrieval (freezing the waste, followed by remote retrieval),⁵ and direct remote retrieval.⁶ Following a series of studies to determine the spread of contamination, it was concluded that conventional retrieval of waste containing finely divided plutonium and dry INEEL soils would be very difficult.^{7,8,9} The concept of applying cryogenic freezing of the waste followed by retrieval was highly effective, but the cost and complexity of freezing the waste was a drawback. Grouting the waste followed by retrieval was a technology similar to the cryogenic approach; however, the grouting process simulated the agglomeration provided by the freezing process without the complexity of freezing the waste. With this new concept, the innovative grout/retrieval technology was tested as a Technology Development project using ordinary Portland cement as the grouting agent with a positive proof of concept.¹⁰

Subsequent studies of grout followed by retrieval, using other grouting agents, including acrylic polymer¹¹ and a paraffin material called WaxFix.¹² During retrieval of a test pit injected with molten Waxfix, observations confirmed that the molten wax had an acceptable degree of penetration into the waste, thus providing extraordinary agglomeration of contaminants and allowing easy retrieval. Because of the positive proof-of-concept, this technology is currently part of INEEL Waste Area Group 7-13/14 CERCLA Treatability Study.¹³

This engineering design file offers a conceptual design of the grouting/retrieval process as a basis for a rough order of magnitude cost estimate. The conceptual design starts with a list of major programmatic assumptions, the main assumption being that full-pit retrieval will be followed by processing for “retrievable disposal” or, alternatively, interim storage. The design assumes that the retrieved and repackaged waste will be treated with a nonthermal encapsulation process and then re-deposited in the original pit or, trench or as an alternative, placed in RCRA-approved storage. This engineering design file estimates the cost for a CERCLA treatability study on a small portion of a transuranic pit or trench and for retrieval of material in WAG 7 13/14 transuranic pits and trenches at the RWMC SDA.

Preconceptual Design

Five distinct phases comprise the grout/retrieval/disposal process, which follows a complete “cradle-to-grave” approach with no deferred decisions on final disposition of the waste. The first phase is site preparation; the second phase is the grouting process; the third phase is retrieval and packaging for transportation to an accumulation area; the fourth stage is transportation and processing at an encapsulation plant; and the fifth stage is reburial of the processed waste with improved confinement in the pits and trenches from which it was removed or optionally placed in interim, above-ground, RCRA storage. Because this process has never been applied, assumptions must be made to anticipate unknown factors in the process. For instance, the process assumes a final logical disposal site for the waste. This approach would require discussion with state of Idaho and local interests groups; however, the concept eliminates interstate agreements which are proving difficult if not impossible to effect.

Furthermore, this preconceptual design eliminates the need to segregate the waste into transuranic waste; transuranic-contaminated, low-level waste; alpha mixed low-level waste; hazardous waste, and clean soil. Rather, all of the waste is treated as one class—retrieved transuranic pit or trench waste. By eliminating segregation, the need for technically questionable assay systems is also eliminated. The assay of heterogeneous retrieved waste may not be possible at any cost. A cost savings from waste minimization would be trivialized by the cost of an assay system that could measure heterogeneous waste within ± 1 nCi/g.

The overall concept of this process eliminates, to the extent possible, deferred or assumed solutions for the final waste material. The retrieval and treatment process requires identification of realistic final disposal options before completing the design. The design, in turn, accommodates the chosen paths for the waste. Therefore, the conceptual designs offered in this engineering design file make reasonable assumptions for the final disposition of the waste (i.e., the waste will go to a specific site rather than an undetermined offsite disposal site). An additional guiding principle is that the waste will be removed and, to the extent possible, repackaged in an improved form (such as, encapsulated in polyethylene) that is suitable for reburial in the very site from which it was removed. Any retrieval and reburial process of the transuranic waste will require innovative negotiations among interested parties and possible exemptions from portions of existing environmental laws. Exact adherence to all laws could preclude accomplishing remediation of transuranic pits and trenches; therefore the preconceptual design offered here obviously is contradicting to some existing laws.

Programmatic Assumptions

Major assumptions for the grout/retrieval/disposal process are:

- The project will be conducted in two steps: (1) a treatability study involving processing a 1-acre pit, and (2) remediation of 9 acres of transuranic pits, using equipment from the treatability study.
- The design will support a 7-year retrieval and reburial (retrievable disposal) in nine acres of the transuranic pits and trenches at the INEEL SDA.
- CERCLA and RCRA will apply to the extent possible, and any deviations or exemptions will be agreed upon by interested parties. In addition, the initial full-pit retrieval will be performed under an interim Record of Decision.
- All of the retrieved and encapsulated waste will be placed in shallow land burial within the pit from which it came. The treated waste can be retrieved in the future for other considerations, if required. As an option, the retrieved and encapsulated waste can be placed in interim RCRA storage rather than "retrievable disposal."
- The entire quantity of retrieved stored waste will be encapsulated with advanced encapsulation schemes involving further size reduction and polyethylene mixing.
- No assay of the waste will be performed, other than that necessary to control contamination spread during the entire process; rather, the waste will be treated as a special case: retrieved buried transuranic waste for processing and reburial.
- The process will require further size reduction prior to encapsulation with off-the-shelf devices.
- Following use, the weather shield complex will only be slightly contaminated and can be buried in one of the last pits excavated as low-level waste.

Design Features

Major design features are site preparation, grouting, retrieval, accumulation area, encapsulation area, and finally either retrievable disposal or interim storage. Each of these features is presented below.

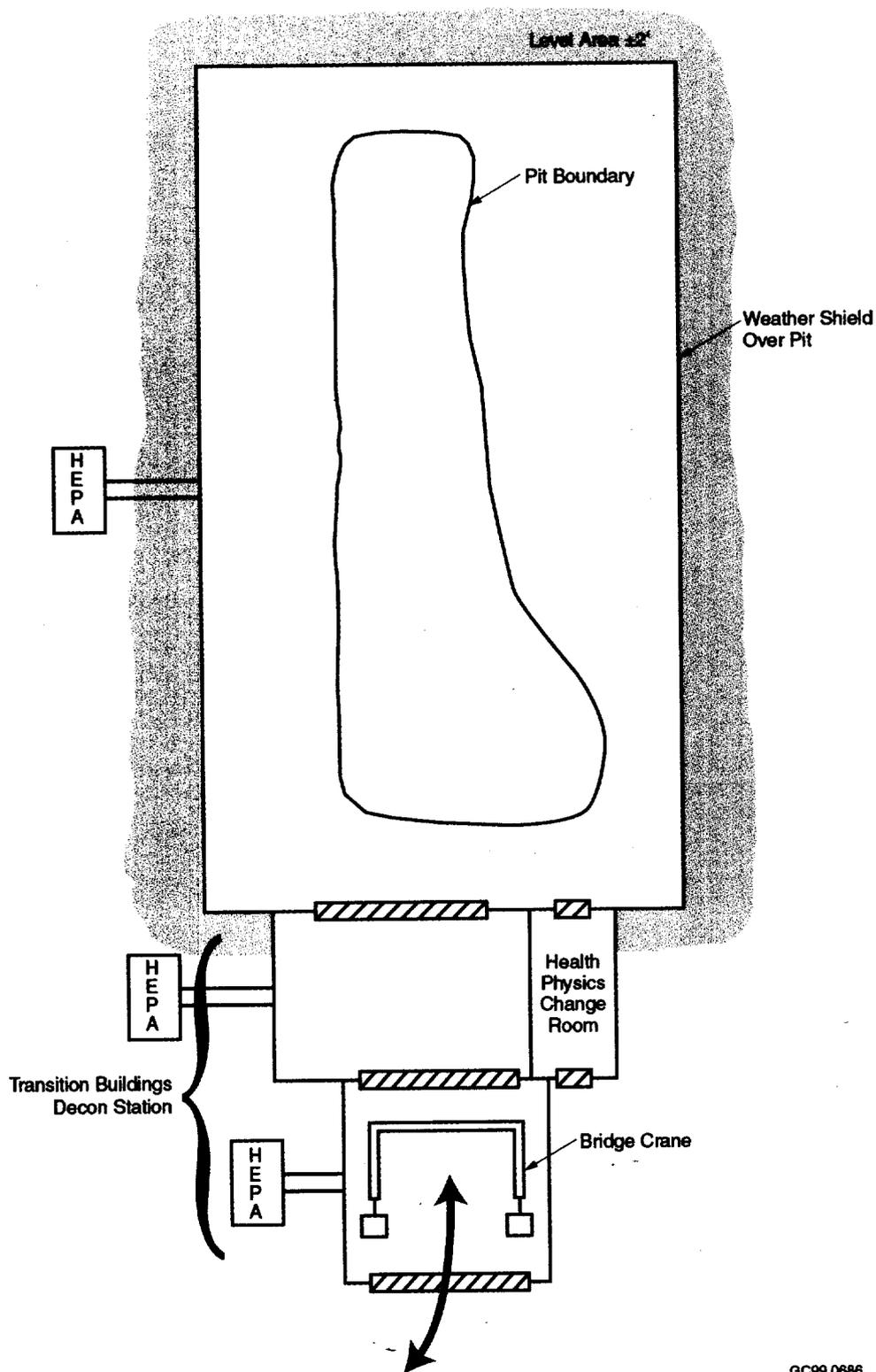
Site Preparation

Site preparation includes preparing the top surface of the pit and placing temporary movable weather shields and ancillary adjacent buildings for the grout/retrieval campaign.

The top surface of the site will be leveled to ± 2 ft over the entire pit, with the assumption that the top 2 ft overburden is clean soil. This soil will be stockpiled within the weather shield to be used in the grouting process as a berming material. Next a series of three adjacent temporary structures will be erected over the pit. One of the structures is assumed to be a SPRUNG structure (approximately 168 \times 400 ft) connected to two packaging/decontamination/transition buildings (approximately 50 \times 50 ft). All buildings will have a slight negative pressure and air flow will be filtered by high-efficiency particulate air (HEPA) filtration systems as shown in Figure 1.

Grouting Phase

Grouting equipment will be placed in the building covering the pit. This equipment will consist of a movable, lightweight, remotely-operated platform (like an x-y positional system) fitted with a drilling apparatus equivalent to a CASA GRANDE C-6 or C-8. The platform will be placed on a bermed area covering about 400 ft² (20 \times 20 ft) of the pit. Drilling equipment on the platform will include the hydraulic equipment and drill mast but not the transport tracks and motor. A separate hydraulic motor will be assembled in the weather shield from which grouting will be monitored.



GC99 0686

Figure 1. Buildings will have a slight negative pressure, and air flow will be cleaned by HEPA filters.

On a predetermined grid pattern, molten Waxfix (including 1g/L of boron to eliminate criticality concerns) will be injected into the waste. From past experience,¹² copious grout returns are anticipated within the confines of the bermed area that will form a reservoir of molten Waxfix. Once the area under the platform has been completely grouted, the platform will be moved by forklift to a new position. By continuing this process, an acre-sized pit can be grouted in a 2-year period, working one shift. Figure 2 shows the overall grouting process with tanker truck access for the delivery of the molten paraffin.

Retrieval Phase

In the conceptual design, the retrieval phase is performed after the entire pit has been grouted. There is an option for the retrieval operation to start after the pit is about half grouted and the delivery of paraffin can be accomplished via side or opposite end access. The retrieval operation requires a sealed, positive pressure cab, heavy excavation equipment, including a large front-end loader/backhoe with Baldersom thumb, a heavy duty fork lift, and a large track mounted shear. The fixation of aerosolizable particulate caused by the Waxfix grouting allows a “sealed cab” approach.

The retrieval will be performed from the bottom via an access ramp as shown in Figure 3. Waste will be retrieved and partially sized at the dig face. There will be no dig face diagnostics; rather, the retrieval equipment will be chosen to accommodate the largest objects buried in the pits. Intact, large, metal vaults will be sized at the dig face, as will large tanks and even vehicle bodies, if encountered. The presized materials will be focused through a funnel into a series of disposable polyethylene 4 × 4 × 8 ft boxes. Reusable contamination control mats will be placed adjacent to the dig face and the heavy equipment and forklift will be operated on these mats. Mats will be added as the dig face is advanced. Objects that cannot be size reduced at the dig face will be evaluated for beta gamma activity and may be declared special case low-level waste material that will be left in the pit. In special cases, the object will be manipulated to the side of the pit such that further retrieval is not impeded.

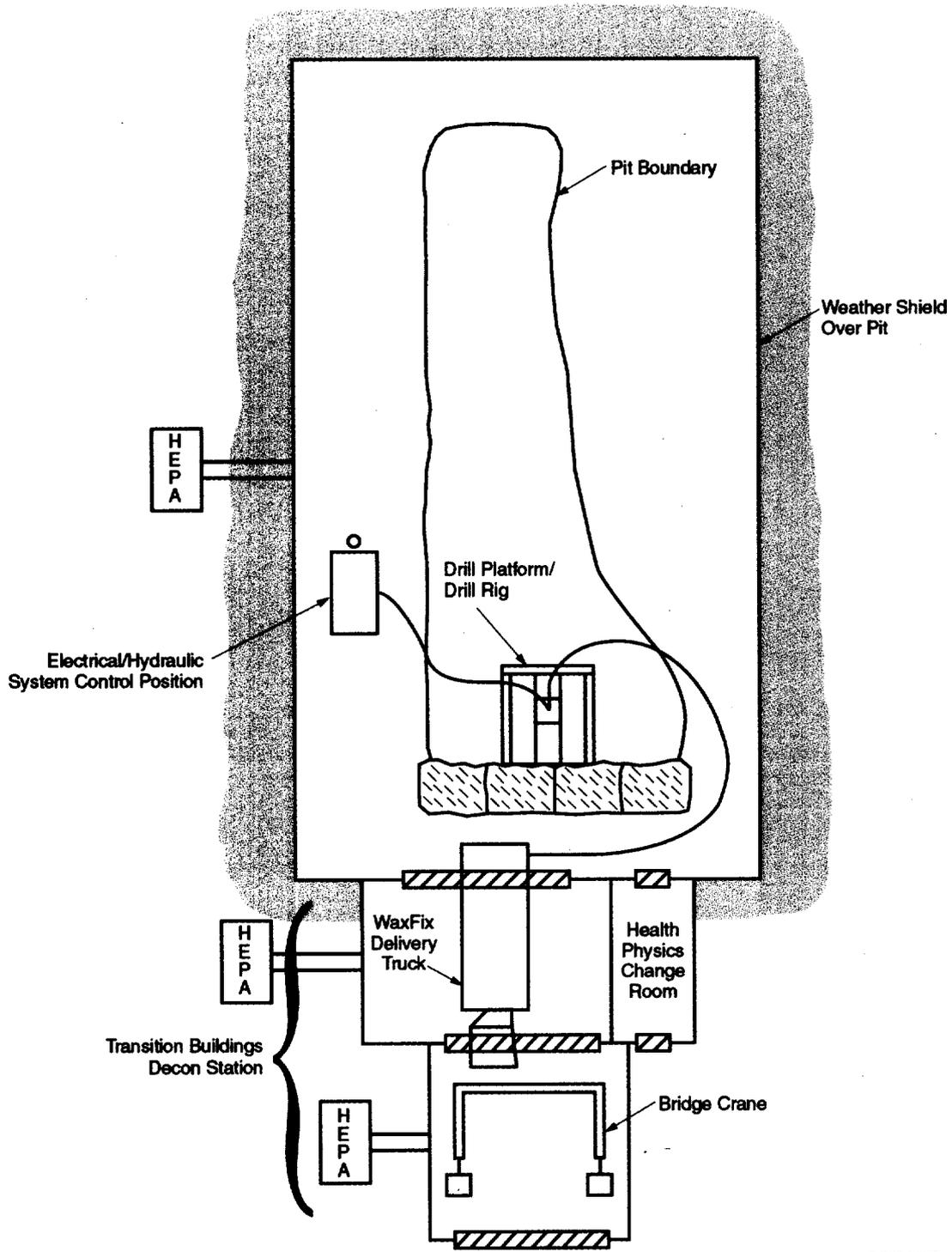
The fork lift will be used to remove the boxes from the dig face to the packaging/decontamination area in the adjoining building. In the packaging area, a shrink wrap will be applied in duplicate to the transporter box and then the boxes will be placed on a semi-tractor open trailer system for transporting to the accumulation storage building and eventually to the encapsulation facility.

Accumulation Area

A RCRA-approved interim (surge) storage accumulation area will be provided for a lag between the retrieval and the encapsulation phase of the operation (see Figure 4). This surge storage is necessary because the retrieval rate of waste may be faster than the encapsulation rate. Also in the event of an extended shutdown to repair retrieval equipment, the storage will be weather protected until the encapsulation facility can process the inventory. The surge storage building design will be similar to the transuranic waste storage building currently used at the RWMC.

Encapsulation Phase

The encapsulation phase, if used, improves the confinement of the retrieved waste by size reduction and then encapsulation of the waste with low-melting temperature polyethylene durability. A schematic of this process is shown in Figure 5. The size reduction and encapsulation processes are based on developmental research sponsored by the DOE Environmental Management Technology Development.¹⁴



GC99 0687

Figure 2. Overall grouting process with tanker truck access for the delivery of the molten paraffin.

Retrieval

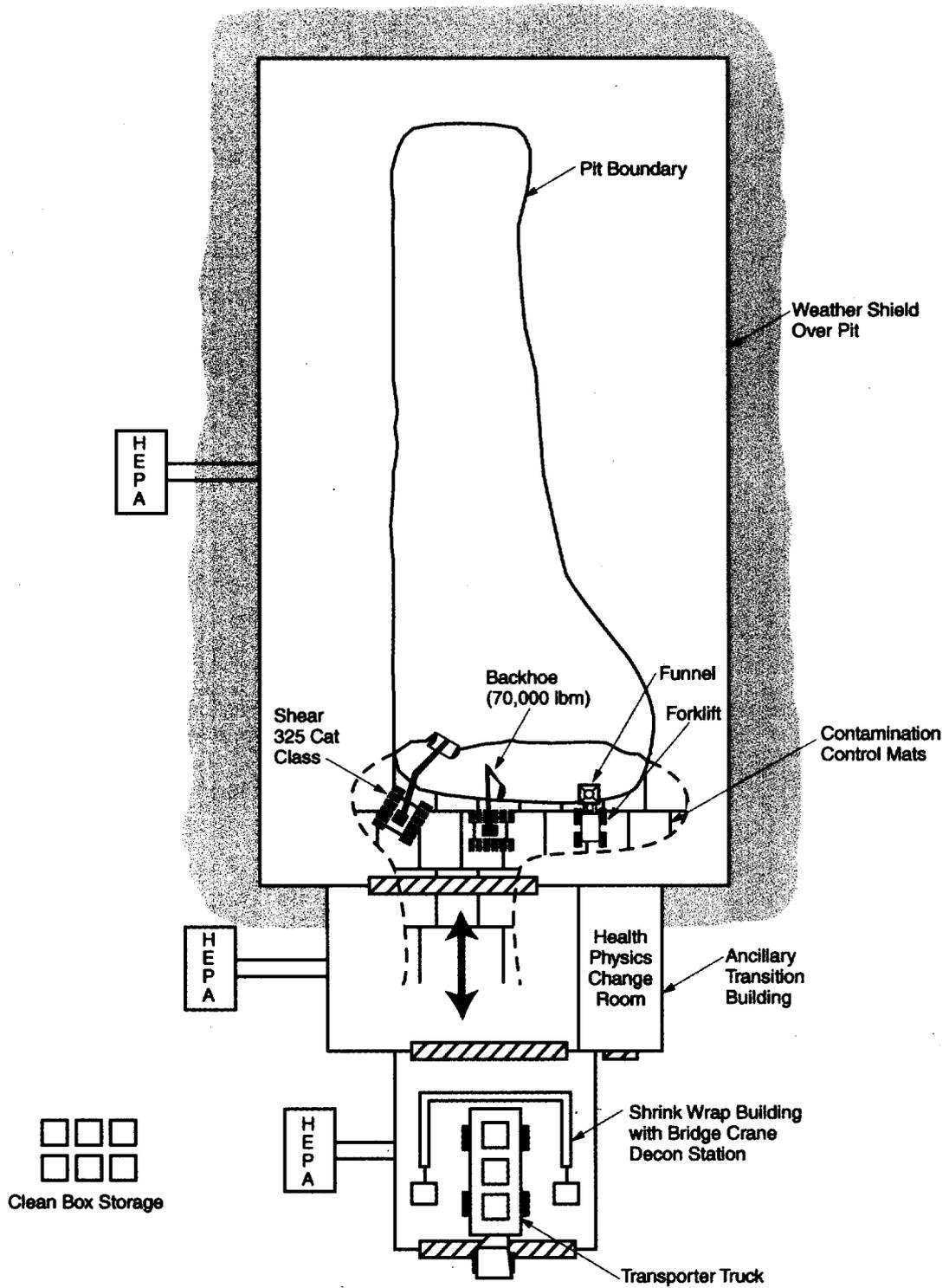
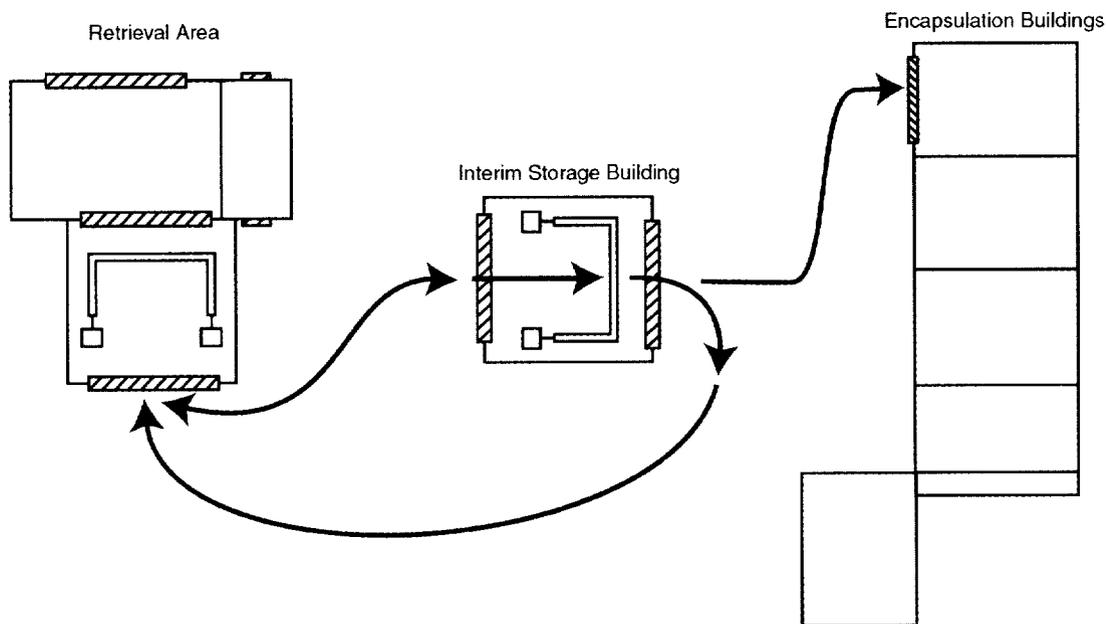


Figure 3. Retrieval will be performed from the bottom via an access ramp.



GC99 0689

Figure 4. Accumulation area will be provided for a lag between retrieval and encapsulation phases.

The size reduction will involve the CRYOFRACTURE system, which is essentially fire and explosion proof. The CRYOFRACTURE system involves freezing the retrieved waste to liquid nitrogen temperatures, then fracturing the brittle waste with a 1,000-ton press, which has a special attachment for cutting stainless steel items. This loose, frozen, sized material (90% will be 3 in. or less) will then be sent to a heater room by a V-belt conveyor. Once at room temperature, the waste will be sent to a mixing room and blended with low-melt temperature (180°F) polyethylene. Once the CRYOFRACTURED waste is blended with the polyethylene material, it is extruded into final waste forms using 4 × 4 × 8-ft molded polyethylene boxes with lids. These boxes have built-in lifting lugs for ease in transport to the disposal facility. The waste form will be designed such that the waste is both micro- and macro-encapsulated. Polyethylene encapsulation technologies have shown that the waste form is extremely durable for possible geologic times.

All of the technologies used in this process (paraffin grout, CRYOFRACTURE for size reduction, and encapsulation in polymers) have inherent contamination control features. Combining the technologies guarantees essentially no spread of contamination throughout the process.

Retrievable Disposal or Interim Storage

Retrievable disposal means that the waste forms while permanently disposed of can easily be removed for transportation to another treatment facility or offsite disposal facility at a later date. Figure 6 shows the final disposal option, in which the retrieved pit is sealed under the weather shield and surrounded with approximately 6-ft-thick concrete bottom and side walls (fully plasticized—low heat of hydration aggregate concrete). Once the concrete walls and bottom are constructed, the weather shield and ancillary buildings are internally sprayed with strippable paint, dismantled, and set up on a different pit to repeat the process.

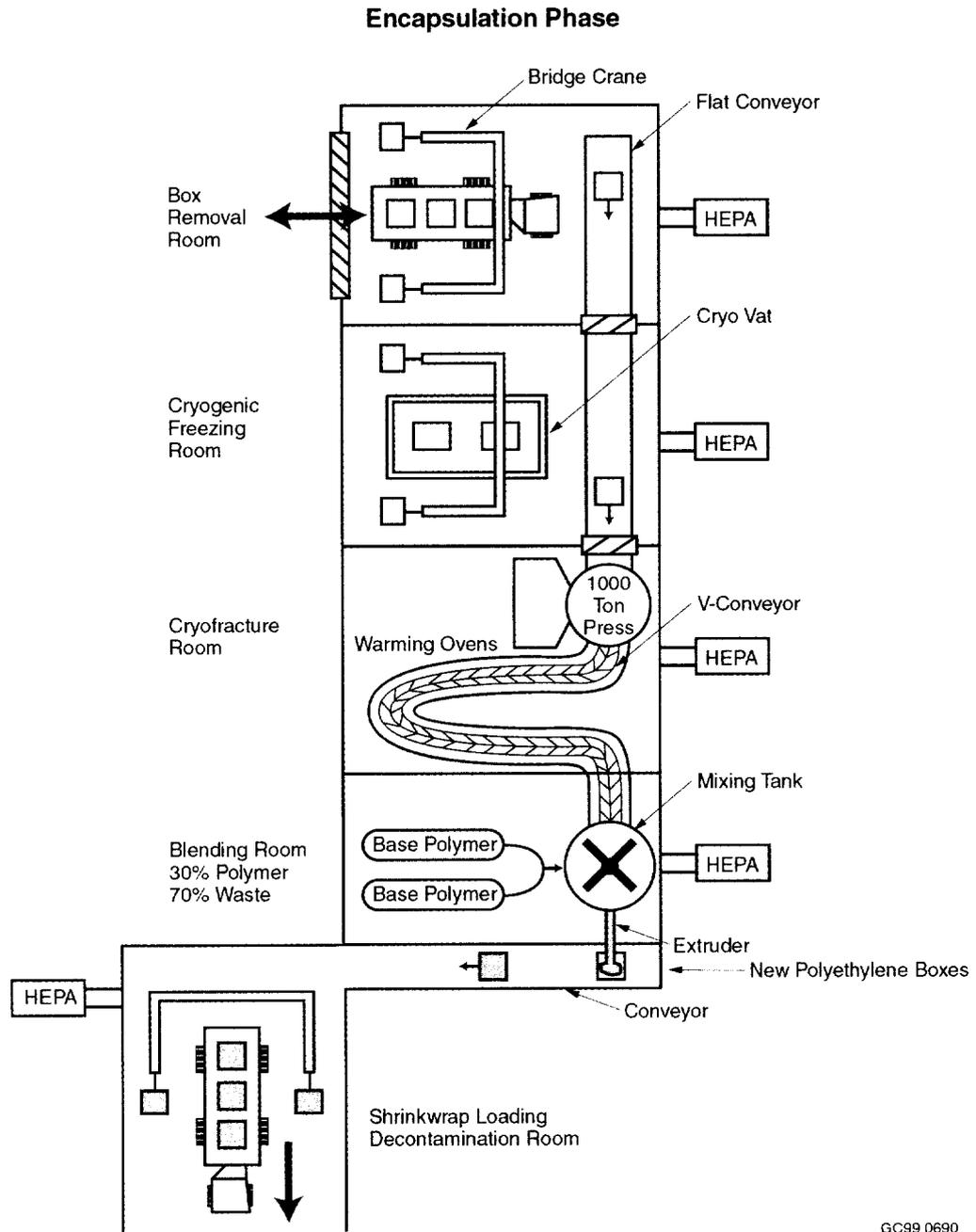
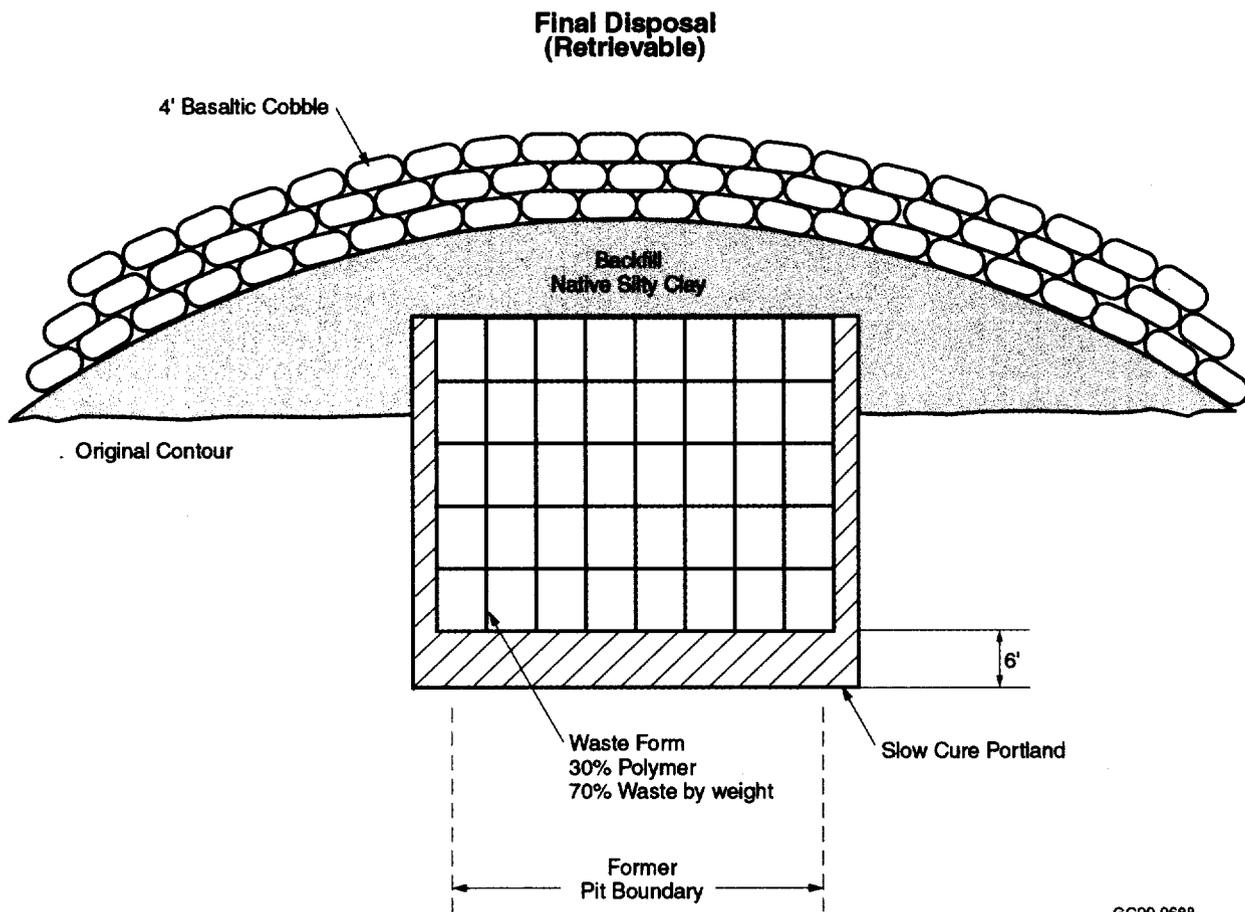


Figure 5. Encapsulation of the waste with low-melting temperature polyethylene.

The encapsulated waste is then stacked as shown in Figure 6 and covered with native soil and basaltic cobble for armoring. The top cover can be easily removed in the future if this technique does not meet performance specifications or other offsite options become expedient. In addition, any capping that is performed will be compatible with a full SDA final cap.

As an alternative, the encapsulated waste could be placed in single RCRA-approved storage buildings.



GC99 0688

Figure 6. Retrieval final disposal option.

Cost Estimate

This section includes (1) a cost estimate for a treatability study performed under CERCLA for demonstration of retrieval to “retrievable disposal” for a one-acre sized transuranic pit, and (2) a cost estimate for complete remediation retrieval to “retrievable disposal” or interim storage for nine acres of buried transuranic waste.

Cost Estimate for a 1-Acre Region of Retrieval

The cost estimate is given for each of the major features: site preparation, grouting, retrieval, accumulation area, encapsulation, and either retrievable disposal or interim storage in RCRA-quality buildings for a the one-acre sized transuranic pit at the INEEL SDA. Also estimated are the total programmatic costs associated with managing the process. Not included in this cost estimate is the cost of producing an interim record of decision (ROD) for this national priorities list site. This permitting process could in all likelihood take longer than the 7 years estimated for the retrieval to “retrievable disposal” activities.

Site Preparation

Site preparation costs are summarized in Table 1.

Table 1. Site preparation cost summary.

	Process	Rate	\$K
Labor and Equipment	Overburden leveling and stockpiling (approximately two ft of soil from one acre)	6 people × 160 hr × \$60/hr	57
	Equipment rental	3 pieces × 30 days × \$500/day	45
	Assay	1,000 samples at \$100/sample	100
	Total equipment labor costs		\$202
Capital Costs	1 Sprung building covering the pit		1,000
	2 Ancillary buildings Entrance and exit control		2,000
	Power 1 MW		250
	Fire suppression Remotely operated system		1,000
	Sample laboratory Rapid transuranic monitoring laboratory		500
	Subtotal capital		4,750
	Procurement labor adder		1,900
	Total capital costs		\$6,650
Total ROM Estimate for Site Preparation			\$6,852

Grouting

Grouting costs are summarized in Table 2.

Table 2. Grouting cost summary.

	Process	Rate	\$K
Labor Costs	Training and consulting	4 people × 1,000 hr × \$100/hr	400
	Grouting	4 people × 3,600 hr × \$60/hr	864
	RADCON support	2 people × 3,600 hr × \$60/hr	432
	Building maintenance support	1 person × 3,600 hr × \$60	216
	Waste management	160 hr × \$60/hr	9
	Waste disposal	200 ft ³ × \$500/ft ³	100
	Laboratory samples	30 samples/day × 400 days × \$100/sample	1,200
	Total Labor Costs		\$3,221
Capital Costs	1 grouting unit (pumps, hoses, metering devices, drill rig, drill steel)		1,000
	1 X/Y support system		250
	Grout (14,000 holes × 100 gal/hole × \$4.43/gal)		6,202
	Subtotal capital		7,452
	Procurement labor adder		2,980
	Total capital costs		\$10,432
Total ROM Estimate for Grouting			\$13,653

Retrieval

Retrieval costs are summarized in Table 3.

Table 3. Retrieval costs summary.

	Process	Rate	\$K
Labor Costs	10 equipment operators, 1 manager, 2 supervisors	13 people × 1,800 hours × \$80/hr	1,872
	RADCON support	2 people × \$80/hr × 1,800 hours	288
	RTML support	30 samples/day × 200 days × \$100/sample	600
	ESH&Q support	1 × \$80/hr × 1,800 hours	144
	Total Labor Costs		\$2,904
Capital Costs	Heavy equipment with sealed cabs	\$500K excavator \$400K shear \$100K forklift	1,000
	Shrink-wrap equipment	Custom built and installed	500
	Mats	43,000 ft ² × \$20	871
	Semi tractor trailer transporter		250
	Polyethylene transport boxes	6,806 × \$50	340
	Gantry crane in shrink-wrap room		250
	Subtotal capital		3,211
	Procurement labor adder		1,477
Total capital costs		4,688	
Total ROM Estimate for Retrieval			\$7,592

Accumulation Area

Accumulation Building costs are summarized in Table 4.

Table 4. Accumulation Building storage costs summary.

	Process	Rate	\$K
Capital Costs	Basic storage building as used to support TSA		3,000
	Procurement adder		1,200
	Road Improvements	Paving \$100/ft × 1,000 ft	100
Total ROM Estimate for Accumulation Building			\$4,300

Encapsulation

Encapsulation costs are summarized in Table 5.

Table 5. Encapsulation costs summary.

	Process	Rate	\$K
Labor and Material Costs	10 operators; 2 supervisors; 1 manager	13 people × 1,800 hours × \$80/hr	1,872
	RADCON support	2 people × 1,800 hours × \$80/hr	288
	ESH&Q support	1 × 1,800 hours × \$80/hr	144
	Contamination control sampling	30 samples/day × 200 days × \$100/sample	600
	Polyethylene	Assume waste is 43,560 ft ² × 12 ft × 100 lb/ft ³ = 52,272,000 lb, and polyethylene at 0.30 mass loading = 15,681,600 at \$1/ lb _m	15,681
	Liquid nitrogen	Per year	20
Total Labor Costs			\$18,605
Capital Costs	Building	4 chambers; loading dock;	3,000
	HEPA		2,000
	Fire suppression		1,000
	3 bridge cranes		750
	Cryofracture system		3,500
	Cryofracture heating system		500
	Conveyor system with controls		500
	Mixing tank and polyethylene heating/storage tanks		3,000
	Shrink-wrap system		500
	Subtotal capital costs		14,750
Procurement adder		5,900	
Total Capital Costs			20,650
Total ROM Estimate for Encapsulation			\$39,255

Retrievable Disposal

Disposal costs are summarized in Table 6.

Table 6. Retrievable Disposal cost estimate.

	Process	Rate	\$K
Labor and Material Costs	Concrete labor	Estimated	1,018
	Tent removal and burial	10 operators × 160 hr × \$80/hr	128
	Total Labor Costs		\$1,146
Capital Costs	Backfill	50,000 ft ² × 3 ft × \$50/yd ³	277
	Backfill cobble	50000 ft ² × 4 ft × \$100/yd ³	740
	Concrete bottom	9,680 yd ³ × \$100/yd ³	968
	Concrete sides	1,000 ft × \$50/ft	50
	Subtotal capital costs		2,035
	Procurement adder		407
	Total Capital Costs		2,442
Total ROM Estimate for Retrievable Disposal			\$3,588

Interim Storage Option

The cost for the interim storage option is simply 10 storage buildings times the estimated cost of \$3 million per building plus \$1 million for labor to move the encapsulated material to the interim storage buildings. There is no cost estimate for the final “unknown” disposition of this material.

Programmatic Costs

For a subcontractor to the DOE, project management costs are assured to be 20% of the total cost estimate. Again, this does not include the cost of obtaining a ROD for this action.

Summary Total ROM Estimate for 1 Acre

Table 7 shows the cost estimate for 1 acre.

Table 7. One-acre cost estimate.

Option 1 – Retrieval Disposal	\$K
Site Preparation	6,852
Grouting	13,653
Retrieval	7,592
Accumulation	4,300
Encapsulation	39,255
Retrieval Disposal	3,588
Subtotal	75,240
Programmatic 1.2×	15,048
Total	\$90,288

Option 2 – Interim Storage	\$K
Site Preparation	6,852
Grouting	13,653
Retrieval	7,592
Accumulation	4,300
Encapsulation	39,255
Interim-Storage (10 buildings + labor)	31,000
Subtotal	102,652
Programmatic 0.2×	20,530
Total	\$123,182

Cost of Application to 9 Acres within the SDA

The cost estimates for application to a nine-acre site are summarized in this section. Some assumptions are necessary when performing these tasks on nine acres, such as:

- Three grouting systems would be employed to reduce the grouting time from 18 years to 6 years
- The grouting systems and associated buildings and equipment would be reusable
- The retrieval effort would involve three retrieval systems and three encapsulation systems
- Grouting the first three pits would require 2 years; grouting, retrieval, encapsulation, and disposal would require an additional 5 years for the remaining 6 pits for a total of 7 years for the project.

None-Acre Cost Estimates

Cost estimates are summarized in Table 8.

Table 8. Nine-acre cost estimate.

Option 1 – Retrievable Disposal		\$K
1.	Site Preparation	
	Labor	2,718
	Capital (3 systems @ 6,650 each)	19,950
2.	Grouting	
	Labor	25,789
	Capital (3 systems and grout)	95,911
3.	Retrieval	
	Labor	26,136
	Capital (3 systems)	14,064
4.	Accumulation	
	Labor	500
	Capital (3 buildings total)	9,000
5.	Encapsulation	
	Labor/Materials	167,445
	Capital	61,950
6.	Retrieval Disposal	
	Disposal of last three pits*	10,764
	Disposal of first six pits	20,760
	Subtotal	454,987
	Project Management × 20%	90,997
	Total	\$545,984
Option 2 – Interim Storage		\$K
1.	Site Preparation	
	Labor	2,718
	Capital (3 systems @ 6,650 each)	19,950
2.	Grouting	
	Labor	25,789
	Capital (3 systems + grout)	95,911
3.	Retrieval	
	Labor	26,136
	Capital (3 systems)	14,064
4.	Interim Storage**	
	Capital (building 90 x 3000)	270,000
	Labor	500
	Subtotal	455,068
	Project Management 20%	91,013
	Total	\$546,081

*Disposal of weather shield on last three pits.

**Encapsulation was included in the one-acre treatability study, but not for the transuranic pits and trenches Interim Storage Option. The reason for this is that the Interim Storage is considered a temporary (20-year) solution, supporting an undefined storage (90 buildings) process for which polyethylene encapsulation may be incompatible.

Discussion

By using multiple units for the various pits, the technical project could be accomplished in 7 years, with a budget of approximately \$545,000. In addition, final design, permitting, operational readiness reviews, safety evaluations, and procurements would require approximately 3 years and \$60,000. The entire project could be accomplished in 10 years from start with a budget of approximately \$605,000.

A cap could eventually be placed over the entire Subsurface Disposal Area. Therefore, the cap proposed in this design would support the final capping alternative in the Feasibility Study.

The estimated retrieval costs are lower than the cost estimates for grouting and encapsulation because the grouted waste can be retrieved with relatively simple equipment. Contamination control is inherently added as the paraffin grout or polyethylene material provide encapsulation and agglomeration of fine particulates. Criticality is a nonissue for all phases because of the 1g/L boron in the grout. The disposal costs in the retrievable disposal option are also relatively low.

The cost of grouting and retrieval for interim storage is about the same as the retrievable disposal option. However, the costs of final disposal associated with the interim storage option have not been included (final disposal costs could cause the estimate for the interim storage option to double while seeking a disposal site).

Conclusions and Recommendations

Costs associated with the 9-acre application are approximately the same as in situ vitrification and in situ thermal desorption technologies and are lower than remote retrieval efforts. When compared to the technical criteria supported by Idaho, the retrieval option (with waivers on certain environmental laws) should be considered in the evaluation. The main environmental law that requires evaluation is the concept of "retrievable disposal" within the pits the waste was originally interred. It is recommended that a formal cost-benefit comparison be performed between the Pit 9 WAG 7-10 process, the processes proposed in this document, and other innovative ideas, such as in situ vitrification followed by retrieval.

References

1. J. R. Bischoff and R. J. Hudson, Early Waste Retrieval Final Report, TREE-1321, August 1979.
2. Kirk B. McKinley and Joseph D. Card, Initial Drum Retrieval Final Report, TREE 1286, August 1978.
3. John McQuary, A Summary of the Environmental Restoration Program Retrieval Demonstration Project at the Idaho National Engineering Laboratory, EGG-WTD-9291, Revision 1, February 1991.
4. D. J. Valentich, Full-Scale Retrieval of Simulated Buried Transuranic Waste, EGG-WTD-10895, September 1993.
5. D. J. Valentich and E. L. Yokuda, Final Report for the Cryogenic Retrieval Demonstration, EGG-WTD-10397, September 1992.
6. Idaho National Engineering Laboratory, Buried Waste Robotics Program, Remote Excavation System Technology Evaluation Report, EGG-2710, Sept 1993.
7. M. R. Winberg, et al., Fugitive Dust Control Experiments Using Directed Air Flow in Dumping Operations, EGG-WTD-10360, July 1992.
8. M. R. Winberg and V. E. Wixom, Fugitive Dust Control Experiments Using Soil Fixatives on Vehicle Traffic Surfaces, EGG-WTD-10354, August 1992.
9. D. N. Thompson et al., Evaluation of the Contamination Control Unit During Simulated Transuranic Waste Retrieval, EGG-WTD-10973, November, 1993.
10. G. G. Loomis and D. N. Thompson, Innovative Grout/Retrieval Demonstration Final Report, INEL-94/0001, January 1994.
11. G. G. Loomis, Innovative Subsurface Stabilization of Transuranic Pits and Trenches, INEL-95/0632, December 1995.
12. G. G. Loomis, A. P. Zdinak, and C. W. Bishop, Innovative Subsurface Stabilization Project Final Report, INEL-96/0439, November 1996.
13. DOE/ID-10690, Operable Unit 7-13/14, In Situ Grouting Treatability Study Work Plan, U. S. Department of Energy Idaho Operations Office, September 1999.
14. G. G. Loomis, D. Osborne, and M. J. Ancho, Executive Summary of the Cryofracture Demonstration Program, EGG-WTD-9916, September 1991.

Appendix J
Grouting Vendor Bid

Appendix J

Grouting Vendor Bid

The following vendor bid was prepared by Applied Geotechnical Engineering and Construction of Richland, Washington. This company was the grouting contractor on the in situ grouting treatability study at the INEEL as well as on past INEEL grouting projects. This bid was prepared as if the vendor was submitting a bid to do the work. There are two parts in the bid: one for the x-y positional system (discussed in Appendix I) and the other is using the thrust block concept that was reported in this report.

Part-1—Thrust Block or Cover Block Approach

A summary bid is provided as follows with the intent to provide guidance and foundation in comparison of technologies for waste management alternatives relative to solid waste interned at the Idaho National Engineering and Environmental Laboratory

GENERAL REQUIREMENTS

1100 Summary

1107 Professional Consultant

Fees for architectural services, construction management, engineering, and surveying are not included under this item.

Consultant fees of \$500,000.00 for professional consultation for engineering/Geotechnical services/grout formulation/etc. over the 10-year duration of the project are included under this item.

1200 Price and Payment Procedures

1290 Payment Procedures

State and local taxes are assumed not applicable and are not included under this cost item.

1300 Administrative Requirements

1310 Project Management/Coordination

Permits will be the responsibility of others throughout the duration of this project.

Bonding including all performance, payment, and other surety bonds and related bonding for this project will be on the order of \$7,130,000.00 which is approximately 1.5 percent of the total project cost. Bonding is estimated at the above rate in part due to the actual or perceived radiological and hazardous conditions at the project site in addition to the construction nature of the project.

The project duration is assumed as approximately 10 years.

Insurance for all aspects of the project for builders risk, equipment, public liability, pollution coverage, and related item are estimated at \$ 1,900,181.00 from inception to completion of this remediation project. This estimate is nominally 0.4 percent of the project cost.

Main office expense, i.e., headquarters expense over the duration of the project are estimated at \$18,526,760.00 or 3.9 percent of the total project cost over the duration of the project.

The overhead and profit for the total project is estimated nominally at 30 (fixed and general) and 10 percent, respectively.

Field personnel for administration include: (1) General manager/Project Manager, (2) Project Superintendent, (3) Project Engineer/Assistant Superintendent, (4) Administrative Assistant, (5) Planner/Scheduler, (6) Safety and Industrial Hygienist, (7) Health Physics Supervisor, (8) Quality Control Engineer, and (9) Secretary/Clerk, and (10) craft personnel. The above will be required for daily operation and a Superintendent/Assistant Superintendent and Health Physics Supervisor will be required off Shift. The total costs for the above over the 10-year project duration is estimated at \$ 104,000,000.00.

1320 Construction Progress Documents

Construction project documents will be provided over the duration of the project by staff of the above item.

1400 Quality Requirements

1450 Quality Control

Quality control determination on materials over the 10 years of this project will be the responsibility of others. Quality control testing of equipment operations and durability however is estimated at \$195,000.00 total cost over the duration of the project. This includes annual inspections and certifications of cranes and crane components, man lifts, truck tractors, truck trailers, rigging, and equivalent critical items.

1500 Temporary Facilities and Controls

1510 Temporary Utilities

Utilities utilized to support temporary offices, materials storage areas, shops, and related areas are the responsibility of others including electrical, mechanical, sanitation, communications, and equivalent site utilities. Costs for utilities for these structures are not included in this item.

1520 Construction Facilities

One general administrative office temporary modular building will be required to support project activities. One general meeting, conference room, and office modular building will be required to support operations and offsite personnel project activities. Three change rooms/lunchrooms will also be required to house site operations forces. Four storage containers will additionally be required for storage of site materials and site forces tools and craft materials. Office trailers and change rooms/lunchrooms are estimated as a one-time cost of \$225,900.00. Storage containers are estimated to cost \$20,000.00 as a one-time cost over the life of the project. Facilities and controls used to support project containment buildings and drilling/grouting operations are estimated at \$245,900.00 over the 10-year duration of the project.

1530 Temporary Construction

All project facilities and all-weather conditions are considered as temporary conditions, hence this Item is considered not applicable.

1540 Construction Aids

Level d personal protective equipment for all site administration and labor over the duration of the project totals \$200,000.00. This includes safety equipment such as hard hats, safety glasses, earplugs, gloves, substantial foot ware, coveralls, and cold weather apparel.

Level c and level b personal protective equipment used in radiological and mixed radiological and hazardous waste areas will include airline bottle-cart and escape-pack systems and breathing air, HEPA filtered masks and cleaning service, radiological coveralls with attached boots and hoods, shoe covers with cleaning service, and surgical gloves and work gloves. The total cost for this item over the 10 years of the project is estimated at \$650,000.00.

1550 Vehicular Access and Parking

An area for staging of office trailers, change trailers, containers, shops, and parking facilities (approximately 2 acres) will be required during initial site activities. The cost for this activity is a one-time cost at \$50,000.00.

1560 Barriers and Enclosures

Barriers and enclosures are not included under this item as the site will be located on U.S. Government property and radiological facilities will be located within a highly controlled fenced area.

Security personnel are not included in this item as it is assumed that security is the responsibility of others.

1580 Project Signs

Project signs will be installed at the onset of site activities relative to occupational safety and health requirements and throughout the duration of the project as required by industrial safety and radiological conditions. The cost estimate for this item is \$10,000.00.

1590 Equipment

Jet grouting track drills will be required for injection of slurry into the subsurface waste materials. Data logging components will also be included to quantify drilling and grout slurry injection parameters. Each jet grouting track drill is expected to last under operating conditions for 5 years. Two track drill units are intended to be used operationally with one unit as a backup unit. This backup unit is necessary due to project schedule constraints. Unit costs are approximated at \$346,000.00 totaling \$2,075,000.00 over the duration of the project. Spare parts for these units include slurry swivels, drilling rod, seals, and bit assemblies. The annual costs for these spare parts are \$90,000.00 with a 10-year operational total of \$900,000.00.

Jet grouting slurry pumps will be required for pumping slurry from a batch plant to the jet grouting track drill under pressure required for injection of slurry into waste and waste matrix materials. Each jet grouting slurry pump is expected to last for 5 years. The jet grouting slurry pump and power pack is an

integral unit which is containerized. The unit cost for each jet grouting slurry pump is \$180,000.00 with a total cost of \$1,800,000.00 for 6 units over the 10-year life of the project. Of the 6 units two will serve as backup units required due to project schedule constraints. Each year a major over haul of the pump system will be required. Hence, a spare parts 2,000-hour over haul kit is required for each unit. Unit costs for each overhaul kit is \$50,000.00 with 24 kits required over the duration of the project that totals \$1,200,000.00.

Jet grouting mix plants will be required for storing preblended dry grout materials, metering dry grout materials and water, and mixing and shearing these materials to produce grout slurry. Additionally, the plant pumps slurry to the above jet grouting slurry pump utilizing a low head centrifugal pump. This mixing plant utilizes an exterior dry materials hopper and auger to feed materials to the mixers/blenders, and a containerized unit with tanks, vortex mixers, and controls. The unit costs for these jet grouting mix plants are \$75,000.00. It is assumed that two units will be used to serve each operational jet grouting slurry pump and one back unit will be kept in reserve with a total of 6 units. The reserve or backup units are required due to project schedule constraints. These units will be replaced each 5 years. The total costs for these units over the 10-year operational life of the project is \$450,000.00. Spare parts for these mix plant units are estimated at \$8,000 each with a requirement of 24 spare parts/rebuild kits over the duration of the project for total costs of \$192,000.00.

Electrical generators are required to power the above jet grouting mix plant and to provide auxiliary power to the mix plant and the jet grouting slurry pump operating in series with the plant. These generators have requirements of 75 kilowatts and are capable of voltage rating up to 480 volts AC. Six generators will be required at a unit cost of \$20,500.00 and a total cost over the duration of the project of \$123,000.00. This estimate assumes replacement of generators twice during the life of the project.

Electrical generators are also required to power HVAC systems supporting grout injection operations. These systems include ventilation and HEPA filter operations within primary and secondary radiological containment areas. The power and voltage requirements for these units are 200 kilowatts and 480 volt AC, respectively. These generator units as above will be replaced twice over the duration of the project. Hence, 6 units will be required. The unit cost for each generator is \$38,000.00 with a total project cost of \$228,000.00.

A hydraulic crane with the capacity of nominally 40 tones will be required to support mobilization, operations, maintenance, transportation, and demobilization project activities. This crane is estimated to cost \$250,000.00.

An end loader capable of multiple activities including initial plant grading, loading/offloading, materials handling, excavation, road/surface maintenance, and structure assembly/disassembly. The end loader will include fork and bucket assemblies. Unit cost for this equipment item is \$320,000.00.

A large capacity water haul vehicle will be required to transport water from a site supplied water source to each bulk mixing plant. This unit will include pump capacity of offload bulk water. The one-time unit cost of this unit is \$600,000.00.

All terrain man lifts are required for support and fabrication of operations. Two units are required for assembly of building structural components as well as disassembly. Additionally, these units will be required to service operations within building structures as a precursor to, during, and subsequent to grout slurry injection. The unit cost for these units is \$120,000.00 with a total cost over the duration of the project of \$240,000.00.

Off road heavy duty semi truck tractors will be required to transport dry bulk materials from bulk handling/railhead facilities. The unit cost for each tractor is estimated at \$91,500.00 wherein four units are required. The total project cost for this item is estimated over the duration of the project at \$366,000.00.

Large heavy duty dry materials transport trailers capable of transporting nominally 30 tonnes of dry product from rail head facilities to each grout dry mix facility are required. These trailers include mechanical/pneumatic systems for on loading and offloading of bulk powder. The unit cost of these trailers is \$105,000.00 with three required over the duration of the project totaling \$315,000.00.

A heavy-duty rock bulk haul trailer is required to support grout slurry cleanout operations. This trailer will haul bulk solidified grout materials from a cleanout area to an onsite stockpile area. The unit cost for this trailer delineated as a one-project time cost is \$50,500.00.

A forklift of all terrain operations capacity is required for support of all project activities. This unit is also required to have high reach capacity in order to support building erection and disassembly. The one-time unit cost for this equipment is estimated at approximately \$100,000.00.

Fuel required for all equipment/vehicles/power units/etc. will be supplied to the project from a local vendor and transported in a bulk fuel truck. The capacity of the fuel load of the truck is required at nominally 2,000 gal. The onetime unit cost for this bulk fuel truck is estimated at \$100K.

A maintenance vehicle will be required to provide support to all jet grouting operations and ancillary project activities. This vehicle will include a large heavy duty off road truck with welder, oxy/acetylene cutting/welding, small capacity lift, tools, lubricants, air compressor, and related maintenance equipment. This one-time procurement unit cost is estimated at approximately \$66.5K.

One off road utility truck with a nominal capacity of 2 tones is required for project support. The unit cost for this truck is \$45K.

Utility trucks with off road capability are required to support this project from onset to completion. Four pickup trucks will be required over the duration of the project with a unit cost of \$35K and a total project cost of \$140K.

Portable light plants will be required to support interior lighting within grout injection confinement structures. Two units will be used per structure. These light plants will be replaced twice during the life of the project. The unit cost is \$7K resulting in a project cost over 10 years of \$42K.

Fuel required for operation of the above is estimated at \$1,522,000 over the 10-year life of the project. This was estimated at 6 gal per hour for electrical generating equipment and 15 gal per hour for rolling stock, i.e., trucks, end loader, crane.

1700 Execution Requirements

1740 Cleaning

Site cleanup after construction and ongoing operations cleaning costs are included in general operations and not itemized here as a cost item.

1800 Facility Operations

1810 Commissioning

Training will be required for all site administrative and operations personnel. Required initial training for site, radiological, and hazardous worker, etc., training categories is estimated at \$2,355.00 per person per year. Re-certification for staff after initial training is estimated at \$840.00 per year. The total project cost for training is estimated at \$ 753,540.00 over the complete 10-year duration of the project.

SITE CONSTRUCTION

IC Site Materials and Methods

2060 Aggregate

Aggregate will be required for site fill in and around offices, change rooms, containers, containment structures, and haul roads. Aggregate will also be used within containment structures during grout slurry injection preparation and operations. The cost for this material is \$310,000.00.

2065 Concrete

Concrete will be required to support containment structures as structural footings. Concrete footings will also be used as a berm for containment of grout spoils. The cost for each footing item relative to each containment structure is nominally \$870.00. Ten footing units will be required for an overall project cost of \$87,000.00.

Concrete or grout is required over the area where grout injection has been completed. This material is used as a primary barrier covering the grout stabilized monolith. The depth of this barrier is 17 inches covering an area of 140 X 400 feet. Each grout barrier is estimated to cost \$354,000.00. Hence, the total cost of 10 barriers is \$3,540,000.00. This concrete grout material is assumed as a 2,500 pounds per cubic foot unconfined compressive strength, synthetic fiber reinforced, and self-leveling material. The unit cost for this material is \$120.00 per cubic yard with 10 barriers required over the project duration. Hence, the total cost of primary barrier placement is \$3,430,000.00.

Grout slurry will be produced on site by transport of dry grout materials from a railhead bulk storage plant to each grout mix plants, addition of water, and pumping through grout pumps and injection systems. On the order of 20,000 grout slurry holes are assumed per operational injection site. There are 10 operational injection sites. Slurry volume per hole is noted at 100 gal. The unit cost for grout slurry is \$2.55 per gal. 24,000,000 gal of grout slurry is required for completion of monolith injection over the total project waste volume over the 10-year project volume. The total cost of grout slurry is estimated at \$61,200,000.00. This assumes approximately 20 percent waste for spoils and cleanout.

5050 Metals

Two large containment structures will be used to confine grout slurry injection activities. Two each smaller support structures will be used to contain grout slurry mixing and shearing operations and grout high pressure pumping. One large containment structure will initially be fabricated along with smaller support structures. While these structures are under operations, another large containment structure and adjoining smaller support structures will be fabricated at another proximal location. Hence, one complete operational jet grouting system will be fabricated while the other is under operations, i.e., each system

will be fabricated initially and moved to another location for a total of 11 locations. This is required to meet a 10-year production schedule.

Each large confinement structure is conceptually designed at an operational height of 35 feet in order to accommodate jet grouting track drill mast height and drill rod stroke. The plan dimensions are assumed at 140 X 400 feet. Fabrication and dismantlement of each structure is assumed at 80 days each.

The cost for two of these large metal structure buildings, and spare/replacement parts assuming a one-time procurement is \$2,620,000.00.

Each small support structure is conceptually designed at 20 X 24 X 50 feet with respect to height, width, and length, respectively. Four each of these structures (2 each) will be used to support jet grout mixing/batching and high pressure pumping for each large containment structure. As per the above large containment buildings each of the small jet grouting support buildings will be moved for a total of 11 locations. One of the above small structures will be utilized as a project maintenance and shop facility. The project maintenance and shop facility will not be moved from its original location.

The cost for 5 support structures is estimated at \$225,000.00 assuming a one-time procurement over the life of the project.

Large cover blocks are required to provide containment of contaminants during jet grouting operations. These structures consist of an upper surface of steel plates with internal steel bracing. Further, these structures are bounded on each side by structural steel with ports used to permit attachment of HEPA filtration systems. The top also has removable ports for insertion of jet grouting drill steel. Ports are especially designed with primary and secondary plastic cylindrical boots used to isolate the drill steel from the personnel work area. The cover block assemblies are of sufficient strength to permit operation of heavy equipment on their surface. The cover blocks are leveled above each drilling location and brought to grade with aggregate.

Cover blocks sufficient to cover a plan area of 11 acres will be required. The total cost of these assemblies is estimated at \$283,800,000.00.

15050 Mechanical

Two large structures will be used to confine jet grouting injection activities. To support work in these structures for year-round operations, heating, air conditioning, and ventilation (HVAC) will be required. Heating will be provided with high capacity propane or equivalent gas. Propane or equivalent gas will be supplied onsite by a 15,000-gal storage tank or two 7,500-gal tanks. Propane storage will require a fenced, lined, and barricaded area. Air conditioning will be supplied with evaporative cooling. Air circulation within each large containment building will be filtered continually through high capacity high efficiency particulate air filters. HEPA filters will be tested by others.

Two HVAC complete systems are estimated at a one-time cost of \$2,500,000.00. This cost assumes utilization of two HVAC complete systems for 5 years, replacement and operation of the systems for the remaining 5 years. Also included are all spare parts and related materials. The cost of propane over the 10-year life of the project is estimated at \$1,000,000.00

16050 Electrical

Two camera systems are required for evaluation of operations of jet grouting drilling, grout slurry placement, spoils returns, and general equipment operations. Two camera/recording/viewing systems will

be positioned to cover each jet grouting track drill injection module. This configuration assumes 2 of the above systems per containment structure, or four systems overall.

The one-time cost of these systems including recording media for all grout slurry injection holes is estimated at \$280,000.00.

R130 Special Construction

It is assumed that a railhead located at the site will be used for delivery and offloading of dry bulk materials from hopper rail cars. Due to the volume of dry bulk materials required per unit time over the duration of the project, 2 large dry bulk storage silos located at the railhead, and 3 smaller silos located proximal to each jet grouting mixing/batching operation are required for efficient supply of grout former materials to each injection operation. Two large silos and mechanical/pneumatic of offloading equipment with a storage capacity of nominally 550 tones each will be constructed at the railhead. Dry bulk materials will be offloaded from these silos as needed and transported to one of 3 each smaller silos servicing each jet grouting operation.

The total cost for these 5 items as a one-time project cost is estimated at \$691,000.00.

Three water portable heated water tanks will be required to support each jet grouting mixing/batching operation, i.e., two operational and one backup tank as above.

The total cost for these tanks over the live of the project is anticipated at approximately \$36,000.00.

Specially designed drill shrouds are required to isolate contaminants from drilling operations entering into personnel spaces within the large containment structure covering each drilling location. Numerous isolation shrouds consisting of coaxial flexible bellows oriented axially over the drill rods and attached to the jet grouting track drill mast assembly will be needed. The operational life of each shroud is estimated at 300 holes. Hence, assuming 30 holes can be drilled with each shroud assembly per each shift then 80 shrouds will be required. With a unit cost of \$15,000.00 the total estimated cost of shrouds over the life of the project is nominally \$1,200,000.00.

Crews

Summary

Administrative staff will be required at conception and throughout the duration of the project. This administrative function is assumed totally dedicated to the project and works directly for the project including interfacing with site personnel and programmatic staff from the site. A building construction crew working under the direction of the administrative staff will be required to build containment building structures, dismantle containment structures, and reassemble these structures over the duration of the project. The building construction crew will also erect bulk storage and materials handling facilities and equipment as well as dismantlement and decommissioning of the site on project completion. Operations crews will complete operations of equipment and support equipment directed to placement of subsurface grout materials.

Administrative staff will be required during one shift with one superintendent working off shift. The building construction crew will only work one shift. Two operations crews will be required, i.e., one crew per two shifts. Shifts are assumed as 10 hours in duration over a 5-day workweek.

Administration

A project general manager will be required with the responsibility of overall project administration, operations, and control. A site superintendent for each of two shifts will be required for supervision of all site operations. One project engineer will be required to support site engineering and to serve as an assistant superintendent. Two administrative assistants will be required to perform clerical and related functions in support of all administrative staff. One bookkeeper/timekeeper will be required to perform accounting and payroll activities. One planner/scheduler will be needed to assist in development and projection of task activities and to determine staffing requirements. An industrial hygienist will be required to develop and control safety and health programs, one per shift. Two health physics supervisors will be required to control and manage radiological programs, one per shift. A quality control administrator will also be required to develop and manage quality programs and to perform inspections. The total administrative staff consists of 13 persons.

Building Construction

Building construction activities will require a dedicated crew consisting of one working general foreman, one crane and heavy equipment operator, 6 ironworkers, and one laborer. The total manpower requirement for this activity is 9. If required, additional staff may be reassigned temporarily from an operations crew.

Operations

Grout placement operations will require a substantial crew. There will be two crews per working day. Each crew consists of one working general foreman to oversee all applicable dry bulk storage and transfer, water transfer, slurry mixing/pumping/and injection, waste haulage, etc., as well as operations of all system components. This functions will in turn be administered by a shift superintendent. Two mechanics will be required to perform preventative as well as general maintenance on all equipment and materials handling components. One electrician and 1 instrument technician will be required to perform component installation, operations, and disassembly related to electrical systems. These staff will also be responsible for electrical generating systems. Four track drill operators, simultaneously operating two injection systems per shift will be needed. Jet grouting pump operations, 2 each supporting each track drill injection system, feeding grout slurry to grout injection track drill components will require 2 operators. In turn, 2 bulk mixing plants supplying mixed and sheared slurry to each jet grouting pump will require 1 operator each. Two relief operators are required to cover each jet grouting drill injection, jet grouting pump, and jet grouting batching/mixing operations as a contingency. One heavy equipment operator will be required to support operations utilizing front-end loader, all terrain fork lift, hydraulic crane, etc.. Six teamsters are required to support grout injection and related project operations. These activities include fuel transport and fueling, water transport, bulk dry materials loading/off loading and transport, parts and materials pick up and delivery, and solidified waste grout transport and landfill operations offloading. Three laborers will be required for general site and operations support including control of radiological personal protective equipment. Two radiological technicians will be required to support each grout injection track drill activity. The total operations staff for two shifts consists of 54 craftsmen. Cross craft activities are also assumed where specific training requirements are not limiting. Where required lead individuals per craft may be assigned for supervisory actions under each shift general foreman and shift superintendent.

ASSUMPTIONS

Numerous assumptions have been delineated for this project limiting the estimate of project cost and cost projections. Assumptions are listed as follows:

- Waste stabilization will be performed under a reusable structure.
- Batching/mixing of grout slurry will be completed on site (at the location of injection).
- Waste stabilization will be completed with an injection density of 2,000 injection holes per acre.
- There are 10 locations of 1 acre each with generalized plan dimensions of 140 X 400 feet.
- Nominally each grout injection hole will be 10 feet in depth through the waste depth interval and 100 gal of grout will be used per injection.
- The project duration is on the order of 10 years.
- All equipment and materials delivered to the site are new and not previously used in production.
- Weekly work duration is 10 hours per day, 5 days per week. Operations will work two shifts of 10 hours each per day for 5 days each week. Administration will work one shift of 10 hours 5 days per week.
- Weather protection will be required for equipment and personnel within confinement and support structures.
- No release of radiological or hazardous contamination will occur as a function of jet grouting operations or support activities.
- Only minimal travel and per diem will be allowed and for only administrative staff.
- Grout placement includes a 17-inch thick grout or equivalent material cap over each drilling location and no further capping is required.
- Costs are provided in government Fiscal Year 2002 and are escalated at 3% per year over the project duration of 10 years where applicable.
- All project work is covered under Price – Anderson and related nuclear and hazardous materials liability and general requirements acts.
- Intellectual property, licensing, and related fees are the responsibility of others and are not included herein.
- All state and government taxes relative to project operations excluding personnel taxes etc., are the responsibility of others if applicable.
- Site security and patrol and site utilities, i.e., overhead electrical, potable and nonpotable process water, telephone, sanitary sewer or equivalent, and noncontaminated solid waste collection and disposal are provided by others.
- HEPA filter testing, and delivery to the project site are the responsibility of others.
- Site engineering surveys and waste location surveys if required are the responsibility of others.

- Costs associated with U.S. Department of Energy and/or Maintenance and Operations or Construction management and oversight are not included in this estimate.
- Administration, engineering, health physics, quality, training, safety, operations and associated records will be transmitted to applicable contractor offices and will be the responsibility of others.
- General site operations facilities and services will be available for project staff at nominal cost including but not limited to, general site training, specific craft training, radiological and hazardous materials training, fire/patrol support and control, and physical examinations.
- At the completion of project operations, jet grouting injection and support structures will be dismantled and staged, administrative and maintenance structures will be cleaned, isolated, winterized, and equipment will be winterized and staged onsite. These facilities/equipment/materials/etc. will be the property of the government.
- All equipment and materials are free on board INEEL.
- Dosimetry/dosimetry records, medical, testing and certification/medical records and associated programs are the responsibility of others.
- Safety analysis reports/reviews will be completed by others prior to contract award.
- Administrative, industrial safety and health, radiological control, quality, operations, etc., programs and plans be sued by the maintenance and operations site contractor will be adopted and approved prior to contract award.
- Engineering design of building structures and support structures/cover block/shroud will be the responsibility of others.

Part 2—X-Y Positional System Gantry Crane Approach

A summary cost estimate is provided as follows with the intent to provide guidance and foundation in comparison of technologies for waste management alternatives relative to solid waste interned at the Idaho National Engineering and Environmental Laboratory

GENERAL REQUIREMENTS

1100 Summary

1107 Professional Consultant

Fees for architectural services, construction management, engineering, and surveying are not Included under this item.

Consultant fees of \$500,000.00 for professional consultation for engineering/Geotechnical services/grout formulation/etc. over the 10-year duration of the project are included under this item.

1200 Price and Payment Procedures

1290 Payment Procedures

State and local taxes are assumed not applicable and are not included under this cost item.

1300 Administrative Requirements

1310 Project Management/Coordination

Permits will be the responsibility of others throughout the duration of this project.

Bonding including all performance, payment, and other surety bonds and related bonding for this project will be on the order of \$3,000,000.00 which is approximately 1.5 percent of the total project cost. Bonding is estimated at the above rate in part due to the actual or perceived radiological and hazardous conditions at the project site in addition to the construction nature of the project.

The project duration is assumed as approximately 10 years.

Insurance for all aspects of the project for builders risk, equipment, public liability, pollution coverage, and related item are estimated at \$791,841.00 from inception to completion of this remediation project. This estimate is nominally 0.4 percent of the project cost.

Main office expense, i.e., headquarters expense over the duration of the project are estimated at \$7,720,445.00 or 3.9 percent of the total project cost over the duration of the project.

The overhead and profit for the total project is estimated nominally at 30 (fixed and general) and 10 percent, respectively.

Field personnel for administration include: (1) General manager/Project Manager, (2) Project Superintendent, (3) Project Engineer/Assistant Superintendent, (4) Administrative Assistant, (5) Planner/Scheduler, (6) Safety and Industrial Hygienist, (7) Health Physics Supervisor, (8) Quality Control Engineer, and (9) Secretary/Clerk, and (10) craft personnel. The above will be required for daily operation and a Superintendent/Assistant Superintendent and Health Physics Supervisor will be required off Shift. The total costs for the above over the 10-year project duration is estimated at \$104,000,000.00.

1320 Construction Progress Documents

Construction project documents will be provided over the duration of the project by staff of the above item.

1400 Quality Requirements

1450 Quality Control

Quality control determination on materials over the 10 years of this project will be the responsibility of others. Quality control testing of equipment operations and durability however is estimated at \$195,000.00 total cost over the duration of the project. This includes annual inspections and certifications of cranes and crane components, man lifts, truck tractors, truck trailers, rigging, and equivalent critical items.

1500 Temporary Facilities and Controls

1510 Temporary Utilities

Utilities utilized to support temporary offices, materials storage areas, shops, and related areas are the responsibility of others including electrical, mechanical, sanitation, communications, and equivalent site utilities. Costs for utilities for these structures are not included in this item.

1520 Construction Facilities

One general administrative office temporary modular building will be required to support project activities. One general meeting, conference room, and office modular building will be required to support operations and offsite personnel project activities. Three change rooms/lunchrooms will also be required to house site operations forces. Four storage containers will additionally be required for storage of site materials and site forces tools and craft materials. Office trailers and change rooms/lunchrooms are estimated as a one-time cost of \$225,900.00. Storage containers are estimated to cost \$20,000.00 as a one-time cost over the life of the project. Facilities and controls used to support project containment buildings and drilling/grouting operations are estimated at \$245,900.00 over the 10-year duration of the project.

1530 Temporary Construction

All project facilities and all-weather conditions are considered as temporary conditions, hence this Item is considered not applicable.

1540 Construction Aids

Level d personal protective equipment for all site administration and labor over the duration of the project totals \$200,000.00. This includes safety equipment such as hard hats, safety glasses, earplugs, gloves, substantial foot ware, coveralls, and cold weather apparel.

Level c and level b personal protective equipment used in radiological and mixed radiological and hazardous waste areas will include airline bottle-cart and escape-pack systems and breathing air, HEPA filtered masks and cleaning service, radiological coveralls with attached boots and hoods, shoe covers with cleaning service, and surgical gloves and work gloves. The total cost for this item over the 10 years of the project is estimated at \$650,000.00.

1550 Vehicular Access and Parking

An area for staging of office trailers, change trailers, containers, shops, and parking facilities (approximately 2 acres) will be required during initial site activities. The cost for this activity is a one-time cost at \$50,000.00.

Barriers and Enclosures

Barriers and enclosures are not included under this item as the site will be located on U.S. Government property and radiological facilities will be located within a highly controlled fenced area.

Security personnel are not included in this item as it is assumed that security is the responsibility of others.

1580 Project Signs

Project signs will be installed at the onset of site activities relative to occupational safety and health requirements and throughout the duration of the project as required by industrial safety and radiological conditions. The cost estimate for this item is \$10,000.00.

1590 Equipment

Jet grouting track drills will be required for injection of slurry into the subsurface waste materials. The track drill equipment will include radio control for remote operations. Data logging components will also be included to quantify drilling and grout slurry injection parameters. Each jet grouting track drill is expected to last under operating conditions for 5 years. Two track drill units are intended to be used operationally with one unit as a backup unit. This backup unit is necessary due to project schedule constraints. Unit costs are approximated at \$346,000.00 totaling \$2,075,000.00 over the duration of the project. Spare parts for these units include slurry swivels, drilling rod, seals, and bit assemblies. The annual costs for these spare parts are \$90,000.00 with a 10-year operational total of \$900,000.00.

Jet grouting slurry pumps will be required for pumping slurry from a batch plant to the jet grouting track drill under pressure required for injection of slurry into waste and waste matrix materials. Each jet grouting slurry pump is expected to last for 5 years. The jet grouting slurry pump and power pack is an integral unit which is containerized. The unit cost for each jet grouting slurry pump is \$180,000.00 with a total cost of \$1,800,000.00 for 6 units over the 10-year life of the project. Of the 6 units two will serve as backup units required due to project schedule constraints. Each year a major over haul of the pump system will be required. Hence, a spare parts 2,000-hour overhaul kit is required for each unit. Unit costs for each overhaul kit is \$50,000.00 with 24 kits required over the duration of the project that totals \$1,200,000.00.

Jet grouting mix plants will be required for storing preblended dry grout materials, metering dry grout materials and water, and mixing and shearing these materials to produce grout slurry. Additionally, the plant pumps slurry to the above jet grouting slurry pump utilizing a low head centrifugal pump. This mixing plant utilizes an exterior dry materials hopper and auger to feed materials to the mixers/blenders, and a containerized unit with tanks, vortex mixers, and controls. The unit costs for these jet grouting mix plants are \$75,000.00. It is assumed that two units will be used to serve each operational jet grouting slurry pump and one back unit will be kept in reserve with a total of 6 units. The reserve or backup units are required due to project schedule constraints. These units will be replaced each 5 years. The total costs for these units over the 10-year operational life of the project is \$450,000.00. Spare parts for these mix plant units are estimated at \$8,000 each with a requirement of 24 spare parts/rebuild kits over the duration of the project for total costs of \$192,000.00.

Electrical generators are required to power the above jet grouting mix plant and to provide auxiliary power to the mix plant and the jet grouting slurry pump operating in series with the plant. These generators have requirements of 75 kilowatts and are capable of voltage rating up to 480 volts AC. Six generators will be required at a unit cost of \$20,500.00 and a total cost over the duration of the project of \$123,000.00. This estimate assumes replacement of generators twice during the life of the project.

Electrical generators are also required to power HVAC systems supporting grout injection operations. These systems include ventilation and HEPA filter operations within primary and secondary radiological containment areas. The power and voltage requirements for these units are 200 kilowatts and 480 volt AC, respectively. These generator units as above will be replaced twice over the duration of the project. Hence, 6 units will be required. The unit cost for each generator is \$38,000.00 with a total project cost of \$228,000.00.

A hydraulic crane with the capacity of nominally 40 tones will be required to support mobilization, operations, maintenance, transportation, and demobilization project activities. This crane is estimated to cost \$250,000.00.

An end loader capable of multiple activities including initial plant grading, loading/offloading, materials handling, excavation, road/surface maintenance, and structure assembly/disassembly. The end loader will include fork and bucket assemblies. Unit cost for this equipment item is \$320,000.00.

A large capacity water haul vehicle will be required to transport water from a site supplied water source to each bulk mixing plant. This unit will include pump capacity of offload bulk water. The one-time unit cost of this unit is \$600,000.00.

All terrain man lifts are required for support and fabrication of operations. Two units are required for assembly of building structural components as well as disassembly. Additionally, these units will be required to service operations within building structures as a precursor to, during, and subsequent to grout slurry injection. The unit cost for these units is \$120,000.00 with a total cost over the duration of the project of \$240,000.00.

Off road heavy duty semi truck tractors will be required to transport dry bulk materials from bulk handling/railhead facilities. The unit cost for each tractor is estimated at \$91,500.00 wherein four units are required. The total project cost for this item is estimated over the duration of the project at \$366,000.00.

Large heavy duty dry materials transport trailers capable of transporting nominally 30 tonnes of dry product from rail head facilities to each grout dry mix facility are required. These trailers include mechanical/pneumatic systems for on loading and offloading of bulk powder. The unit cost of these trailers is \$105,000.00 with three required over the duration of the project totaling \$315,000.00.

A heavy-duty rock bulk haul trailer is required to support grout slurry cleanout operations. This trailer will haul bulk solidified grout materials from a cleanout area to an onsite stockpile area. The unit cost for this trailer delineated as a one-project time cost is \$50,500.00.

A forklift of all terrain operations capacity is required for support of all project activities. This unit is also required to have high reach capacity in order to support building erection and disassembly. The one-time unit cost for this equipment is estimated at approximately \$100,000.00.

Fuel required for all equipment/vehicles/power units/etc. will be supplied to the project from a local vendor and transported in a bulk fuel truck. The capacity of the fuel load of the truck is required at nominally 2000 gal. The onetime unit cost for this bulk fuel truck is estimated at \$100,000.00.

A maintenance vehicle will be required to provide support to all jet grouting operations and ancillary project activities. This vehicle will include a large heavy duty off road truck with welder, oxy/acetylene cutting/welding, small capacity lift, tools, lubricants, air compressor, and related maintenance equipment. This one-time procurement unit cost is estimated at approximately \$66,500.00.

One off road utility truck with a nominal capacity of 2 tones is required for project support. The unit cost for this truck is \$45,000.00

Utility trucks with off road capability are required to support this project from onset to completion. Four pickup trucks will be required over the duration of the project with a unit cost of \$35,000.00 and a total project cost of \$140,000.00.

Portable light plants will be required to support interior lighting within grout injection confinement structures. Two units will be used per structure. These light plants will be replaced twice during the life of the project. The unit cost is \$7,000.00 resulting in a project cost over 10 years of \$42,000.00.

Fuel required for operation of the above is estimated at \$1,522,000.00 over the 10-year life of the project. This was estimated at 6 gal per hour for electrical generating equipment and 15 gal per hour for rolling stock, i.e., trucks, end loader, crane.

1700 Execution Requirements

1740 Cleaning

Site cleanup after construction and ongoing operations cleaning costs are included in general operations and not itemized here as a cost item.

1800 Facility Operations

1810 Commissioning

Training will be required for all site administrative and operations personnel. Required initial training for site, radiological, and hazardous worker, etc., training categories is estimated at \$2,355.00 per person per year. Re-certification for staff after initial training is estimated at \$840.00 per year. The total project cost for training is estimated at \$753,540.00 over the complete 10-year duration of the project.

SITE CONSTRUCTION

IC Site Materials and Methods

2060 Aggregate

Aggregate will be required for site fill in and around offices, change rooms, containers, containment structures, and haul roads. Aggregate will also be used within containment structures during grout slurry injection preparation and operations. The cost for this material is \$310,000.00.

2065 Concrete

Concrete will be required to support containment structures as structural footings. Concrete footings will also be used as a berm for containment of grout spoils. The cost for each footing item relative to each containment structure is nominally \$870.00. Ten footing units will be required for an overall project cost of \$87,000.00.

Concrete or grout is required over the area where grout injection has been completed. This material is used as a primary barrier covering the grout stabilized monolith. The depth of this barrier is 17 inches covering an area of 140 X 400 feet. Each grout barrier is estimated to cost \$354,000.00. Hence, the total cost of 10 barriers is \$3,540,000.00. This concrete grout material is assumed as a 2,500 pounds per cubic foot unconfined compressive strength, synthetic fiber reinforced, and self-leveling material. The unit cost for this material is \$120.00 per cubic yard with 10 barriers required over the project duration. Hence, the total cost of primary barrier placement is \$3,430,000.00.

Grout slurry will be produced on site by transport of dry grout materials from a railhead bulk storage plant to each grout mix plants, addition of water, and pumping through grout pumps and injection systems. On the order of 20,000 grout slurry holes are assumed per operational injection site. There are 10 operational injection sites. Slurry volume per hole is noted at 100 gal. The unit cost for grout slurry is \$2.55 per gal. 24,000,000 gal of grout slurry is required for completion of monolith injection over the total project waste volume over the 10-year project volume. The total cost of grout slurry is estimated at \$61,200,000.00. This assumes approximately 20 percent waste for spoils and cleanout.

5050 Metals

Two large containment structures will be used to confine grout slurry injection activities. Two each smaller support structures will be used to contain grout slurry mixing and shearing operations and grout high pressure pumping. One large containment structure will initially be fabricated along with smaller support structures. While these structures are under operations, another large containment structure and adjoining smaller support structures will be fabricated at another proximal location. Hence, one complete operational jet grouting system will be fabricated while the other is under operations, i.e., each system will be fabricated initially and moved to another location for a total of 11 locations. This is required to meet a 10-year production schedule.

Within each large containment structure is affixed an internal radiological containment liner and change out/decontamination liner. This liner is attached to the large metal containment structure overhead and is of approximate dimensions of 35 X 120 X 400 feet with respect to height, width, and length. This liner is sacrificed at the culmination of jet grouting operations for each location.

The cost for two of these large metal structure buildings, and spare/replacement parts assuming a one-time procurement is \$2,620,000.00.

The unit cost for each containment liner is estimated at \$160,000.00 with a total project cost of \$1,760,000.00.

Each large confinement structure is conceptually designed at an operational height of 35 feet in order to accommodate jet grouting track drill mast height and drill rod stroke. The plan dimensions are assumed at 140 X 400 feet. Fabrication and dismantlement of each structure is assumed at 80 days each.

Each small support structure is conceptually designed at 20 X 24 X 50 feet with respect to height, width, and length, respectively. Four each of these structures (2 each) will be used to support jet grout mixing/batching and high pressure pumping for each large containment structure. As per the above large containment buildings each of the small jet grouting support buildings will be moved for a total of 11 locations. One of the above small structures will be utilized as a project maintenance and shop facility. The project maintenance and shop facility will not be moved from its original location.

The cost for 5 support structures is estimated at \$225,000.00 assuming a one-time procurement over the life of the project.

15050 Mechanical

Two large structures will be used to confine jet grouting injection activities. To support work in these structures for year-round operations, heating, air conditioning, and ventilation (HVAC) will be required. Heating will be provided with high capacity propane or equivalent gas. Propane or equivalent gas will be supplied onsite by a 15,000-gal storage tank or two 7,500-gal tanks. Propane storage will require a fenced, lined, and barricaded area. Air conditioning will be supplied with evaporative cooling.

Air circulation within each large containment building will be filtered continually through high capacity high efficiency particulate air filters. HEPA filters will be tested by others.

Two HVAC complete systems are estimated at a one-time cost of \$2,500,000.00. This cost assumes utilization of two HVAC complete systems for 5 years, replacement and operation of the systems for the remaining 5 years. Also included are all spare parts and related materials. The cost of propane over the 10-year life of the project is estimated at \$1,000,000.00

16050 Electrical

Two camera systems are required for evaluation of operations of jet grouting drilling, grout slurry placement, spoils returns, and general equipment operations. Two camera/recording/viewing systems will be positioned to cover each jet grouting track drill injection module. This configuration assumes 2 of the above systems per containment structure, or four systems overall.

The one-time cost of these systems including recording media for all grout slurry injection holes is estimated at \$280,000.00.

R130 Special Construction

Three gantry modules capable of movement longitudinally and laterally in a planar configuration over the footprint of the waste location within a large containment structure are required. Two used operationally, and 1 as a backup. This is required in order to meet production schedules. These gantries additionally are required to penetrate through the soil overburden and into the waste zone containing soil matrix materials and waste materials. Penetration will be achieved by insertion of drill steel and appropriate drilling bits and jet grouting appurtenances. Hence, the gantry is functionally a mechanical system capable of three-dimensional operation, i.e., in the x, y, and z axes. The gantry and attached track drill mast and drilling assembly will be powered by a hydraulic power pack operated outside of the primary large metal confinement structure. Operation of the gantry will be remotely by radio control.

The cost of each gantry and all associated railings, hydraulics, equipment, power supplies, servomotors, radio controllers etc, is estimated at \$2,100,000.00 with a total one-time project procurement cost of \$6,300,000.00.

It is assumed that a railhead located at the site will be used for delivery and offloading of dry bulk materials from hopper rail cars. Due to the volume of dry bulk materials required per unit time over the duration of the project, 2 large dry bulk storage silos located at the railhead, and 3 smaller silos located proximal to each jet grouting mixing/batching operation are required for efficient supply of grout former materials to each injection operation. Two large silos and mechanical/pneumatic of offloading equipment with a storage capacity of nominally 550 tones each will be constructed at the railhead. Dry bulk materials will be offloaded from these silos as needed and transported to one of 3 each smaller silos servicing each jet grouting operation.

The total cost for these 5 items as a one-time project cost is estimated at \$691,000.00.

Three water portable heated water tanks will be required to support each jet grouting mixing/batching operation, i.e., two operational and one backup tank as above.

The total cost for these tanks over the live of the project is anticipated at approximately \$36,000.00.

Crews

Summary

Administrative staff will be required at conception and throughout the duration of the project. This administrative function is assumed totally dedicated to the project and works directly for the project including interfacing with site personnel and programmatic staff from the site. A building construction crew working under the direction of the administrative staff will be required to build containment building structures, dismantle containment structures, and reassemble these structures over the duration of the project. The building construction crew will also erect bulk storage and materials handling facilities and equipment as well as dismantlement and decommissioning of the site on project completion. Operations crews will complete operations of equipment and support equipment directed to placement of subsurface grout materials.

Administrative staff will be required during one shift with one superintendent working off shift. The building construction crew will only work one shift. Two operations crews will be required, i.e., one crew per two shifts. Shifts are assumed as 10 hours in duration over a 5-day workweek.

Administration

A project general manager will be required with the responsibility of overall project administration, operations, and control. A site superintendent for each of two shifts will be required for supervision of all site operations. One project engineer will be required to support site engineering and to serve as an assistant superintendent. Two administrative assistants will be required to perform clerical and related functions in support of all administrative staff. One bookkeeper/timekeeper will be required to perform accounting and payroll activities. One planner/scheduler will be needed to assist in development and projection of task activities and to determine staffing requirements. An industrial hygienist will be required to develop and control safety and health programs, one per shift. Two health physics supervisors will be required to control and manage radiological programs, one per shift. A quality control administrator will also be required to develop and manage quality programs and to perform inspections. The total administrative staff consists of 13 persons.

Building Construction

Building construction activities will require a dedicated crew consisting of one working general foreman, one crane and heavy equipment operator, 6 ironworkers, and one laborer. The total manpower requirement for this activity is 9. If required, additional staff may be reassigned temporarily from an operations crew.

Operations

Grout placement operations will require a substantial crew. There will be two crews per working day. Each crew consists of one working general foreman to oversee all applicable dry bulk storage and transfer, water transfer, slurry mixing/pumping/and injection, waste haulage, etc., as well as operations of all system components. This function will in turn be administered by a shift superintendent. Two mechanics will be required to perform preventative as well as general maintenance on all equipment and materials handling components. One electrician and 1 instrument technician will be required to perform component installation, operations, and disassembly related to electrical systems. These staff will also be responsible for electrical generating systems. Four track drill operators, simultaneously operating two injection systems per shift will be needed. Jet grouting pump operations, 2 each supporting each track drill injection system, feeding grout slurry to grout injection track drill components will require 2

operators. In turn, 2 bulk mixing plants supplying mixed and sheared slurry to each jet grouting pump will require 1 operator each. Two relief operators are required to cover each jet grouting drill injection, jet grouting pump, and jet grouting batching/mixing operations as a contingency. One heavy equipment operator will be required to support operations utilizing front-end loader, all terrain fork lift, hydraulic crane, etc.. Six teamsters are required to support grout injection and related project operations. These activities include fuel transport and fueling, water transport, bulk dry materials loading/off loading and transport, parts and materials pick up and delivery, and solidified waste grout transport and landfill operations offloading. Three laborers will be required for general site and operations support including control of radiological personal protective equipment. Two radiological technicians will be required to support each grout injection track drill activity. The total operations staff for two shifts consists of 54 craftsmen. Cross craft activities are also assumed where specific training requirements are not limiting. Where required lead individuals per craft may be assigned for supervisory actions under each shift general foreman and shift superintendent.

ASSUMPTIONS

Numerous assumptions have been delineated for this project limiting the estimate of project cost and cost projections. Assumptions are listed as follows:

- Waste stabilization will be performed under a reusable structure.
- Batching/mixing of grout slurry will be completed on site (at the location of injection).
- Waste stabilization will be completed with an injection density of 20,000 injection holes per acre.
- There are 11 locations of 1 acre each with generalized plan dimensions of 140 x 400 feet.
- Nominally each grout injection hole will be 10 feet in depth through the waste depth interval and 100 gal of grout will be used per injection.
- The project duration is on the order of 10 years.
- All equipment and materials delivered to the site are new and not previously used in production.
- Weekly work duration is 10 hours per day, 5 days per week. Operations will work two shifts of 10 hours each per day for 5 days each week. Administration will work one shift of 10 hours 5 days per week.
- Weather protection will be required for equipment and personnel within confinement and support structures.
- No release of radiological or hazardous contamination will occur as a function of jet grouting operations or support activities.
- Only minimal travel and per diem will be allowed and for only administrative staff.
- Grout placement includes a 17-inch thick grout or equivalent material cap over each drilling location and no further capping is required.

- Costs are provided in government Fiscal Year 2002 and are escalated at 3% per year over the project duration of 10 years where applicable.
- All project work is covered under Price – Anderson and related nuclear and hazardous materials liability and general requirements acts.
- Intellectual property, licensing, and related fees are the responsibility of others and are not included herein.
- All state and government taxes relative to project operations excluding personnel taxes etc., are the responsibility of others if applicable.
- Site security and patrol and site utilities, i.e., overhead electrical, potable and nonpotable process water, telephone, sanitary sewer or equivalent, and noncontaminated solid waste collection and disposal are provided by others.
- HEPA filter testing, and delivery to the project site are the responsibility of others.
- Site engineering surveys and waste location surveys if required are the responsibility of others.
- Costs associated with U.S. Department of Energy and/or Maintenance and Operations or Construction management and oversight are not included in this estimate.
- Administration, engineering, health physics, quality, training, safety, operations and associated records will be transmitted to applicable contractor offices and will be the responsibility of others.
- General site operations facilities and services will be available for project staff at nominal cost including but not limited to, general site training, specific craft training, radiological and hazardous materials training, fire/patrol support and control, and physical examinations.
- At the completion of project operations, jet grouting injection and support structures will be dismantled and staged, administrative and maintenance structures will be cleaned, isolated/winterized, and equipment will be winterized and staged on site. These facilities/equipment/materials/etc. will be the property of the government.
- All equipment and materials are free on board INEEL.
- Dosimetry/dosimetry records, medical, testing and certification/medical records and associated programs are the responsibility of others.
- Safety analysis reports/reviews will be completed by others prior to contract award.
- Administrative, industrial safety and health, radiological control, quality, operations, etc., programs and plans to be used by the maintenance and operations site contractor will be adopted and approved prior to contract award.
- Engineering design of building structures and support structures/gantry systems will be the responsibility of others.

Equipment/Materials	Cost (each)	Quantity	X,Y,Z Gantry	Cover Block
PPE, Control System	\$850,000		\$850,000	\$850,000
Mobilization/Site set up	na		na	na
Aggregate (parking lot, roads, etc.)			\$360,000	\$360,000
Office trailers		5	\$225,900	\$225,900
Signs	\$10,000		\$10,000	\$10,000
Storage boxes	\$5,000		\$20,000	\$20,000
Pit building foundation	\$8,700	11	\$95,700	\$95,700
140 x 400-ft structure (shipping/insulate) 2 bldgs moved 4 times each		2	\$2,620,000	\$2,620,000
Light plant (ea) interior lighting	\$7,000	6	\$42,000	\$42,000
Liner (140 x 400 ft)	\$160,000	11	\$1,760,000	
TV 4-camera system (50K camera system; cd disc per year for 10 years)		2	\$280,000	\$280,000
Structure (24 x 50) equipment, maintenance etc.	\$45,000	1	\$45,000	\$45,000
Structure (24 x 50) for grout plant and pump	\$45,000	4	\$180,000	\$180,000
Ventilation/heat system for structure includes propane heat system w/15K gal tank; vaporating cooling system; HEPA filters; spare parts; moved 8 times; 5-year life cycle (systems required over lifetime of project)		1	\$3,500,000	\$3,500,000
480 Volt Generator 200 kw	\$38,000	6	\$228,000	\$228,000
Drill shrouds 300 holes per shroud; 30 holes per shift	\$15,000	80		\$1,200,000
Cover Blocks (11 acres)	\$25,800,000	Per acre		\$283,800,000
Cover Block HEPA system		3 units		\$145,000

Equipment/Materials	Cost (each)	Quantity	X,Y,Z Gantry	Cover Block
x,y,z gantry	\$2,100,000	3	\$6,300,000	
dry storage facility & supply system 2-550 ton silos & 3-30 ton silos (installed [10 days])	\$691,000		\$691,000	\$691,000
C-8 trackdrill or equivalent w/Jean Lutz system radio controlled; each system to last 5 years; includes one backup	\$346,000	6	\$2,075,000	\$2,075,000
Spare parts	\$30,000	30	\$900,000	\$900,000
High pressure pump each system to last 5 years; includes one backup	\$180,000	6	\$1,080,000	\$1,080,000
Pump spare parts (2000 hr kit)	\$50,000	24	\$1,200,000	\$1,200,000
Grout Mixing plant (each system to last 5 years; includes one backup)	\$75,000	6	\$450,000	\$450,000
480 volt generator 75kw	\$20,500	6	\$123,000	\$123,000
Grout mixer spare parts (2000 hr kit)	\$8,000	24	\$192,000	\$192,000
Polytank 10,000 gal	\$12,000	2	\$24,000	\$24,000
Tractor	\$91,500	4	\$366,000	\$366,000
dry product trailer	\$105,000	3	\$315,000	\$315,000
Maintenance truck Ford 550 4x4 equipped (welder; misc tools)	\$66,500	1	\$66,500	\$66,500
JLG Hili ft (80 ft) all-terrain	\$120,000	2	\$240,000	\$240,000
All-terrain forklift (ea)	\$100,000	1	\$100,000	\$100,000
Fuel truck 2000 gal	\$100,000	1	\$100,000	\$100,000
40 ton hydro	\$250,000	1	\$250,000	\$250,000
2-ton truck	\$45,000	1	\$45,000	\$45,000
Pick-up truck	\$35,000	4	\$140,000	\$140,000
Endloader	\$320,000	1	\$320,000	\$320,000
10,000 gal water-wagon	\$600,000	1	\$600,000	\$600,000
Rock bed	\$50,500	1	\$50,500	\$50,500

Equipment/Materials	Cost (each)	Quantity	X,Y,Z Gantry	Cover Block
Quality Control on equipment	\$200,000	na	\$200,000	\$200,000
Equipment service (general maint.)	\$400,000	na	\$400,000	\$400,000
Fuel (6 gal/hr per generator; 15 gal/shift per rig)			\$1,522,000	\$1,522,000
24,000,000 gal grout dry materials (includes 20% waste)	\$2.55/gal	24,000,000	\$61,200,000	\$61,200,000
Grout cap over treated pit 17 in. depth 7 ft soil cap not included	\$354,000	10	\$3,540,000	\$3,540,000
Demobilization/Site Cleanup		Na		
Total			\$92,706,600	\$369,791,600
Consulting			\$500,000	\$500,000
Required training per person Initial	178,980.00	2355.00 pp for first year	\$178,980	\$178,980
Recertification cost per person per year	574,560.00	840.00 pp/py (9 years)	\$574,560	\$574,560
*76 employees at \$137,500.00 per person; \$104 Million over 10 years	\$137,500	76	\$104,000,000	\$104,000,000
Subtotal			\$197,960,140	\$475,045,140
Performance bond	1.5%		\$2,969,402	\$7,125,677
Insurance	0.4%		\$791,841	\$1,900,181
Main office expense	3.9%		\$7,720,445	\$18,526,760
Profit	10.00%		\$9,396,014	\$37,100,000
Total			\$218,837,842	\$539,697,758
Total with—3% escalation 10 years			\$251,663,518	\$620,962,922

Personnel*

<u>Exempt</u>	<u>Need</u>	<u>Nonexempt</u>	<u>Need</u>
General Manager	1	General Foreman	1
Superintendent	2	Mechanics	2
Quality Control Administrator	1	Electricians	1
Project Engineer/Assistant Superintendent	1	Instrument Technician	1
Administrative Assistant	2	C-8 Operator	4
Timekeeper/bookkeeper	1		
Planner/scheduler	1		
Safety Industrial Hygienist	2	HP Pump Operator	2
Health Physics Supervisor	2	Mixing Plant Operator	2
1st shift personnel	9	Equipment Operator hydro, forklift, endloader	1
2nd shift personnel	4	Teamster fuel truck, h2o, 2 ton truck	3
	13	Teamster dry batch trucks, rock truck	3
<u>Building construction crew:</u>		Laborer 1 for PPE control	3
General Foreman	1	Radiological Technicians w/equipment	2
Operator	1	Relief Operator	2
Ironworker	6	1st shift personnel	27
Laborer	1	2nd shift personnel	27
1st shift personnel	9		54

*76 total at \$137,500.00 per person; \$104 million over 10 years

Administrative

Required training per person Initial	76	2355.00 pp for first year	178,980.00
Recertification cost per person per year	76	840.00 pp/py (9 years)	574,560.00
			753,540.00
		Cover block	
Performance bond	1.50% 3.5M		7.3M
Insurance	0.04% 1M		1.9M
Main office expense	3.90% 9M		19M
Profit	10% 9.3M		34.5M
Consulting			500,000.00

Appendix K

Thermocouple Data During Implementability Testing

Appendix K

Thermocouple Data During Implementability Testing

Table 1. Summary of thermocouple data for implementability testing.

	Year	Day	Hr/min	Panel temp	U.S. Grout	GMMENT-1 2	TECT HG
101	2001	107	1640	26.14	23.58	32.27	25.66
101	2001	107	1700	24.41	25.35	32.97	23.92
101	2001	107	1720	23	23.89	33.39	22.58
101	2001	107	1740	23.01	24.6	33.88	23.75
101	2001	107	1800	22.96	21.29	34.46	23.18
101	2001	107	1820	21.9	20.33	34.99	21.21
101	2001	107	1840	21.09	22.57	35.7	20.22
101	2001	107	1900	20.65	30.11	36.5	19.89
101	2001	107	1920	20.3	30.88	37.46	19.59
101	2001	107	1940	19.53	31.55	38.46	18.7
101	2001	107	2000	18.63	32.13	39.41	17.71
101	2001	107	2020	17.82	32.67	40.24	16.84
101	2001	107	2040	17.07	33.23	40.98	16.04
101	2001	107	2100	16.36	33.82	41.66	15.3
101	2001	107	2120	15.81	34.39	42.31	14.76
101	2001	107	2140	15.29	34.96	42.97	14.25
101	2001	107	2200	14.78	35.51	43.66	13.73
101	2001	107	2220	14.27	36.07	44.41	13.2
101	2001	107	2240	13.85	36.62	45.23	12.8
101	2001	107	2300	13.53	37.2	46.13	12.53
101	2001	107	2320	13.29	37.81	47.1	12.35
101	2001	107	2340	13.12	38.45	48.15	12.24
101	2001	107	2400	13.03	39.11	49.24	12.25
101	2001	108	20	13.04	39.78	50.39	12.37
101	2001	108	40	13.13	40.48	51.6	12.61
101	2001	108	100	13.13	41.18	52.88	12.66
101	2001	108	120	13.03	41.86	54.21	12.55
101	2001	108	140	12.88	42.52	55.61	12.34
101	2001	108	200	12.73	43.12	57.07	12.17
101	2001	108	220	12.55	43.69	58.61	11.93
101	2001	108	240	12.37	44.21	60.2	11.73
101	2001	108	300	12.24	44.69	61.85	11.62
101	2001	108	320	12.08	45.14	63.53	11.44
101	2001	108	340	11.81	45.57	65.22	11.16
101	2001	108	400	11.5	45.95	66.91	10.86
101	2001	108	420	11.23	46.28	68.52	10.59
101	2001	108	440	11	46.57	70	10.34
101	2001	108	500	10.83	46.83	71.4	10.15
101	2001	108	520	10.68	47.06	72.5	10.01

101	2001	108	540	10.56	47.27	73.4	9.89
101	2001	108	600	10.44	47.45	74.2	9.79
101	2001	108	620	10.35	47.61	74.8	9.7
101	2001	108	640	10.25	47.74	75.3	9.61
101	2001	108	700	10.18	47.85	75.7	9.57
101	2001	108	720	10.13	47.95	76	9.54
101	2001	108	740	10.13	48.03	76.2	9.59
101	2001	108	800	10.19	48.08	76.4	9.69
101	2001	108	820	10.28	48.13	76.5	9.86
101	2001	108	840	10.4	48.16	76.6	10.06
101	2001	108	900	10.54	48.18	76.7	10.31
101	2001	108	920	10.69	48.19	76.8	10.56
101	2001	108	940	10.95	48.17	76.8	11.01
101	2001	108	1000	11.19	48.16	76.7	11.38
101	2001	108	1020	11.39	48.14	76.7	11.6
101	2001	108	1040	11.61	48.11	76.7	11.85
101	2001	108	1100	12.6	48.07	76.6	13.41
101	2001	108	1120	13.08	48.08	76.5	14.26
101	2001	108	1140	12.95	48.03	76.5	13.29
101	2001	108	1200	12.84	47.98	76.4	13
101	2001	108	1220	12.84	47.93	76.2	13.03
101	2001	108	1240	12.74	47.88	76.1	13.1
101	2001	108	1300	12.83	47.81	76	13.69
101	2001	108	1320	13.02	47.77	75.8	13.76
101	2001	108	1340	13.3	47.71	75.7	14.29
101	2001	108	1400	13.86	47.65	75.5	15.54
101	2001	108	1420	15.02	47.59	75.4	16.74
101	2001	108	1440	15.97	47.62	75.2	16.92
101	2001	108	1500	16.44	47.65	75.1	16.64
101	2001	108	1520	16.58	47.63	74.9	18.25
101	2001	108	1540	16.89	47.61	74.8	29.43
101	2001	108	1600	17.43	47.53	74.5	30.61
101	2001	108	1620	18.03	47.61	74.4	30.89
101	2001	108	1640	17.84	47.69	74.3	31.2
101	2001	108	1700	17.46	47.71	74.1	31.45
101	2001	108	1720	17.41	47.78	73.9	31.7
101	2001	108	1740	17.09	47.86	73.8	31.97
101	2001	108	1800	16.57	47.9	73.6	32.2
101	2001	108	1820	15.95	47.95	73.4	32.44
101	2001	108	1840	15.25	47.99	73.2	32.71
101	2001	108	1900	14.56	48.02	73	33.03
101	2001	108	1920	13.88	48.05	72.8	33.45
101	2001	108	1940	13.22	48.08	72.6	34.02
101	2001	108	2000	12.6	48.1	72.5	34.72
101	2001	108	2020	12.01	48.12	72.3	35.57
101	2001	108	2040	11.62	48.12	72.1	36.58
101	2001	108	2100	11.24	48.13	71.9	37.77

101	2001	108	2120	10.66	48.15	71.7	39.14
101	2001	108	2140	9.99	48.15	71.5	40.65
101	2001	108	2200	9.35	48.12	71.3	42.25
101	2001	108	2220	8.79	48.1	71.1	43.85
101	2001	108	2240	8.25	48.07	70.9	45.31
101	2001	108	2300	7.79	48.03	70.7	46.59
101	2001	108	2320	7.26	48	70.5	47.74
101	2001	108	2340	6.692	47.95	70.3	48.81
101	2001	108	2400	6.252	47.9	70.1	49.84
101	2001	109	20	5.853	47.83	69.95	50.88
101	2001	109	40	5.622	47.76	69.75	51.95
101	2001	109	100	5.452	47.69	69.55	53.02
101	2001	109	120	5.302	47.62	69.37	54.08
101	2001	109	140	5.174	47.56	69.19	55.13
101	2001	109	200	4.905	47.5	69.01	56.18
101	2001	109	220	4.71	47.43	68.83	57.22
101	2001	109	240	4.467	47.36	68.65	58.25
101	2001	109	300	4.181	47.28	68.47	59.27
101	2001	109	320	3.959	47.21	68.29	60.28
101	2001	109	340	3.833	47.12	68.11	61.28
101	2001	109	400	3.634	47.06	67.94	62.28
101	2001	109	420	3.393	46.98	67.77	63.3
101	2001	109	440	3.011	46.9	67.6	64.36
101	2001	109	500	2.717	46.82	67.43	65.45
101	2001	109	520	2.478	46.73	67.25	66.56
101	2001	109	540	2.263	46.64	67.07	67.65
101	2001	109	600	2.108	46.55	66.9	68.7
101	2001	109	620	1.931	46.47	66.74	69.66
101	2001	109	640	1.835	46.38	66.57	70.5
101	2001	109	700	1.915	46.31	66.41	71.2
101	2001	109	720	2.176	46.18	66.22	71.8
101	2001	109	740	3.158	46.01	65.98	72.3
101	2001	109	800	4.811	45.88	65.78	72.7
101	2001	109	820	6.094	45.85	65.65	73.1
101	2001	109	840	7.59	45.73	65.47	73.4
101	2001	109	900	8.86	45.67	65.33	73.6
101	2001	109	920	10.73	45.55	65.13	73.8
101	2001	109	940	12.96	45.51	64.99	74
101	2001	109	1000	14.75	45.5	64.88	74.1
101	2001	109	1020	16.18	45.44	64.75	74.3
101	2001	109	1040	17.94	45.37	64.61	74.3
101	2001	109	1100	19.55	45.36	64.52	74.4
101	2001	109	1120	20.2	45.31	64.41	74.5
101	2001	109	1140	21.21	45.2	64.24	74.5
101	2001	109	1200	23.09	45.1	64.06	74.4
101	2001	109	1220	24.98	45.02	63.91	74.4
101	2001	109	1240	26.84	45	63.81	74.4

101	2001	109	1300	27.8	44.89	63.64	74.4
101	2001	109	1320	29.28	44.86	63.54	74.3
101	2001	109	1340	30.58	44.86	63.46	74.3
101	2001	109	1400	30.15	44.73	63.29	74.2
101	2001	109	1420	30.79	44.73	63.22	74.2
101	2001	109	1440	31.06	44.55	63.01	74
101	2001	109	1500	32.32	44.43	62.85	73.9
101	2001	109	1520	32.79	44.39	62.74	73.8
101	2001	109	1540	33.15	44.36	62.64	73.7
101	2001	109	1600	31.89	44.31	62.54	73.6
101	2001	109	1620	31.93	44.04	62.28	73.4
101	2001	109	1640	31.87	44.06	62.23	73.4
101	2001	109	1700	28.82	44.18	62.22	73.3
101	2001	109	1720	25.61	44.03	62.06	73.2
101	2001	109	1740	24.42	43.75	61.8	72.9
101	2001	109	1800	24.12	43.64	61.65	72.8
101	2001	109	1820	23.32	43.62	61.55	72.6
101	2001	109	1840	21.89	43.57	61.44	72.5
101	2001	109	1900	20.45	43.48	61.31	72.3
101	2001	109	1920	19.18	43.4	61.18	72.2
101	2001	109	1940	17.7	43.33	61.06	72
101	2001	109	2000	16.11	43.25	60.95	71.9
101	2001	109	2020	14.68	43.16	60.81	71.7
101	2001	109	2040	13.53	43.06	60.67	71.5
101	2001	109	2100	12.49	42.97	60.53	71.4
101	2001	109	2120	11.49	42.89	60.4	71.2
101	2001	109	2140	10.64	42.79	60.25	71
101	2001	109	2200	9.95	42.69	60.11	70.8
101	2001	109	2220	9.44	42.6	59.96	70.6
101	2001	109	2240	8.87	42.52	59.84	70.5
101	2001	109	2300	8.32	42.43	59.71	70.3
101	2001	109	2320	7.82	42.35	59.58	70.1
101	2001	109	2340	7.38	42.26	59.45	69.92
101	2001	109	2400	6.848	42.19	59.34	69.75
101	2001	110	20	6.242	42.12	59.22	69.58
101	2001	110	40	5.813	42.01	59.07	69.38
101	2001	110	100	5.497	41.93	58.95	69.21
101	2001	110	120	5.121	41.87	58.84	69.04
101	2001	110	140	4.787	41.79	58.71	68.86
101	2001	110	200	4.548	41.71	58.59	68.68
101	2001	110	220	4.374	41.62	58.46	68.5
101	2001	110	240	4.35	41.53	58.33	68.32
101	2001	110	300	4.367	41.45	58.21	68.15
101	2001	110	320	4.278	41.39	58.1	67.99
101	2001	110	340	4.167	41.31	57.98	67.82
101	2001	110	400	4.008	41.24	57.87	67.65
101	2001	110	420	3.881	41.17	57.75	67.48

101	2001	110	440	3.798	41.09	57.64	67.31
101	2001	110	500	3.645	41.03	57.53	67.15
101	2001	110	520	3.38	40.98	57.43	66.99
101	2001	110	540	3.012	40.92	57.33	66.83
101	2001	110	600	2.635	40.85	57.21	66.65
101	2001	110	620	2.419	40.76	57.09	66.48
101	2001	110	640	2.399	40.67	56.97	66.31
101	2001	110	700	2.623	40.59	56.84	66.14
101	2001	110	720	3.051	40.51	56.72	65.97
101	2001	110	740	3.678	40.41	56.59	65.78
101	2001	110	800	4.432	40.32	56.46	65.61
101	2001	110	820	5.453	40.23	56.33	65.43
101	2001	110	840	6.564	40.18	56.23	65.27
101	2001	110	900	7.47	40.14	56.14	65.12
101	2001	110	920	8.19	40.1	56.04	64.94
101	2001	110	940	9.03	40.03	55.94	64.81
101	2001	110	1000	10.21	39.93	55.81	64.63
101	2001	110	1020	11.66	39.85	55.7	64.47
101	2001	110	1040	13.24	39.78	55.59	64.31
101	2001	110	1100	14.78	39.75	55.5	64.17
101	2001	110	1120	16.75	39.63	55.35	63.99
101	2001	110	1140	19.13	39.64	55.29	63.87
101	2001	110	1200	21.34	39.61	55.2	63.71
101	2001	110	1220	24.11	39.54	55.09	63.54
101	2001	110	1240	25.6	39.63	55.11	63.49
101	2001	110	1300	26.52	39.57	55.01	63.34
101	2001	110	1320	27.35	39.52	54.93	63.21
101	2001	110	1340	27.47	39.54	54.89	63.12
101	2001	110	1400	26.93	39.46	54.8	62.98
101	2001	110	1420	27.13	39.35	54.66	62.81
101	2001	110	1440	27.98	39.26	54.54	62.65
101	2001	110	1500	26.92	39.4	54.6	62.63
101	2001	110	1520	24.34	39.3	54.48	62.48
101	2001	110	1540	23.77	39.19	54.36	62.33
101	2001	110	1600	22.74	39.08	54.22	62.15
101	2001	110	1620	22.66	38.95	54.06	61.96
101	2001	110	1640	24.25	38.71	53.81	61.69
101	2001	110	1700	24.87	38.88	53.89	61.68
101	2001	110	1720	23.24	38.89	53.87	61.61
101	2001	110	1740	22.13	38.79	53.74	61.46
101	2001	110	1800	21.33	38.72	53.64	61.32
101	2001	110	1820	20.48	38.67	53.55	61.2
101	2001	110	1840	19.36	38.63	53.47	61.08
101	2001	110	1900	18.16	38.58	53.38	60.94
101	2001	110	1920	17.06	38.5	53.27	60.8
101	2001	110	1940	16.14	38.43	53.16	60.66
101	2001	110	2000	15.39	38.37	53.07	60.52

101	2001	110	2020	14.71	38.31	52.97	60.38
101	2001	110	2040	14.19	38.24	52.87	60.24
101	2001	110	2100	13.69	38.19	52.78	60.11
101	2001	110	2120	13.19	38.14	52.68	59.98
101	2001	110	2140	12.57	38.1	52.61	59.86
101	2001	110	2200	11.65	38.07	52.54	59.74
101	2001	110	2220	10.74	38.01	52.45	59.62
101	2001	110	2240	9.89	37.96	52.36	59.48
101	2001	110	2300	9.13	37.89	52.25	59.35
101	2001	110	2320	8.5	37.83	52.15	59.21
101	2001	110	2340	7.97	37.76	52.05	59.07
101	2001	110	2400	7.5	37.69	51.95	58.93
101	2001	111	20	7.11	37.62	51.85	58.8
101	2001	111	40	6.75	37.57	51.76	58.67
101	2001	111	100	6.303	37.54	51.68	58.55
101	2001	111	120	5.931	37.48	51.59	58.43
101	2001	111	140	5.646	37.41	51.49	58.3
101	2001	111	200	5.312	37.35	51.4	58.17
101	2001	111	220	4.97	37.3	51.3	58.05
101	2001	111	240	4.636	37.25	51.23	57.93
101	2001	111	300	4.209	37.21	51.15	57.82
101	2001	111	320	3.909	37.14	51.05	57.69
101	2001	111	340	3.87	37.06	50.94	57.55
101	2001	111	400	3.784	37.01	50.85	57.44
101	2001	111	420	3.659	36.95	50.76	57.32
101	2001	111	440	3.562	36.9	50.68	57.2
101	2001	111	500	3.372	36.86	50.6	57.09
101	2001	111	520	3.101	36.82	50.52	56.98
101	2001	111	540	2.945	36.76	50.42	56.85
101	2001	111	600	2.794	36.71	50.35	56.74
101	2001	111	620	2.56	36.66	50.26	56.63
101	2001	111	640	2.435	36.61	50.17	56.51
101	2001	111	700	2.382	36.55	50.08	56.39
101	2001	111	720	2.717	36.44	49.94	56.22
101	2001	111	740	3.845	36.33	49.8	56.06
101	2001	111	800	5.234	36.27	49.69	55.93
101	2001	111	820	6.553	36.22	49.61	55.82
101	2001	111	840	7.93	36.2	49.54	55.73
101	2001	111	900	9.32	36.18	49.48	55.64
101	2001	111	920	10.59	36.17	49.43	55.55
101	2001	111	940	11.94	36.15	49.36	55.45
101	2001	111	1000	13.54	36.11	49.28	55.34
101	2001	111	1020	15.03	36.1	49.22	55.24
101	2001	111	1040	16.39	36.07	49.16	55.15
101	2001	111	1100	17.78	36.03	49.09	55.04
101	2001	111	1120	19.26	35.97	49	54.93
101	2001	111	1140	20.99	35.95	48.93	54.82

101	2001	111	1200	22.58	35.95	48.88	54.75
101	2001	111	1220	24.35	35.91	48.8	54.65
101	2001	111	1240	25.79	35.92	48.75	54.57
101	2001	111	1300	27.46	35.87	48.67	54.46
101	2001	111	1320	28.84	35.85	48.6	54.37
101	2001	111	1340	30.24	35.77	48.49	54.24
101	2001	111	1400	31.22	35.75	48.43	54.15
101	2001	111	1420	31.57	35.69	48.35	54.05
101	2001	111	1440	32.64	35.69	48.31	53.98
101	2001	111	1500	32.47	35.71	48.29	53.93
101	2001	111	1520	32.34	35.59	48.16	53.77
101	2001	111	1540	32.53	35.55	48.09	53.69
101	2001	111	1600	32.88	35.47	47.99	53.57
101	2001	111	1620	31.52	35.66	48.09	53.62
101	2001	111	1640	28.85	35.61	48.02	53.54
101	2001	111	1700	26.51	35.57	47.98	53.48
101	2001	111	1720	24.31	35.51	47.89	53.38
101	2001	111	1740	22.23	35.45	47.81	53.28
101	2001	111	1800	20.34	35.39	47.72	53.16
101	2001	111	1820	19.13	35.28	47.59	53.01
101	2001	111	1840	18.82	35.18	47.47	52.88
101	2001	111	1900	18.36	35.16	47.4	52.79
101	2001	111	1920	17.2	35.17	47.38	52.73
101	2001	111	1940	16.1	35.1	47.29	52.62
101	2001	111	2000	15.19	35.07	47.21	52.53
101	2001	111	2020	14.28	35.04	47.14	52.44
101	2001	111	2040	13.52	34.98	47.05	52.34
101	2001	111	2100	12.73	34.96	46.98	52.26
101	2001	111	2120	11.94	34.9	46.9	52.15
101	2001	111	2140	11.3	34.87	46.82	52.06
101	2001	111	2200	10.48	34.83	46.76	51.97
101	2001	111	2220	9.81	34.79	46.68	51.88
101	2001	111	2240	9.08	34.76	46.62	51.79
101	2001	111	2300	8.37	34.73	46.55	51.7
101	2001	111	2320	7.72	34.67	46.46	51.6
101	2001	111	2340	7.33	34.59	46.36	51.49
101	2001	111	2400	6.99	34.54	46.28	51.39
101	2001	112	20	6.6	34.52	46.22	51.31
101	2001	112	40	6.302	34.47	46.14	51.21
101	2001	112	100	6.026	34.44	46.07	51.12
101	2001	112	120	5.702	34.39	45.99	51.03
101	2001	112	140	5.561	34.34	45.91	50.93
101	2001	112	200	5.508	34.29	45.83	50.83
101	2001	112	220	5.532	34.24	45.75	50.74
101	2001	112	240	5.474	34.21	45.68	50.66
101	2001	112	300	5.367	34.16	45.61	50.56
101	2001	112	320	5.522	34.11	45.53	50.47

101	2001	112	340	5.916	34.06	45.45	50.37
101	2001	112	400	6.269	34.04	45.39	50.29
101	2001	112	420	6.388	34.05	45.35	50.23
101	2001	112	440	6.017	34.08	45.33	50.18
101	2001	112	500	5.517	34.02	45.25	50.09
101	2001	112	520	5.07	33.98	45.18	50.01
101	2001	112	540	4.502	33.96	45.12	49.93
101	2001	112	600	4.024	33.9	45.05	49.84
101	2001	112	620	3.926	33.85	44.96	49.74
101	2001	112	640	3.867	33.79	44.88	49.64
101	2001	112	700	4.133	33.74	44.79	49.55
101	2001	112	720	4.792	33.63	44.66	49.41
101	2001	112	740	5.87	33.57	44.57	49.3
101	2001	112	800	6.708	33.53	44.49	49.21
101	2001	112	820	7.82	33.49	44.41	49.11
101	2001	112	840	9.11	33.45	44.34	49.02
101	2001	112	900	10.24	33.43	44.29	48.95
101	2001	112	920	11.52	33.38	44.22	48.85
101	2001	112	940	13.02	33.36	44.16	48.76
101	2001	112	1000	14.49	33.34	44.1	48.69
101	2001	112	1020	15.86	33.36	44.07	48.64
101	2001	112	1040	17.26	33.32	44	48.56
101	2001	112	1100	18.09	33.36	43.99	48.53
101	2001	112	1120	18.69	33.29	43.9	48.44
101	2001	112	1140	19.81	33.22	43.8	48.33
101	2001	112	1200	21.24	33.21	43.75	48.25
101	2001	112	1220	22.61	33.25	43.74	48.22
101	2001	112	1240	23.88	33.21	43.67	48.14
101	2001	112	1300	25.18	33.23	43.65	48.09
101	2001	112	1320	26.44	33.18	43.56	47.99
101	2001	112	1340	27.4	33.14	43.5	47.91
101	2001	112	1400	28.43	33.19	43.51	47.9
101	2001	112	1420	28.24	33.26	43.54	47.9
101	2001	112	1440	27.53	33.19	43.45	47.81
101	2001	112	1500	27.18	33.16	43.4	47.75
101	2001	112	1520	26.92	33.1	43.31	47.65
101	2001	112	1540	25.8	33.15	43.32	47.65
101	2001	112	1600	23.9	33.1	43.26	47.58
101	2001	112	1620	23.05	32.99	43.17	47.49
101	2001	112	1640	22.21	32.93	43.12	47.43
101	2001	112	1700	21.45	32.94	43.05	47.34
101	2001	112	1720	21.18	32.87	42.95	47.24
101	2001	112	1740	21.07	32.86	42.89	47.16
101	2001	112	1800	20.62	32.85	42.84	47.1
101	2001	112	1820	19.85	32.84	42.82	47.07
101	2001	112	1840	18.63	32.84	42.79	47.03
101	2001	112	1900	17.34	32.81	42.74	46.97

101	2001	112	1920	16.31	32.75	42.66	46.88
101	2001	112	1940	15.38	32.72	42.6	46.8
101	2001	112	2000	14.39	32.7	42.54	46.73
101	2001	112	2020	13.45	32.67	42.48	46.65
101	2001	112	2040	12.69	32.63	42.41	46.56
101	2001	112	2100	12.09	32.59	42.35	46.49
101	2001	112	2120	11.58	32.53	42.27	46.41
101	2001	112	2140	11.07	32.5	42.21	46.34
101	2001	112	2200	10.61	32.46	42.14	46.26
101	2001	112	2220	10.29	32.42	42.07	46.18
101	2001	112	2240	10.03	32.39	42.01	46.11
101	2001	112	2300	9.75	32.36	41.95	46.04
101	2001	112	2320	9.44	32.34	41.89	45.98
101	2001	112	2340	9.15	32.32	41.84	45.9
101	2001	112	2400	8.91	32.29	41.78	45.83
101	2001	113	20	8.7	32.26	41.72	45.76
101	2001	113	40	8.4	32.25	41.67	45.7
101	2001	113	100	7.95	32.24	41.63	45.65
101	2001	113	120	7.62	32.2	41.56	45.58
101	2001	113	140	7.26	32.19	41.52	45.52
101	2001	113	200	6.939	32.15	41.45	45.44
101	2001	113	220	6.768	32.08	41.36	45.35
101	2001	113	240	6.851	32.03	41.29	45.27
101	2001	113	300	7.05	32	41.23	45.2
101	2001	113	320	7.16	31.98	41.18	45.14
101	2001	113	340	7.28	31.96	41.13	45.08
101	2001	113	400	7.16	31.95	41.09	45.03
101	2001	113	420	7.03	31.91	41.02	44.95
101	2001	113	440	7.09	31.88	40.96	44.88
101	2001	113	500	7.16	31.85	40.91	44.81
101	2001	113	520	7.28	31.83	40.86	44.75
101	2001	113	540	7.3	31.81	40.81	44.69
101	2001	113	600	7.31	31.78	40.76	44.63
101	2001	113	620	7.27	31.76	40.71	44.57
101	2001	113	640	7.32	31.72	40.65	44.49
101	2001	113	700	7.53	31.69	40.58	44.42
101	2001	113	720	7.93	31.65	40.52	44.35
101	2001	113	740	8.56	31.6	40.45	44.25
101	2001	113	800	9.39	31.58	40.4	44.19
101	2001	113	820	10.26			
101	2001	113	840	11.85			
101	2001	113	900	15.42			
101	2001	113	920	17.21			
101	2001	113	940	18.11			
101	2001	113	1000	18.71			
101	2001	113	1020	19.15			
101	2001	113	1040	19.47			

101	2001	113	1100	19.71
101	2001	113	1120	19.93
101	2001	113	1140	20.07
101	2001	113	1200	20.11
101	2001	113	1220	20.15
101	2001	113	1240	20.17
101	2001	113	1300	20.18
101	2001	113	1320	20.19
101	2001	113	1340	20.21
101	2001	113	1400	20.24
101	2001	113	1420	20.28
101	2001	113	1440	20.31
101	2001	113	1500	20.35
101	2001	113	1520	20.39
101	2001	113	1540	20.43
101	2001	113	1600	20.46
101	2001	113	1620	20.5
101	2001	113	1640	20.54
101	2001	113	1700	20.58
101	2001	113	1720	20.62
101	2001	113	1740	20.7
101	2001	113	1800	20.8
101	2001	113	1820	20.84
101	2001	113	1840	20.86
101	2001	113	1900	20.85
101	2001	113	1920	20.83
101	2001	113	1940	20.82
101	2001	113	2000	20.79
101	2001	113	2020	20.77
101	2001	113	2040	20.75
101	2001	113	2100	20.72
101	2001	113	2120	20.69
101	2001	113	2140	20.66
101	2001	113	2200	20.63
101	2001	113	2220	20.59
101	2001	113	2240	20.56
101	2001	113	2300	20.52
101	2001	113	2320	20.48
101	2001	113	2340	20.44
101	2001	113	2400	20.4
101	2001	114	20	20.35
101	2001	114	40	20.31
101	2001	114	100	20.27
101	2001	114	120	20.24
101	2001	114	140	20.22
101	2001	114	200	20.18
101	2001	114	220	20.15

101	2001	114	240	20.12
101	2001	114	300	20.08
101	2001	114	320	20.05
101	2001	114	340	20.05
101	2001	114	400	20.08
101	2001	114	420	20.05
101	2001	114	440	20.03
101	2001	114	500	20.08
101	2001	114	520	20.05
101	2001	114	540	20
101	2001	114	600	19.96
101	2001	114	620	19.98
101	2001	114	640	19.96
101	2001	114	700	19.9
101	2001	114	720	19.88
101	2001	114	740	19.97
101	2001	114	800	20.14
101	2001	116	1320	21.9

Appendix L

ICP-MS Evaluation of Smears and Air-Filter Samples

Appendix L

ICP-MS Evaluation of Smears and Air-Filter Samples

Discussed in this appendix are details of sample preparation and sample evaluation using inductively coupled plasma-mass spectroscopy (ICP-MS).

Dissolution Procedure for Smears

Reagents and standards:

- 50% nitric acid solution
- Surrogate spiking solution. 1,000 ppm Ho and Pr * 2.5 mL /100 mL = 25 ppm Ho, Pr (2.5% HNO₃)
- LCS Spiking Solution. 1,000 ppm terbium * 1.0 mL/100 mL = 10 ppm terbium
- 30% hydrogen peroxide solution

The smears were weighed and placed into a Teflon beaker. A 0.5-mL aliquot of the surrogate spiking solution was added to each beaker and allowed to dry. 10 mL of the 50% HNO₃ solution was added to each beaker, and a Teflon watch glass was placed on top. The samples were refluxed for ~1 hour, removed from the heat and cooled. Approximately 3 mL of the 30% H₂O₂ solution was added to each beaker, the beaker covered, and then heated for ~1 hour. 3 mL more of the 30% H₂O₂ was added and the sample reheated again. The samples were then cooled, filtered samples into 50-mL volumetric flasks, diluted to volume. Prep blanks, prep blank filters and prep spike filters were also processed along with the smears. Samples were diluted 1/10 before analysis. An internal standard (100 ppb indium) was added to each sample before analysis.

Dissolution Procedure for Filters

Reagents and standards:

- 50% nitric acid solution
- Surrogate Spiking Solution. 1,000 ppm holmium and praseodymium * 2.5 mL /100 mL = 25 ppm holmium, praseodymium (2.5% HNO₃)
- LCS Spiking Solution. 1,000 ppm terbium * 1.0 mL/100 mL = 10 ppm terbium
- 30% hydrofluoric acid solution

Seven large rectangle filters from an air sampler had previously been combined together as a single sample and placed in a bag. An 8-cm-diameter circle was cut from the center of the stack of filters. The circles were weighed and placed into a Teflon beaker. A 0.5-mL aliquot of the surrogate spiking solution was added to each sample and allowed to dry. 50 mL of the HF solution was added to each beaker, the beaker covered and heated for about 2 hours. After dissolving the glass fiber filter material, the cover was removed and the sample allowed to evaporate to near dryness. The sample was then removed from the

heat and cooled. 10 mL of the HNO₃ solution was added, the sample covered and heated for ~1 hour. The samples were then cooled, filtered into a 50-mL volumetric flask and diluted to volume. Duplicate samples for some samples were cut from the filters and processed along with the rest of the samples.

Sample Results

Results from all of the samples are listed below. Overall, the results for the smears are quite reasonable relative to the sample blanks and spikes. In an attempt to get the best detection limits for the filters, the analyst opted to use only a 1:100 dilution of the sample digest. As noted in the In recovery and the variations in the Ho and Pr, this may have caused some error in the terbium numbers, however the sample spikes seem reasonable. The extremely high concentration of other elements in the sample matrix (most notably Ba) caused some degradation in instrument performance during the filter runs because of buildup on the sampling cones and lens stack of the ICP-MS. Dilution factors of 1,000+ would probably have alleviated this problem as it did with the smears. The results with the blank filters at 1:500 dilutions were comparable to the actual samples at the 1:100 dilutions, leading to the conclusion that the filtered did not collect any measurable quantities of terbium-contaminated dirt.

	Pr 141 ng/mL	Tb 159 ng/mL	Ho 165 ng/mL	In 115 % recovery	Sample Mass (gms)	Dilution Factor	ng/smear or ng/sample	Area (cm ²)	Total Tb (ng)	ng Tb/cm ²	ng Tb/g
ISG20401fw-smear 1/10	23.957	0.007	24.058	79.7	0.1166	500.0	< 11.8				
ISG20501fw-smear 1/10	25.424	0.005	25.451	79.5	0.1138	500.0	< 11.8				
ISG20601fw-smear 1/10	24.320	0.044	24.371	79.3	0.1146	500.0	21.8				
ISG24101fw-smear 1/10	25.888	0.033	25.909	75.4	0.1211	500.0	16.3				
ISG24201fw-smear 1/10	25.671	0.019	25.806	76.8	0.1224	500.0	< 11.8				
ISG24301fw-smear 1/10	26.063	0.014	26.223	77.6	0.1228	500.0	< 11.8				
ISG24401fw-smear 1/10	26.179	0.006	26.051	88.7	0.1201	500.0	< 11.8				
ISG24401fw-smear spk 1/10	26.128	27.408	25.773	89.2		500.0					
true spk		25.000									
% recovery		109.6									
ISG24501fw-smear 1/10	24.598	0.070	24.200	83.6	0.1179	500.0	35.2				
ISG24501fw-smear spk 1/10	26.359	27.781	26.088	89.6		500.0					
true spk		25.000									
% recovery		110.8									
ISG24601fw-smear 1/10	24.641	0.011	24.494	79.9	0.1188	500.0	< 11.8				
ISG24701fw-smear 1/10	24.874	0.028	24.696	78.8	0.1230	500.0	14.2				

	Pr 141 ng/mL	Tb 159 ng/mL	Ho 165 ng/mL	In 115 % recovery	Sample Mass (gms)	Dilution Factor	ng/smear or ng/sample	Area (cm ²)	Total Tb (ng)	ng Tb/cm ²	ng Tb/g
ISG27901fw-smear 1/10	25.425	0.003	25.673	78.2	0.1140	500.0	< 11.8				
ISG28001fw-smear 1/10	24.599	-0.003	24.946	85.8	0.1145	500.0	< 11.8				
ISG28101fw-smear 1/10	26.116	-0.001	26.651	82.0	0.1122	500.0	< 11.8				
ISG28401fw-smear 1/10	25.920	0.004	25.843	93.3	0.1179	500.0	< 11.8				
ISG28501fw-smear 1/10	27.179	0.021	27.200	82.0	0.1236	500.0	< 11.8				
ISG28601fw-smear 1/10	24.384	0.024	23.926	88.3	0.1252	500.0	12.2				
ISG28701fw-smear 1/10	25.893	0.011	25.633	88.3	0.1296	500.0	< 11.8				
ISG28701fw-smear dup 1/10	26.736	0.014	26.823	79.1		500.0	< 11.8				
avg		0.013									
ISG28801fw-smear 1/10	26.544	0.056	25.997	88.0	0.1317	500.0	28.0				
ISG28901fw-smear 1/10	25.177	0.003	25.324	76.8	0.1175	500.0	< 11.8				
ISG28901fw-smear dup 1/10	25.879	0.064	25.747	92.1		500.0	32.2				
avg		0.034					32.2				
ISG29001fw-smear 1/10	25.910	0.003	25.861	76.6	0.1143	500.0	< 11.8				
ISG34701fw-H2O 1/10	26.842	0.088	24.354	87.1	100 mL	1000.0	87.8				

	Pr 141 ng/mL	Tb 159 ng/mL	Ho 165 ng/mL	In 115 % recovery	Sample Mass (gms)	Dilution Factor	ng/smear or ng/sample	Area (cm ²)	Total Tb (ng)	ng Tb/cm ²	ng Tb/g
ISG34701fw-H2O spk 1/10	26.347	25.280	23.655	89.3		500.0	12639.9				
true spk		25.000									
% recovery		100.8									
ISG52601fw-smear 1/10	26.377	0.043	25.428	92.9	0.1300	500.0	21.5				
ISG55001fw-smear 1/10	25.859	0.002	25.781	91.7	0.1180	500.0	< 11.8				
ISG55001fw-smear dup 1/10	26.329	0.010	26.398	89.7		500.0	< 11.8				
avg		0.006									
ISG55101fw-smear 1/10	26.054	0.001	26.514	83.0	0.1152	500.0	< 11.8				
ISG55201fw-smear 1/10	26.212	0.000	26.437	87.1	0.1126	500.0	< 11.8				
ISG55301fw-smear 1/10	25.599	0.017	25.518	76.8	0.1235	500.0	< 11.8				
ISG55401fw-smear 1/10	24.864	0.004	25.009	77.8	0.1983	500.0	< 11.8				
ISG62501fw-smear 1/10	25.902	-0.001	26.131	89.8	0.1169	500.0	< 11.8				
ISG62601fw-smear 1/10	26.109	-0.001	26.098	91.6	0.1120	500.0	< 11.8				
ISG62701fw-smear 1/10	24.402	-0.002	24.463	92.9	0.1149	500.0	< 11.8				
QC - 0 ng/mL - mean	0.000	0.000	0.000	89.5							

	Pr 141 ng/mL	Tb 159 ng/mL	Ho 165 ng/mL	In 115 % recovery	Sample Mass (gms)	Dilution Factor	ng/smear or ng/sample	Area (cm ²)	Total Tb (ng)	ng Tb/cm ²	ng Tb/g
Prep blk #1 1/10	26.805	-0.004	27.480	80.7							
Prep blk #2 1/10	25.697	-0.003	26.235	86.4							
Prep blk #3 1/10	25.769	-0.004	26.104	88.1							
Prep blk filter #1 1/10	27.132	0.000	27.283	79.0							
Prep blk filter #2 1/10	26.183	-0.002	26.208	84.3							
smear blk 1	21.468	0.010	23.273	72.3	0.1128	500.0	5.2				
smear blk 2	20.838	0.008	22.483	73.8	0.1175	500.0	< 4.6				
smear blk 3	21.048	0.004	22.493	76.5	0.1195	500.0	< 4.6				
smear blk 4	21.568	0.004	22.893	79.4	0.1187	500.0	< 4.6				
smear blk 5	20.828	0.006	22.143	80.6	0.1200	500.0	< 4.6				
smear blk 6	21.068	0.003	22.513	81.8	0.1221	500.0	< 4.6				
smear blk 6 spk	113.078	99.276	122.713	84.8	0.1221						
true spike	100.000	100.000	100.000								
% recovery	92.010	99.273	100.200								
Blank Smear Mass - Mean					0.1190						
Blank Smear Mass - Stdev					0.0032						
QC - 0 ng/mL - mean	0.000	0.000	0.000	75.5							
QC - 0 ng/mL - Stdev	0.004	0.003	0.003	21.6							

	Pr 141 ng/mL	Tb 159 ng/mL	Ho 165 ng/mL	In 115 % recovery	Sample Mass (gms)	Dilution Factor	ng/smear or ng/sample	Area (cm ²)	Total Tb (ng)	ng Tb/cm ²	ng Tb/g
% recovery		69.3					69.3				
ISG23201fw-filter 1/2	168.351	4.917	82.194	49.4	2.8560	100.0	491.7	351.9	491.7	1.4	172.2
ISG23301fw-filter 1/2	111.351	3.980	72.074	95.1	2.8864	100.0	398.0	351.9	398.0	1.1	137.9
ISG23401fw-filter 1/2	151.651	4.984	85.864	93.2	2.8868	100.0	498.4	351.9	498.4	1.4	172.7
ISG23501fw-filter 1/2	143.351	3.929	59.814	90.5	2.8638	100.0	392.9	351.9	392.9	1.1	137.2
ISG23501fw-filter 1/2 dup	161.551	5.270	77.274	80.0	2.9050	100.0	527.0	351.9	527.0	1.5	181.4
ISG23601fw-filter 1/2	171.951	5.784	88.544	70.6	2.9321	100.0	578.4	351.9	578.4	1.6	197.3
ISG23701fw-filter 1/2	83.231	2.549	51.514	80.0	2.9534	100.0	254.9	351.9	254.9	0.7	86.3
ISG23801fw-filter 1/2	133.451	3.815	71.644	66.7	2.8851	100.0	381.5	351.9	381.5	1.1	132.2
ISG23901fw-filter 1/2	176.751	5.834	77.854	55.8	3.1029	100.0	583.4	351.9	583.4	1.7	188.0
ISG24001fw-filter 1/2	174.351	5.565	88.294	50.3	2.8640	100.0	556.5	351.9	556.5	1.6	194.3
ISG50001fw-filter 1/2	177.551	5.173	84.864	44.2	2.8207	100.0	517.3	351.9	517.3	1.5	183.4
ISG50101fw-filter 1/2	155.151	4.803	100.614	45.4	2.8248	100.0	480.3	351.9	480.3	1.4	170.0
ISG50101fw-filter 1/2 dup	163.151	4.689	81.054	41.4	2.8467	100.0	468.9	351.9	468.9	1.3	164.7

