

**OAK RIDGE NATIONAL LABORATORY
OLD HYDROFRACTURE FACILITY TANK-CLOSURE PLAN AND
GROUT-DEVELOPMENT STATUS REPORT FOR FY 1999**

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April 2000

Prepared by the
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831
Managed and Operated by
UT-BATTELLE, LLC
For the
U.S. DEPARTMENT OF ENERGY
Under contract DE-AC05-00OR22725

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ABSTRACT

U.S. Department of Energy (DOE) facilities across the country have radioactive waste underground storage tanks, which will require either complete removal of the tank contents and tank shells or in-place stabilization of sludge heels. Complete removal of the sludge and tank shells can become costly while providing little benefit to health, safety, and the environment. An alternative to the removal of the residual wastes and tank shells is the use of in situ solidification and stabilization techniques to immobilize the Resource Conservation and Recovery Act (RCRA) and radioactive components present in waste storage tanks.

One technology for in situ remediation of tank wastes is Ground Environmental Service's (GES's) Multi-Point-Injection (MPI™) technology. MPI technology is a patented delivery system, which uses simple and inexpensive injection tools for rapid delivery of grout or other treatment agents, as well as for the emplacement of subsurface barriers. Through the use of tailored grout formulations in conjunction with a system of specially designed grout injection tools, MPI technology is capable of producing a uniform mixture of sludge and grout. Grouts can be tailored for the immobilization of specific RCRA and radioactive constituents. The system of injection tools is designed to maximize the mixing efficiency of the grout with the wastes in the tank.

MPI technology has been successfully demonstrated on the solidification of shallow buried wastes at the Oak Ridge Y-12 Plant and in large-scale pumping and mixing tests in both cylindrical and horizontal simulated waste tanks. Hot demonstration of the technology will be accomplished during the closure of the Old Hydrofracture Facility (OHF) tank at the Oak Ridge National Laboratory (ORNL) in fiscal year 2000. This report describes the closure plan for the OHF tanks and presents the status of grout formulation development at ORNL.

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ACRONYMS

ALARA	As low as reasonably achievable
ANS	American Nuclear Society
ANSI	American National Standards Institutes
ASTM	American Society for Testing and Materials
BJC	Bechtel Jacobs Company
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CSH	Calcium silicate hydrate
DOE	U.S. Department of Energy
EDX	Energy dispersive X-ray microanalysis
EPA	U.S. Environmental Protection Agency
ES&H	Environment, safety & health
FFCA	Federal Facilities Compliance Agreement
FY	Fiscal year
GAAT	Gunite and Associated Tanks
GES	Ground Environmental Services, Inc.
HES	Halliburton Energy Services
MPI	Multi-Point-Injection™
MVST	Melton Valley Storage Tanks
NIST	National Institute of Standards and Technology
OBG	Old Burial Ground
OHF	Old Hydrofracture Facility
ORNL	Oak Ridge National Laboratory
ORO	Oak Ridge Operations
OSHA	Occupational Safety and Health Act
PMP	Pulsating Mixer Pump
PPE	Personal protective equipment
QA	Quality assurance
QC	Quality control
RCRA	Resource Conservation and Recovery Act
SEM	Scanning electron microscopy
SRS	Savannah River Site
TCLP	Toxic Characteristic Leach Procedure
TFA	Tank Focus Area
TRU	Transuranic
TTP	Technical Task Plan
W:S	Water-to-solids ratio

1. INTRODUCTION

U.S. Department of Energy (DOE) facilities across the country have radioactive waste underground storage tanks (USTs), which will require either complete removal or in-place stabilization of sludge heels. Complete removal of the sludge and tank shells can become costly while providing little benefit to health, safety, and the environment. An alternative to the removal of the residual wastes and tank shells is the use of in situ solidification and stabilization techniques to immobilize the Resource Conservation and Recovery Act (RCRA) and radioactive components present in waste storage tanks.

One technology for in situ remediation of tank wastes is Ground Environmental Service's (GES's) Multi-Point-Injection (MPI™) technology. MPI technology is a patented delivery system, which uses simple and inexpensive injection tools for the rapid delivery of grout or other treatment agents, as well as for the emplacement of subsurface barriers. Through the use of tailored grout formulations in conjunction with a system of specially designed grout injection tools, MPI technology is capable of producing a uniform mixture of sludge and grout. Grouts can be tailored to immobilize specific RCRA and radioactive constituents. The system of injection tools is designed to maximize the mixing efficiency of the grout with the wastes in the tank.

MPI technology was successfully demonstrated on the solidification of shallow buried wastes at the Oak Ridge Y-12 Plant. The technology transformed a heterogeneous mixture of buried waste into a uniform monolith. The resulting monolith had a hydraulic conductivity in the range of 10^{-6} to 10^{-7} cm/s. MPI technology has also been demonstrated in large-scale pumping and mixing tests in both cylindrical and horizontal simulated waste tanks. In the initial test, a 38-ton uniform monolith of waste surrogate and grout was successfully produced. In the second test surrogate waste in two 8-ft-diam, 22-ft-long horizontal tanks was aggressively mixed with a similar reducing grout, which was developed using surrogate wastes and then proven using actual tank wastes.

This report describes the closure plan for the Old Hydrofracture Facility (OHF) tanks at the Oak Ridge National Laboratory (ORNL) and presents the status of grout formulation development at ORNL. The grout formulation development for in situ tank-closure at ORNL was initially directed at closure of the Gunitite and Associated Tanks (GAAT). Specifically, tank TH-4 was selected for demonstration of in situ tank-closure technology at ORNL. However, a change in program direction shifted the emphasis on tank TH-4 to complete removal of the wastes and thus diminished the need for in situ closure technology. This direction may be reconsidered in the future depending on the quantities of residual waste remaining in TH-4 after retrieval. A more detailed discussion of the GAAT is presented in Sect. 5 of this report.

Closure of the OHF tanks will be accomplished using MPI technology to mix the residual wastes in the tanks with a robust reducing grout. The grout formulation, which was developed for use in the GAAT, can be successfully used to stabilize and immobilize a variety of high-pH wastes. Both cold and hot tests have shown the grout to be capable of retaining RCRA and radioactive constituents, such as Cr, Hg, Pb, Cs, Sr, U, and others.¹⁻³

2. OHF TANKS

2.1 TANK DESCRIPTION

The OHF site was used to dispose of radioactive waste by injecting grout into shale formations 1,000 ft below ground. When operations ended, ~53,000 gal of radioactive transuranic (TRU) waste was left in five USTs at the site. Because of the age of the tanks, the radioactive TRU waste was retrieved and transferred to the Melton Valley Storage Tanks (MVSTs) for processing and disposal during the MVST-TRU Waste Treatment and Disposal Project.

The OHF tanks at ORNL consist of five horizontal, carbon-steel tanks, ranging in capacity from 13,000 to 25,000 gal. The tanks are 8 to 10.5 ft in diameter and 23 to 44 ft long. Two of the tanks are rubber lined. Table 1 provides a summary description of each of the tanks and an estimate of the residual inventory of waste present. The tanks were in service from 1963 through 1980. Each tank has an 18-in.-diam riser in the center and a 26-in.-diam riser on each end. The tanks contained an estimated 9,000 gal of remote-handled TRU sludge and 44,000 gal of supernatant before the retrieval operations. A borehole miner with an extendable nozzle was used to retrieve the sludges and supernates from these tanks.

Table 1. OHF tank descriptions

Tank	Nominal capacity (gal)	Diameter (ft)	Approximate length (ft)	Material	Residual inventory (gal)	Internal components
T-1	15,000	8.0	44.0	Carbon steel	111	Multiple air spargers
T-2	15,000	8.0	44.0	Carbon steel	222	Multiple air spargers
T-3	25,000	10.5	42.5	Carbon steel; rubber lined	40	Multiple air spargers and internal connections
T-4	25,000	10.5	42.5	Carbon steel; rubber lined	373	Multiple air spargers and internal connections
T-9	13,000	10.0	23.0	Carbon steel	228	Multiple air spargers and submersible pumps

The baseline approach for removing bulk sludges from the horizontal waste storage tanks was to use traditional single-point sluicing technology, which requires large quantities of sluice water and cannot effectively be used for heel removal. Deployment of the

borehole miner with an extendible nozzle at the OHF resulted in reductions in the amount of sludge water required to remove bulk quantities of sludge and allowed more focused efforts on hard-to-remove sludges.

The diverse geometry of the OHF tanks provides a unique opportunity to demonstrate a closure technology on a variety of horizontal tank conditions. The diversity of the tank geometry and internal conditions will serve to demonstrate the utility of MPI technology over a wide range of conditions including a short tank, moderately long tanks, rubber-lined tanks, and tanks with internal obstructions.

2.2 DESCRIPTION OF RESIDUAL WASTE

The composition of the sludge and supernate in the OHF tanks before their retrieval is given by Keller, Giaquinto, and Meeks.⁴ The major metals observed in the tank sludges consist primarily of Th, Ca, Al, and Fe at average concentrations across the tanks of 60,107, 23,837, 15,332, and 14,492 mg/kg, respectively. Other metals in the sludges include Na, Mg, U, K, Sr, and Mn at average concentrations across the tanks of 7,398, 2,439, 2,944, 2,630, 604, and 310 mg/kg, respectively. The major radionuclides, other than Th and U, in the tank sludges consist primarily of ⁶⁰Co, ⁹⁰Sr, ¹³⁷Cs, and ¹⁵²Eu with average concentrations across the tanks of 5.85E+04, 1.22E+07, 4.79E+05, and 4.82E+04 Bq/g, respectively. Other radionuclides include ²³³U, ²³⁸Pu, ^{239/240}Pu, ²⁴¹Am, and ²⁴⁴Cm with average concentrations across the tanks of 8.37E+03, 2.01E+04, 7.03E+03, 1.69E+04, and 2.69E+05 Bq/g, respectively.

The remaining waste volume in the OHF tanks is given in Table 1. The volume measurements were made using differential pressure measurements across bubbler tubes, which have since been removed from the tanks. A small quantity of additional flush water was added to each tank after the volume measurements in Table 1 were made. These volumes are unaccounted for in Table 1. The unaccounted waste volume may be significantly more than the amount reported in Table 1 for tank T-9, because of the addition of large amounts of flush water during equipment dismantlement. It is estimated that the residual slurry in the OHF tanks contains no more than ~30 wt % solids.

3. CLOSURE APPROACH

3.1 MISSION

The mission of the OHF tank-closure demonstration is to close the OHF tanks in a manner consistent with required regulations while providing a detailed example that the process and technology used can also be employed to close other, similar tanks within the DOE Complex.

3.2 OVERVIEW

The OHF site must be prepared, and inventory estimates for residual waste in the tanks must be verified before closure operations. As discussed in Sect. 2.2, a significant amount of water may be present in tank T-9. Some of this water may need to be removed before injecting grout into the tanks. Other site-preparation activities will primarily consist of removing the equipment and interface hardware used during the retrieval of waste from the tanks.

Once the site-preparation activities have been completed, the MPI technology will be deployed to inject a specially formulated reducing grout into the wastes remaining in one or more of the five OHF tanks. Disposable grout-injection tools will be used in each tank. Flexible hoses will be used to attach the injection tools to a grout-distribution manifold. At the completion of the injection operation, the in-tank hoses will be filled with grout and disposed of in the tanks as part of the tank-closure operation. The site will be prepared, and the grout-injection tools will be placed in the tanks before the blending and pumping facilities are brought on-site. The capital equipment needed to pump the grout into the tanks will be placed outside the contamination areas and away from contaminated materials. Hardened steel piping will be used to connect the grout-distribution manifold to the grout pumps. Standard oil-field, commercial grade, hardened-steel piping and connectors will be used. Dry-blend ingredients will be transported to the site and pneumatically transferred to a truck-mounted grout mixer containing water and prehydrated bentonite gel. The resulting grout will then be fed to one or more truck-mounted grout pumps. The grout will then be rapidly fed into the tanks to mix with the residual waste already inside. The grout will be injected into each tank at a flow rate of ~400 gal/min until the tanks are about one-third full. Then the flexible injection hoses will be cut and pushed into the tank for disposal. During injection, the grout-supply pumps will operate at ~6,000 psi, while the tanks will remain at or near atmospheric pressure. After the high-pressure-injection step, the grout-supply pressure will be reduced, and the tanks will be filled using low pump pressure.

3.3 CLOSURE CRITERIA

No major closure criteria have been imposed on the OHF tank closure. The grout used during closure must be substantially equivalent or superior to a flowable fill material, which has been used during tank closures at ORNL. The recipe for the flowable fill is a low-strength Harrison Mix 80, which consists of 600 lb of Type F fly ash (supplier: Southcast Flyash Co.), 50 lb of portland Type II cement, 2,400 lb of concrete sand (either manufactured or river run), and 50 gal of water. This type of mix has no free water and is

primarily used to provide structural stability to the tank and prevent subsidence and further additions to the tanks. The grout, which is to be used in the MPI process, offers superior strength and added waste-retention properties for the RCRA and radioactive constituents present in the tank heel.

3.4 ORGANIZATION AND CONTRACTING MECHANISM

ORNL will contract with GES to provide the tooling, materials, cold-checkout, and designs for the technology for the tank- closure demonstration. Bechtel Jacobs Company (BJC) will contract for the actual on-site hot-demonstration services. Funds for the tank-closure action are being provided jointly through the DOE Environmental Management (EM) Technology Development Program (EM-50) Tank Focus Area (TFA) and the Environmental Restoration Program (EM-40). BJC is the management and integration contractor responsible for the OHF site and will provide oversight and coordination of the entire closure action. The DOE EM-40 organization will provide funding to BJC for coordination of infrastructure support, site preparation, and final site dispositioning for the closing the OHF.

3.5 ROLES AND RESPONSIBILITIES

The detailed roles and responsibilities of ORNL, BJC, and GES are listed in the appendix. The performance of the hot demonstration of the use of the MPI process during the closure of the OHF tanks is a cooperative effort with the following breakdown of responsibilities:

1. ORNL has funding and contractual responsibility for designing and fabricating tooling, procuring long lead-time materials, and reporting and conducting a cold-checkout of the systems required for implementing the MPI process.
2. BJC has funding and contractual responsibility for site preparation, the on-site hot demonstration, and oversight of the OHF tank-closure actions.
3. GES is responsible for providing the technology, equipment, and operators necessary to demonstrate the use of the MPI process during the closure of the OHF tanks. GES will be a subcontractor to both ORNL and BJC or to BJC's prime subcontractors.

4. CLOSURE SCHEDULE

Closure of the OHF tanks is currently scheduled for the middle of fiscal year (FY) 2000. The tank closure is scheduled for completion well in advance of a Federal Facilities Agreement milestone for completing a remedial action report on the closure of the OHF tanks and two run-off basins. The remedial action report is due to the State of Tennessee by the end of FY 2000 and is BJC's responsibility.

Initiation of the tank-closure activity is dependent on (1) the availability of funding, (2) successfully negotiating subcontracts with GES for performing the hot demonstration, and (3) successfully negotiating a subcontract with a site preparation/support contractor.

5. GROUT-DEVELOPMENT STATUS

5.1 SUMMARY OF PAST ACTIVITIES

The GAATs were constructed at ORNL between 1943 and 1951 and were used for many years to collect radioactive and chemical wastes, which were generated by ORNL operations. These tanks are currently inactive and have not been used to collect waste solutions and sludges for many years. Much of the sludge that accumulated in these tanks was removed and disposed of in the 1980s. Thus, some tanks are virtually empty, while others still contain significant amounts of sludge and supernatant. The sludges contain high levels of radioactivity (mainly ^{90}Sr and ^{137}Cs). Some RCRA metal concentrations are high enough in the available total constituent analysis such as for the GAAT sludges to be potentially RCRA hazardous. For example, the GAAT sludges have been found characteristically hazardous for mercury based on the Toxicity Characteristics Leaching Procedure (TCLP) tests; therefore, these sludges are presumed to be mixed waste.

A grout formulation was originally developed in 1996 to work with GES's MPI process, for possibly in situ grouting of the GAAT.³ The tank-sludge remediation method that is currently preferred by DOE is to exhume the tank sludge via sluicing and pump the diluted sludge to another holding tank for future treatment. Current exhumation techniques rely primarily upon water-jetting technology coupled with various slurry-pumping methods. Sludge-volume increases can be on the order of 500% of the original sludge volume to mobilize and remove the waste from the bottom of a tank. After exhumation, residual contaminated material remains in the walls and at the bottom of the tank (heel material). Exhumation does not address issues related to infiltration of surface water back into "empty" tanks, nor is the long-term structural stability of the tanks addressed. The temporary storage of the exhumed sludge only postpones future considerations of longer-term treatment at an associated increased cost as a result of the issues related to exhumation and temporary storage.

A project is currently underway to move the remainder of the GAAT sludge and supernatant to the MVST for solidification and disposal with other ORNL tank sludges. The robotic apparatus being used for this removal does not fit into the smaller TH-4 tank, so in situ grouting was proposed to demonstrate closure of this tank. A cold-field campaign in 1997, which was targeted at TH-4 closure, demonstrated the efficacy of the MPI technique in mixing the sludge and grout formulation and in suspending the sludge and forming a satisfactory monolith.² Bench-scale testing with surrogate and actual TH-4 sludge in 1998 confirmed the suitability of this grout for in tank solidification of the TH-4 sludge and closure of Tank TH-4.¹ However, the decision has subsequently been made to retrieve the TH-4 sludge and transfer it to the MVST using Russian Pulsating Mixer Pump (PMP) technology, which will be used to mobilize and mix the waste in the tank. In situ grouting of the TH-4 heel remaining after retrieval is not thought to require the aggressive agitation produced by the MPI process. However, this decision may be revisited if the quantity of waste remaining after retrieval is sufficiently large. Closure of TH-4 has been delayed from FY 2000 to FY 2001. This report summarizes the grout-

technology status for the in situ grouting of the TH-4 tank heel. Guidance is provided on the ground granulated slag to be used in the grout formulation. The evolution of the grout is presented from the original, more fluid grout developed in the laboratory to the thicker grout, which has proven to be pumpable in cold-field demonstrations.

5.1.1 Formula Development

Bench-scale testing at ORNL in 1996 proved that a grout formulation based on slag, fly ash, and clay prevented the physical segregation (35 wt % waste loading) of zeolite-sized particles and produced little or no free water upon curing.¹ The compressive strength of the stabilized RCRA-radioactive surrogate was relatively low at 100 to 500 psi, but that pressure can adequately ensure the stability of the tank shell. These low compressive strengths allow for conventional exhumation (clamshell or backhoe) of the stabilized waste in the future (if required). The RCRA metals [mercuric chloride salts, lead, and chromium(VI)] were stabilized within TCLP limits, and the grout provided excellent leach resistance for the radionuclides (⁸⁵Sr and ¹³⁷Cs). Leachability indices were measured in excess of 10 using the American Nuclear Society (ANS)/American National Standards Institute (ANSI) ANS/ANSI-16.1. Processability of the grout proved the most difficult property to evaluate in the laboratory. Halliburton Energy Services (HES) engineers indicated that a Fann™ viscometer reading could be used as a rough guide of known pumpable grouts for the high-pressure pumps used by MPI. This guidance was used as the target processability property in the grout-formulation development. The cold-field demonstration proved that a much thicker formulation was pumpable. Thus, the laboratory formulation was conservatively fluid, and much less bentonite–water gel was required in the field to make the grout pumpable.

The successful development of a grout capable of immobilizing all the contaminants of concern for Gunitite tank TH-4 resulted in an in-tank cold-field demonstration of the MPI process.² A near full-scale mock-up of tank TH-4 was set up at the Duncan, Oklahoma, test facility of HES in December 1997. The success of the MPI system to deliver the ORNL grout was confirmed by the exhumation of the treated sand-clay surrogate. Visual observations confirmed that the internal structure of the monolith was uniform across the 15-ft diam and 40-in. thickness of the treatment. Eleven tons of surrogate were transformed into a relatively homogeneous 32-ton monolith in less than 8 min of field operations. Furthermore, the procurement for the test setup, execution, and report documentation of the cold test was accomplished in about a month.

Additional bench-scale testing (surrogate and actual TH-4 sludge), which was conducted in 1998 with the field adjustments to a thicker grout and adding the field fluidizer, confirmed the laboratory performance observed in 1996 with the more fluid grout.³ These results proved that the 38 ton of dry blend, which was mixed by HES, during the in-tank cold-field demonstration, was also capable of immobilizing all the RCRA metals and radioactive contaminants present in the TH-4 sludge. The properties of the kilogram-size samples used during bench-scale testing were successfully replicated on a much larger scale (38 ton), which is similar in scale to that required for hot deployment. The HES bulk, dry-blend material was used in bench-scale tests to immobilize RCRA metals below the universal treatment standards even at a waste loading as high as 65 wt %. The 1998 ORNL studies also revealed that the unconfined compression strength of the treated

GAAT sludge was not well correlated to the grout's ability to immobilize RCRA metals and radionuclides. A strong correlation was established between leach resistance and the percentage of slag, fly ash, and cement in the final mixture. The composition of the monolith formed during the cold-field demonstration had the highest concentration of these three constituents when compared with nine other grout formulations tested at ORNL.

5.1.2 Guidance On Slag

5.1.2.1 History

Blast furnace slag is a normal by-product of the iron and steel industry. In general, the slag is cooled in two ways (1) air-cooling and (2) water-quenching (granulation). Air-cooling produces inert crystalline slag, which is useful as an inert fill material but useless as a cement substitute. The essential components of slag are the same oxides as are present in portland cement, but “. . . for use as a cement, rapid cooling is necessary to quench the material to form a reactive glass and to prevent the crystallization of unreacted chemical compounds.”⁵ Granulated slag hydrates slowly on contact with water, but is activated by caustics (e.g., calcium hydroxide or sodium hydroxide, calcium sulfate, sodium carbonate, and sodium sulfate).⁵ The granulated slag is finely ground and marketed as a substitute for cement. The granulated blast furnace slags “. . . have physical properties similar to those of ordinary portland cements. The distribution of particle size and the surface area of blast-furnace slags depend on the method of manufacture, but in general their fineness is similar to that of Portland cements.”^{5,6}

Slags have been substituted for cement for decades.⁷ Slags hydrate slowly to form calcium silicate hydrate (CSH), the same product formed by cements, but slag alters the morphology and properties of the final product, sometimes in subtle ways, but beneficially in general.^{5,6,8-14}

1. Early strength development is slower.
2. Heats of hydration are lower.
3. Sulfate resistance is improved.
4. There is lower permeability despite increased total porosity.
5. There is improved frost resistance.
6. There are lower ionic diffusion rates.
7. There is increased salt stability.
8. There is reduced setting rate.
9. There is extended working time.
10. Pore water contains sulfur species in addition to hydroxide anions.
11. There is high pH and low oxygen potential.
12. There is reduced solubility of most contaminants.
13. There is a reduced rate of corrosion of steel containers.
14. Other physical and mechanical properties similar to portland cements (e.g., density and compressive strength).

A slag-cement combination of 75:25 virtually eliminates calcium hydroxide as a hydration product (i.e., the presence of excess slag prevents buildup of this cement hydration product).⁵ This implies that the proper proportion of slag to cement can replace cement-fly ash to stabilize ⁹⁰Sr. In addition, a combination of 85:15 or higher slag produces a strong reducing environment within the matrix, which is an environment suitable for reducing pertechnetates or chromates.^{15,16} Thus, slags have been used in grouts developed for radioactive and mixed wastes for a long time.^{14,15,17-24}

Table 2 gives three American Society for Testing Materials (ASTM) strength grades of ground, granulated blast furnace slag for use in concrete and mortars as based on the slag activity index.²⁵

Table 2. Slag activity index for various ASTM slag grades

ASTM slag grade	Minimum average slag activity index, %	
	7 days	28 days
80	...	75
100	75	95
120	95	115

These slag grades are important for construction, but they are not necessarily important for waste treatment, where strength requirements are usually minimal. The chemical properties normally present in commercially available slag are the most important property for waste treatment and are generally not specified in the ASTM standard. Perhaps the most important property (for waste treatment) measured in the standard is the air permeability or Blaine fineness, although no limits are specified.²⁷ Finer slag usually means a lower permeability, not only in the dry slag, but also in the resulting cementitious matrix. A lower permeability implies “improved resistance to frost, lower diffusion rates of ions through the hardened cement, and improved stability in the presence of salts, such as chloride and sulphate.”^{5,12} Typically, portland cement has a Blaine fineness of 3,000–4,000 cm²/g and slag, of 4,000–5,000 cm²/g, but slag >5,000 cm²/g or even >6,000 cm²/g can sometimes be acquired. In general, the finer (i.e., higher Blaine fineness values) is better, although it is unlikely that special requests for finer grinding is worth the additional costs. Any commercially available slag, which is suitable as a cement substitute, generally improves the matrix properties and imparts the desired properties to the final waste form. A finer size and/or higher grade usually implies a faster set with a stronger product. Thus if delayed set or a weaker excavatable product is desired, then a course size and/or lower grade slag should be specified.

5.1.2.2 Characterization of Slags Used in Cold Demonstration and Bench-Scale Testing

HES purchased slag from the Lone Star Co. for the cold demonstration in December 1997. Samples of the field blend and the individual ingredients used in this cold demonstration were provided for the bench-scale testing done in 1998. These field ingredients were used for all the bench testing done in 1998, except for the slag used in the sensitivity testing. The sensitivity testing required varying the composition of the

individual ingredients in the grout formulation—meaning that the field blend could not be used, only the samples of the individual ingredients. However, there was not enough of the field slag (from Lone Star) for the bench-scale sensitivity tests, and the field supply of this slag had been discarded after the cold demonstration and was no longer available. A slag from the Holnam Co. was substituted for the field slag in these sensitivity tests. Because both slags were commercially available products and met the American Society for Testing and Materials (ASTM C 989) criteria, their use was considered an acceptable substitution.

Both slags gave acceptable leach-resistance performance, but lack of strength development or slower strength development was noted in the grouts prepared using the Holnam slag (as compared to the HES field slag). Significant differences in performance, such as rate of strength development, are not unusual for these construction raw materials and are considered acceptable as long as they meet the industry criteria for final strength development. Since the developed formulation was already a low-strength material, this was not considered a serious drawback because strength development was not part of the criteria. This observed difference did prompt an attempt to characterize any observable difference between the two slags. This section reports the results of these characterization efforts. This characterization consists of the following:

1. total composition by total dissolution analysis (nitric acid and hydrofluoric acid digestion followed by inductively-coupled plasma spectroscopy analysis of the elements in the extract),
2. microanalytical examination (X-ray diffraction, energy dispersive X-ray microanalysis (EDX), optical microscopy examination, and scanning electron microscopy (SEM) with dot matrix mapping),
3. head-to-head comparison of strength development in the jetting slurry (the mix of dry blend and bentonite slurry, which is to be pumped and jetted into the tank), and
4. measurement of the total sulfur content.

Table 3 lists the composition measured in both slag samples by total dissolution analysis. Although significant differences in the measured composition can be found between the two slags, it is not clear that these differences account for the observed difference in strength development. Basically, the slag compositions listed in Table 3 are the expected composition for a slag (i.e., an alkaline-alkali-alumino-silicate matrix), and the observed differences in Table 3 may be fairly typical of the differences among slags.

The diffractograms and EDX spectra are included in the appendix of the 1998 report.³ Comparisons of the diffractograms and the EDX spectra did not show any significant differences between the Holnam and the HES slags. As expected for granulated slags, the diffractograms show that both materials are largely amorphous, and the EDX elemental analysis shows that both samples contain Al, Ca, Cu, Fe, K, Mg, Mn, S, Si, Ti, and Zn in roughly the same amounts and in similar proportions in the two slags. The EDX results confirm the measured total composition results for the metals but, more

importantly from the perspective of contaminant stabilization, they indicate that both contain roughly the same amount of sulfur.

Table 3. Total concentrations measured in the slag samples (mg/kg)

Analyte	Lone Star slag		Holnam slag		NIST standard glass ^a		
	1	2	1	2	1	2	Actual from NIST
Silver	<10	<10	<9	<10	115	156	254
Aluminum	48,174	36,407	37,180	45,712	10,114	10,106	10,580
Arsenic	<40	<40	<37	<39	335	352	
Barium	876	728	317	374	392	398	
Beryllium	5	4	11	13	426	433	
Calcium	221,852	180,762	239,970	259,390	74,999	71,472	85,700
Cadmium	<4	<4	<4	<4	259	268	
Chromium	28	27	62	70	404	410	
Copper	<16	26	<15	<16	424	443	444
Iron	7,492	7,134	3,933	4,722	473	566	458
Potassium	2,566	2,562	3,206	3,599	466	950	461
Magnesium	37,404	27,591	56,034	67,373	749	852	
Manganese	3,049	2,992	5,464	5,991	425	430	485
Sodium	4,233	5,957	5,185	2,246	91,447	90,499	103,800
Nickel	<16	<16	<15	<16	424	444	459
Lead	<28	<28	<26	<27	392	409	426
Selenium	<40	<40	<37	<39	48	91	
Antimony	<40	<40	<37	<39	380	393	
Silicon	229,204	268,531	234,043	272,459	314,473	288,718	336,570
Strontium	347	297	244	284	401	413	515
Thorium	1,508	1,487	1,460	1,572	675	<491	457
Titanium	3,087	3,030	9,696	11,027	391	391	437
Thallium	<32	<32	<29	<31	81	135	62
Uranium	892	865	833	890	1,122	1,005	462
Vanadium	<40	<40	<37	<39	434	441	
Zinc	43	56	44	52	444	453	433
Zirconium	<40	<40	240	249	466	462	

^aA National Institute of Standards and Technology (NIST) standard was used to check the accuracy of the technique.

Microscopic optical analysis does show a difference between the slags. The HES slag appears to consist of smaller glass particles and bigger crystalline particles than does the Holnam slag. This difference would be consistent with slower strength development in the Holnam slag, but this is not necessarily the definitive cause of the observed slower strength development. The glassy particles are the key to the slag activity and its participation in the cementitious reactions; hence, smaller glass particles in the HES slag imply a faster rate of reaction and strength development. However, the qualitative observation of larger glass particles under an optical microscope does not definitively

establish a mechanistic relationship of lower rate of strength development. The samples were also evaluated using SEM and dot matrix mapping in an attempt to better quantify the glass particle size difference. Glass and crystalline particles could not be distinguished under SEM, and little difference was noted between the two slags. It was hoped that a tagging element could be determined to distinguish between crystalline and glass particles by dot matrix mapping, but no such distinguishing element could be discerned.

The observation of a difference in grout strength performance in 1998, (with the only difference in composition being the two slags) was based on only two observations, leaving open the question of whether there was a real difference between the two slags or whether there is some other explanation, including experimental error. For this reason, the strength development of the jetting slurry made with the cold-field blend containing the HES slag was monitored and compared to the strength development with a blend made in the laboratory using the Holnam slag and all the other ingredients from the field demonstration. Ostensibly, the only difference between these two grout mixes was the slag used. Since only a limited supply of the field materials remains, especially of the individual ingredients, only a limited number of samples were prepared, giving only a limited number of data points. Table 4 lists the unconfined compressive strengths measured and their cure time.

Table 4. Compressive strength development of the jetting slurry developed for TH-4

Time (d)	Unconfined compressive strength (psi)		Ratio of Holnam strength to HES strength
	HES field blend	Laboratory blend with Holnam slag	
1	<100	---	---
2	948	280	0.295
3	1686	798	0.473
11	3353	---	---
15	4090	---	---
21	3220	---	---
29	3168	---	---

Fig. 1 illustrates the slower strength development of the grout prepared with Holnam slag under these conditions, apparently confirming a real difference between the two slags with regard to rate of strength development. As stated previously, there was no strength criterion for the formulation developed, and the final monolith was low strength. Reformulation can improve the strength, if needed, and caustic activation can increase the rate of strength development, if needed.

Both slags had satisfactory leach resistance, and the sulfide content of the slag is a key to stabilizing some contaminants, such as, chromates, mercury, or technetium. For this guidance document, the total sulfur content of both of the slags that have proven

laboratory leach resistance was considered important. The total sulfur and carbon content of both slags as measured by a LECO™ analyzer is given in Table 5.

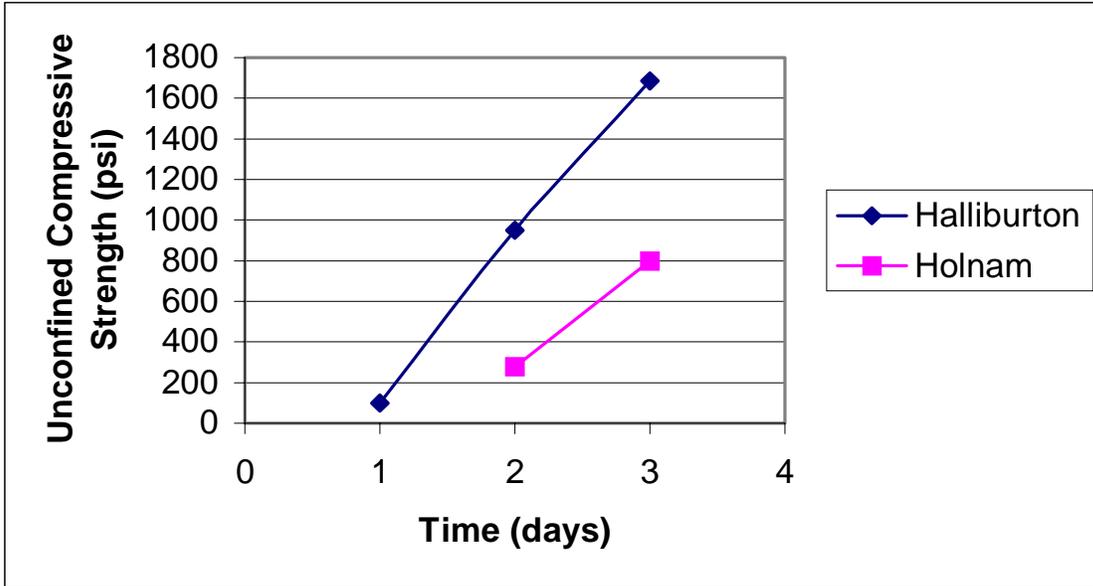


Fig. 1. Compressive strength development of the TH-4 jetting slurry.

Table 5. Sulfur content of slags

Slag	Total sulfur (wt %)	Total carbon (wt %)
HES	0.80	0.25
Holnam	0.86	0.10

5.1.2.3 Recommendations on Slag

The ground, granulated, blast-furnace slag used for the in situ grouting of tank wastes should meet the requirements of ASTM C 989, “Standard Specification for Ground, Granulated Blast-Furnace Slag for Use in Concrete and Mortars.”²⁵ Although this standard was developed for the substitution of slag for cement in construction, it gives access to readily available commercial sources of slag with an attendant quality assurance/quality control (QA/QC), which is satisfactory for stabilizing tank wastes. Although the ASTM standards for portland cement and fly ash contain chemical composition requirements for the basic matrix of these two materials, this is not the case for the ASTM standard for slag. The main focus of the ASTM slag standard is on its reactivity and strength development with cement, not its chemical composition. The only chemical requirement in the slag standard is for maximum sulfur content. Although strength development is not the primary focus for waste treatment, the standard does ensure that the slag in question will be a fine, glassy powder that will readily react with cement to form a cementitious matrix, CSH.

Tables 6 and 7 reproduce the tables of physical requirements and chemical requirements, respectively, for ground, granulated, blast-furnace slag as presented in ASTM C 989-89.

The slag activity index, which is used to determine the slag grade, is the ratio of the measured compressive strength of a slag-cement (50:50) mortar mix relative to the reference cement mortar without slag, reported as a percentage. The index is calculated as follows in Eq. (1):

$$\text{Slag activity index, \%} = (SP/P) \times 100, \quad (1)$$

where [term definitions as quoted in Ref. 26],

SP = average compressive strength of slag-reference cement mortar cubes at designated age, psi (MPa), and

P = average compressive strength of reference cement mortar at designated age, psi (MPa).

Table 6. Physical requirements for slag presented in ASTM C 989-89

Fineness: Amount retained when wet screened on a 45- μ m (No. 325) sieve, max %		20
Specific surface by air permeability, ASTM C 204 to be determined and reported although no limits are required		
Air content of slag mortar, max %		12
	Average of last 5 consecutive samples	Any individual sample
Slag activity index, min. %		
<u>7-d index</u>		
Grade 80	---	---
Grade 100	75	70
Grade 120	95	90
<u>28-d index</u>		
Grade 80	75	70
Grade 100	95	90
Grade 120	115	110

Table 7. Chemical requirements for slag presented in ASTM C 989-89

Sulfide sulfur (S), max. %	2.5
Sulfate ion reported as SO ₃ , max. %	4.0

Typical slag suppliers routinely test their products according to these procedures and often measure the chemical composition of their products as well. Examples of such past certification analyses can be requested before acquiring the products. Any large slag purchase should be sampled and tested by the supplier according to ASTM C 989, and the results should be provided with the shipment. A large batch of slag may be prepared and certified according to the standard and then marketed according to demand. In this case, analyses of the product are available for inspection before its purchase.

In general, finer grades of slag and higher sulfide contents improve the stabilization properties of the final waste form. Thus, given a choice, it is better to choose a slag with higher sulfide content (depending on the contaminants to be stabilized and staying within the maximum limit of 2.5%, as prescribed by ASTM C 989), lower percentage of retention on a 45- μ m (No. 325) sieve, and higher air permeability (Blaine fineness). A higher-grade slag may improve strength development and improve the basic matrix of the waste form with regard to lower permeability and porosity, but strength development is not the primary criteria for waste forms as it is in construction. Generally, mixing with waste degrades the ultimate strength and gives an inferior construction material. Since the intent in the proposed use is to mix the grout components with waste, it is not clear that the higher cost of the higher-grade construction slag is justified—provided that the lower grade still accomplishes the stabilization goals. If possible, it is better to test the actual waste with the purchased stabilizing agents prior to full-scale application in order to compare the actual performance with the anticipated performance.

Based on laboratory experience to date, commercially available slags exhibit satisfactory stabilizing performance, even at high waste loadings and/or when the final waste form has little or no rigid strength (i.e., it is compressible as in unconsolidated materials like soil). Apparently, typical slags contain enough soluble sulfide sufficient for stabilization. A slag without sulfide would not be acceptable for waste treatment, where stabilization depends on sulfide precipitation or reduction, although such a slag would be acceptable per ASTM C 989. The available evidence indicates that the slags currently marketed do contain sulfide, enough such that ASTM apparently felt compelled to limit the sulfide content in its standard. Fortunately, this maximum sulfide limit in the standard requires analysis of the slag for sulfide, so the routine certification according to the standard will contain a measurement of the slag sulfide content, and this value can then be checked for assurance that it is a finite, nonzero value for waste treatment. The minimum slag sulfide content necessary for waste disposal is uncertain without further research, but likely varies on a case-by-case basis.

5.1.3 Evolution of the Jetting-Slurry Grout

The bench-scale study at ORNL in 1996 to develop GAAT tank sludge treatment agents was a collaborative effort between ORNL and GES.¹ ORNL provided its expertise and understanding on formulating robust grouts for treating tank sludge. GES provided its knowledge of the MPI process to help ensure that the grout and delivery system were compatible and capable of producing a homogeneous monolith of treated tank sludge. The major questions answered during the ORNL bench-scale study focused on whether a slag-fly ash grout could be used to treat a GAAT tank surrogate to

- Produce leach-resistant treatment for radionuclides (especially, strontium and cesium);
- Stabilize the RCRA metals (mercury, lead, chromium) within TCLP limits;
- Uniformly suspend zeolite-sized particles (0.5- to 0.8-mm) within a 2-ft column of grout to help assure formation of uniform, monolithic treatment;
- Produce little or no free water upon setting; and
- Result in compression strengths that would support the tank shell yet leave treated waste inside the tank that could be exhumed in the future (if required).

To meet these objectives, a slag-fly ash-illite blend was mixed into a bentonite-gel slurry as the in situ grout formulation. The prehydrated bentonite gel made the formulation resistant to solids segregation and bleed-water generation, while the blend ingredients stabilized the contaminants. Issues related to the pumpability of the slag-fly ash grout could only be partially resolved during the ORNL bench-scale tests because of the limited correlation between laboratory-performance testing and the high-pressure-pump capabilities. The jetting-slurry grout, which was developed in the laboratory, was limited to a measurable Fann viscometer reading of about 150° at 600 rpm as based on rough guidance from the HES engineers. This criterion dictated the bentonite concentration that could be used and the relative amounts of bentonite gel and dry blend to be pumped. To provide the greatest treatment possible while maintaining the fluidity believed to be required for the jetting slurry, a small portion of the slag and fly ash was added to the surrogate sludge as dry material, although most of the slag and fly ash were added to the surrogate in the bentonite gel. This type of operation mimicked mechanically stowing the dry slag and fly ash onto the sludge before in situ grouting.

Table 8 lists the grout formulation developed in the laboratory in 1996 based on the criteria used at that time. Note that to meet the laboratory processability criterion, the following was required:

1. the water-to-solids ratio for the jetting slurry was 1.0,
2. the bentonite-to-water ratio for the bentonite gel slurry was 0.03, and
3. some of the stabilizing agent was to be mechanically stowed onto the waste before the in situ grouting.

However, it was later shown that the oil-field, grout-mixing equipment provided by HES could mix and deliver all dry-blend components that were required in the ORNL grout

formulation. HES's equipment eliminated the need for the mechanical stowing of slag and fly ash and thus simplified the sludge treatment process. The actual processing capability of the field equipment and the use of a fluidizer (lignosulfate) in the field not only eliminated the need for mechanical stowing, but also allowed the reduction of the water content needed to mix, pump and jet this grout formulation. This reduction in water not only decreased the water to solids ratio, but also increased the bentonite-to-water ratio. This situation emphasizes the difficulty of extending bench-scale solidification studies to full-scale field operations.

Table 8. 1996 in situ grout formulation developed for the GAAT sludges in the laboratory before the field demonstration

Component	wt %
<i>Material mechanically stowed through the top prior to in situ grouting</i>	
Fly ash	8.8
Slag	8.8
<i>Jetting slurry pumped and jetted into the tank</i>	
Bentonite (prehydrated with grout mixing water)	1.3
Grout mixing water	41.2
Fly ash	14.0
Slag	14.8
Cement	7.0
Illite	4.1

Direct injection of all the dry solidification agents in the ORNL formulation requires the mixing of the dry blend components, which are given in Table 9. The formulation in Table 9 for the slag and fly ash was obtained by adding the contributions from the jetting slurry and dry material added by stowing. A one-to-one ratio of slag to fly ash was established to make blending of bulk material easier for field applications. Using the grout water content, which is specified in Table 8, with the dry blend in Table 9 results in a water-to-solids ratio of 0.7, a reduction from the ratio of 1.0 for the jetting slurry, which is specified in Table 8.

The percentages of the dry-blend material given in Table 9 were used to make the grout injected during the 1997 cold-field demonstration of the MPI process. However, the water-to-(dry-blend)-solid ratio (W:S) was further reduced to about 0.48 by the robust characteristics of the field equipment and the addition of a minor amount (0.4%) of a dispersant (lignosulfate) recommended by HES's oil field cementing service engineers.

The cold-field demonstration grout was higher in solids and required lower injected grout water. Obviously, both are more attractive grout characteristics especially if the grout can be pumped in the field.

Table 9. Dry-blend solidification agents used in ORNL formulation

Agent	wt %
Granulated blast furnace slag	40
Class F fly ash	40
Indian red pottery clay	7
Portland Type I cement	10
Bentonite (prehydrated with grout mixing water)	3

This minimization of the grout water content in the field demonstration resulted in a bentonite-to-water ratio of 0.06. This jetting-slurry grout formulation with the addition of 0.4 wt % lignosulfate had satisfactory performance during laboratory testing in 1998. These tests were directed at the TH-4 sludge at sludge loadings of 35–65 wt %.³ It was projected that in situ grouting of the TH-4 tank without excavation of its sludge would give a sludge loading of 35 wt %. It is currently planned to excavate and remove the sludge in TH-4, leaving only a sludge heel for the in situ grouting. This sludge heel will result in a sludge loading far less, perhaps as low as 5-wt %. The dry blend formulation in Table 9 has proven stabilization and leach resistance characteristics for the TH-4 tank sludge. It also has proven solids segregation resistance at water to solids ratios of 0.48–0.70.

5.2 COLD TESTS (FY 1999)

In addition to demonstrating closure of the TH-4 tank, the MPI technique can be used in the in situ grouting closure of tanks covered by the Federal Facilities Compliance Agreement (FFCA) at two DOE sites: ORNL and the Savannah River Site (SRS). At ORNL, the demonstration plans to close the five OHF tanks that were already scheduled for closure by in situ grouting during FY 2000. At the SRS, in situ grouting is being considered for closure of 21 solvent tanks at the Old Burial Ground (OBG) and tank S21 was selected for demonstration of in situ grouting by MPI. Subsequently, two simulants were designed to represent the tank heels remaining in the OHF tanks and S21, as based on the available characterization information. Table 10 lists the composition of these two simulants. Radionuclides and RCRA hazardous components were purposely excluded from these two simulants based on the scope of the current work and the available performance criteria of the end users. Stabilization of such contaminants has been demonstrated for the grout in question at much higher waste loadings (up to 65 wt %) in the previous development work. A small organic phase was noted floating on the aqueous solution of the S21 simulant, but not the OHF simulant. This is consistent with the anecdotal description of the actual tank heels for the two sites and was expected for these two simulants, which were tested in the laboratory for compatibility with the in situ grout, after formulation modification and testing in a cold-field demonstration at Odessa,

Texas. In addition, this grout was found to be compatible with a sample of actual sludge from one of the OHF tanks. This section summarizes the results of these cold and hot tests.

Table 10. Composition of the FFCA tank simulants

OHF simulant		SRS S21 simulant	
	<u>wt %</u>		<u>wt %</u>
Water	90.00	Liquid solution	90.56
TBP	0.02	Undissolved solids	9.44
Solids	9.98	Total	100.00
Total	100.00		

Solids composition (w/o TBP)		Liquid solution	wt %
Compound	(wt %)		
		NaNO ₃	0.20
Al(OH) ₃	23.52	Na ₂ (CO ₃)	3.96
CaCO ₃	41.44	NaOH	2.17
Fe ₂ O ₃	4.22	TBP	7.42
K ₂ CO ₃	3.29	Water	86.24
MgCO ₃	5.43	Total	100.00
SiO ₂ (Ottawa sand)	16.73		
NaNO ₃	1.84	Undissolved solids	wt %
Na ₂ SO ₄	1.26	Al(OH) ₃	50.00
Na ₂ CO ₃	2.28	SiO ₂ (Ottawa sand)	50.00
Total	100.00	Total	100.00

The cold-field demonstration was conducted in Odessa, Texas, on July 27, 1999. The laboratory cold-testing for the OHF tanks and SRS OBG Tank S21 was designed based on preliminary information from this cold demonstration. For bentonite prehydration of 2 h, batch mixing the day before the injection demonstration proved that the field equipment could mix and pump a grout with density of up to 13.2 lb/gal, without a fluidizing admixture; while the Duncan demonstration proved that a grout density of up to 14.5 lb/gal could be accommodated using a better mixer and a fluidizing admixture. Consequently, jetting-slurry grout densities of 13 and 14 lb/gal were selected as the targets for cold laboratory testing with the surrogate OHF and S21 sludges. It was later revealed that using the bentonite that prehydrated overnight during the injection lowered the density that could be mixed and pumped (without a fluidizer) down to about 11 lb/gal.

The composition of the dry blend and bentonite gel, which are mixed to make the jetting-slurry grout, were fixed at the same values as those established by the Duncan, Oklahoma, demonstration for these cold laboratory tests. The target densities are achieved by varying the relative ratios of these two field ingredients. The grout density was measured as a function of this ratio in the laboratory, using the same raw materials suppliers anticipated to be used for the hot demonstration, resulting in the correlation illustrated in Fig. 2. The equation resulting from linear regression was used to fix the ratios at 1.34 and 1.80 g dry blend/g gel for target densities of 13 and 14 lb/gal, respectively, for the cold-laboratory tests. Batches of grout were prepared in triplicate at 1.34 and 1.80 g dry blend/g gel and mixed at 5-wt % surrogate sludge loading with each of the OHF and S21 surrogates. The densities of the freshly made grouts containing surrogate waste were measured and samples for penetration resistance and compressive strength cast. The samples were placed inside a humid environment at room temperature for curing. Free water was noted for only one batch after curing overnight and then only a modest amount (<0.5 vol %). The free water for this sample disappeared within 2 d; thus, little or no free water was generated from the completion of mixing and any surface wetness or collection of surface water disappeared within two days.

All of the samples appeared firm and had measurable penetration resistances overnight. All penetration resistance samples had been measured within 6 d from preparation. Fig. 3 illustrates a significant difference in strength development between the grouts prepared from the jetting slurry at 13 lb/gal compared to the one at 14 lb/gal, as expected. The denser grouts began exceeding the equipment upper limit of 8,000-psi (implying hard products of about 400-500 psi compressive strength) within two days. The “lighter” grouts only began approaching this limit after 6 d. Fig. 4 illustrates the 28-d unconfined compressive strength of these grouts with a 5-wt % loading of OHF or S21 simulant. A considerable overlap exists between the grouts at target densities of 13 and 14 lb/gal, but there was a significant increase in strength from about 1,000 to about 1,500 psi on average with increase in target density, with a slightly higher increase for 5 wt % of OHF simulant.

In summary, both simulants proved compatible with the in situ grout at these target densities, forming a thick, homogeneous paste that exhibited no free water and set into a strong monolithic solid within a few days of mixing.

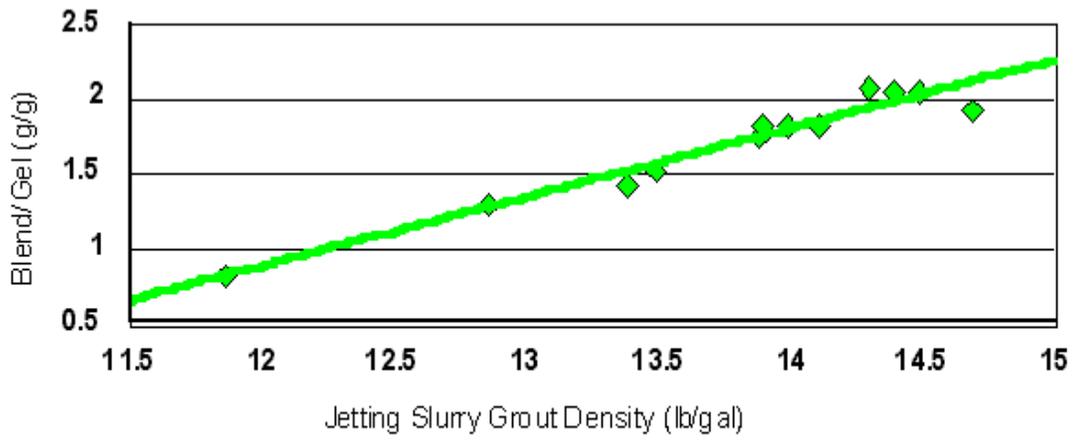


Fig. 2. Ratio blend/gel vs resulting density (bentonite/water × 100 = 6% gel).

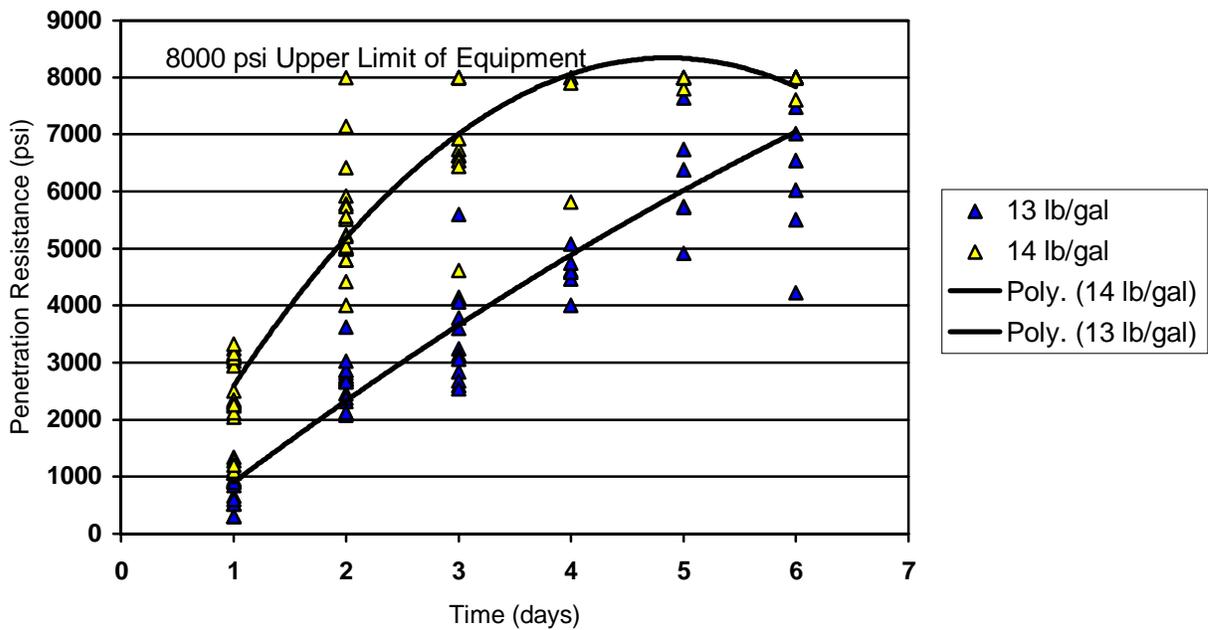


Fig. 3. Penetration resistance for FFCA surrogates (5-wt % sludge loading).

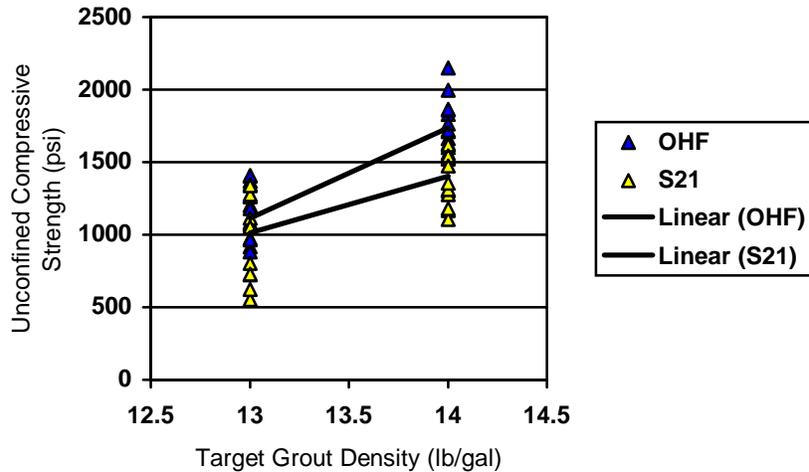


Fig. 4. 28-d unconfined compressive strengths for the FFCA grouts with 5-wt % simulant loading.

5.3 HOT TESTS (FY 1999)

After further evaluation of the cold-field demonstration in Odessa, Texas, further significant modifications were made in the in situ grout formulation to be used in the hot demonstration on the OHF tanks scheduled for FY 2000. Based on the difficulties of working with the 5–6% bentonite gel (this is a field term defined as the mass ratio of bentonite to water times 100: $\text{g bentonite/g water} \times 100 = \text{percentage of bentonite gel}$) at Odessa without a fluidizer, it was decided to target 3.75% bentonite gel and a lower target density for the OHF hot demonstration in FY 2000. With these targets, GES calculated the grout formulation for the MPI grouting of the OHF tanks, using a water-to-solids ratio (W:S) of 0.8 as a guide, and estimated a density of 12.7 lb/gal for the jetting-slurry grout to be injected into the OHF tanks. The grout formulation for a jetting-slurry grout density of 11 lb/gal (W:S of 1.5) was also calculated to provide the maximum leverage for field adjustment to maintain a mixable, pumpable grout. Basically, the hot demonstration will use the thickest grout that can be mixed and pumped in the field, but the field operation must have the option of field adjustment of the grout thickness and/or density. Since a grout of 11 lb/gal could be mixed and pumped for a 6% gel after hydrating overnight at Odessa, it is likely a higher density can be used for a 3.75% gel, even if hydrated overnight. It is expected that a 12.7-lb/gal grout will be used with same day hydration, but contingencies must be provided. These adjustments address the processability needs for the field operation and are not expected to significantly affect the stabilization potential of the baseline dry blend. For these reasons, the hot-laboratory testing used the grout formulations calculated by GES for the hot demonstration.

An actual sample of OHF sludge was diluted with water to match the estimated water content of the OHF tank heels (sludge and supernate), after sluicing, retrieval, and washing of the retrieval equipment. A sample of this diluted OHF sludge was then mixed with each of two jetting-slurry grouts—estimated densities of 11 and 12.7 lb/gal—to give a final monolith waste loading of 5 wt %. Table 11 lists the composition of each

monolith in terms of the injection water, bentonite, the baseline dry blend without bentonite, and waste. Table 11 also lists the waste composition in terms of the target sludge solids, sludge water, and supernate, based on the estimated OHF tank heels composition provided by the end user.

As expected, the 3.75% gel was more watery than the 6% gel used for the cold-laboratory testing and the grouts were significantly more fluid. However, no free water formed after mixing and the grouts became firm and solid overnight, indicating compatibility between the grout and actual OHF sludge. Samples of each were submitted to the radiochemical analytical laboratory for TCLP testing after curing for a few days.

Table 11. Composition of the final monolith and OHF waste for the hot laboratory testing

Grout composition	Wt % in final monolith	
	Estimated grout density	
	11 lb/gal	12.7 lb/gal
Injection water	57.00	42.22
Bentonite	2.14	1.58
Dry blend (w/o bentonite)	35.86	51.19
Waste	5.00	5.00
<u>Waste composition</u>		
OHF sludge solids	16.66	
Sludge water	38.90	
Supernate (water)	44.44	

5.4 RECOMMENDATIONS—GROUT FORMULATION STUDIES

Although the grout formulation described in this document is expected to perform satisfactorily for the hot demonstrations planned for Oak Ridge, refinements to meet changing expectations and field challenges are continuously under consideration. For example, further testing and refinement would be required to apply the technique to tank wastes at other sites such as Hanford. Additional work to improve the predictability and application of the formulation selection technique can be tailored to fit the available resources and requirements of the job (e.g., from few thousand gallons to the millions of gallons at Hanford or Savannah River). This work can range in scale from simple adjustments to make the grout formulation work for a given different situation to statistically designed experiments to generate predictive surface response models. Recommendations for further work include the following:

1. The formulation was altered slightly after the 1999 cold demonstration (bentonite-to-water gel ratio reduced from 6 to 3.75% and target density reduced from 14 to 12.7 lb/gal). Further laboratory testing is recommended to expand the database, especially in the area of the refined recipe.

2. The cold demonstration reports noted a need to increase the "set time" to 24 h. An indication of the variation of set time with composition is needed to allow the recipe to be adjusted according to changing needs and/or goals.
3. Measure the strength of grouted waste forms as a function of composition so the grout formulation can be designed to allow the monolith to be exhumable, if desired.
4. Measure the chemical properties (e.g., pH and Eh) of the monolith as a function of composition, so the stabilizing potential of the grout can be predicted.
5. Determine the minimum level of stabilizing agents to pass the regulatory leach tests (TCLP or ANS/ANSI-16.1) and obtain useful treatment of the waste.
6. Develop a laboratory method of measuring rheology accurately. Typical laboratory rotating viscometers are not accurate for complex bentonite-grout gel rheologies. Rheology should be measured as a function of composition and attempts made to relate laboratory rheology measurements to field processability (mixability and pumpability) data.
7. Measure solids segregation resistance as a function of grout composition.

The work described above will serve to (1) generalize the formulation and selection techniques utilized and (2) make the grout formulation more broadly applicable to other tank wastes, including the DOE tanks of interest to TFA. Ultimately, however, the selected formulation must be tested with the actual tank waste to be treated prior to implementation to identify possible incompatibilities and attempt to avoid surprises during field operations.

6. SUMMARY

This document presents a path forward for hot demonstration of GES's MPI technology at the OHF site and gives a brief status of the grout formulation development effort in support of tank-closure activities at Oak Ridge. The proposed closure plan includes the following primary elements and assumptions:

1. ORNL will continue the existing contractual arrangements with GES to perform the initial stages of the hot demonstration on the OHF tanks.
2. BJC will contract with GES for the actual on-site hot-demonstration effort.
3. The demonstration will include grouting of all five OHF tanks to show the expanded capabilities of the MPI process under a variety of horizontal waste-tank conditions and compare the performance of the MPI process with a low-pressure grout pour in one or more of the OHF tanks.
4. BJC will have oversight over the demonstration and will prepare the site and perform the final site closure after the MPI demonstration is completed.
5. GES will provide all tooling, tank interface equipment, grout supplies, mixing equipment, pumping equipment, piping, hoses, and operators to successfully perform the demonstration.
6. ORNL and/or BJC or BJC subcontract personnel will perform all tasks inside radiation control areas to minimize the number of GES personnel requiring Radiation Worker and Hazardous Waste Operations training.
7. BJC or BJC's subcontractors will provide a health and safety plan and waste management plan for the tank-closure action.
8. BJC will provide all utilities, services, and facilities (e.g., water, air, electrical, off-gas, restrooms, break room, air conditioning, personal protective equipment) required for the hot demonstration.

The grout formulation development efforts at Oak Ridge have resulted in the development of a grout with superior RCRA and radioactive contaminant retention properties. The formulation exhibits excellent physical suspension capabilities for the solids present in waste tanks. The Oak Ridge formulation inhibits and prevents the formation of bleed water and can also be tailored to meet and/or exceed strength requirements. Several variations on the formulation have been successfully used in laboratory simulant tests, hot laboratory tests with actual tank wastes, and two large-scale cold demonstrations of the MPI process.

7. REFERENCES

1. R. D. Spence, J. L. Kauschinger, and R. D. Hunt, *Grout Performance in Support of In Situ Grouting of the TH4 Tank Sludge*, ORNL/TM-13739, Oak Ridge National Laboratory, Oak Ridge, Tennessee, April 1999.
2. J. L. Kauschinger, R. D. Spence, B. E. Lewis, *In Situ Grouting Technology Demonstration and Field Specifications Overview for Hot Deployment of the Multi-Point-Injection™ System in Gunitite and Associated Tank TH-4*, ORNL/TM-13710, Oak Ridge National Laboratory, Oak Ridge, Tennessee, October 1998.
3. R. D. Spence and J. L. Kauschinger, *Grout Performance in Support of In Situ Stabilization/Solidification of the GAAT Tank Sludges*, ORNL/TM-13389, Oak Ridge National Laboratory, Oak Ridge, Tennessee, May 1997.
4. J. M. Keller, J. M. Giaquinto, and A. M. Meeks, *Characterization of the Old Hydrofracture Facility (OHF) Waste Tanks Located at ORNL*, ORNL/TM-13394, Oak Ridge National Laboratory, Oak Ridge, Tennessee, April 1997.
5. International Atomic Energy Agency, *Improved Cement Solidification of Low and Intermediate Level Radioactive Wastes*, Technical Reports Series No. 350, International Atomic Energy Agency, Vienna, 1993.
6. R. W. Nurse, "Slag Cements," pp. 37–68 in *The Chemistry of Cements Vol. 2*, ed., H. F. W. Taylor, Academic Press, New York, 1984.
7. J. Daube and R. Bakker, "Portland Blast Furnace Slag Cement: A Review," pp. 5–14 in *Blended Cements*, ed., Geoffrey Frohnsdorff, ASTM STP 897, ASTM, Philadelphia, 1986.
8. G. Frigione, "Manufacture and Characteristics of Portland Blast-Furnace Slag Cements," pp. 15–28 in *Blended Cements*, ed. Geoffrey Frohnsdorff, ASTM STP 897, ASTM, Philadelphia, 1986.
9. V. S. Dubovoy, S. H. Gebler, P. Klieger, and D. A. Whiting, "Effects of Ground Granulated Blast-Furnace Slags on Some Properties of Pastes, Mortars, and Concretes," pp. 29–48 in *Blended Cements*, ed. Geoffrey Frohnsdorff, ASTM STP 897, ASTM, Philadelphia, 1986.
10. R. H. Mills, "Chemical Shrinkage and Differential Sorptions in Mixtures of Portland Cement and Blast-Furnace Slag," pp. 49–61 in *Blended Cements*, ed. Geoffrey Frohnsdorff, ASTM STP 897, ASTM, Philadelphia, 1986.

11. A. M. Neville, *Properties of Concrete*, 3rd ed., Edward Arnold, Glasgow, 1970.
12. J. D. Palmer and D. L. G. Smith, *The Incorporation of Low- and Medium-Level Radioactive Wastes (Solids and Liquids) in Cement*, EUR-10561-EN, Commission of the European Communities, Luxembourg, 1986.
13. O. Brown, D. J. Lee, M. S. T. Price, and D. L. G. Smith, "Cement Based Processes for the Immobilization of Intermediate-Level Waste," pp. 145-152 in *Proc. Radioactive Waste Management*, British Nuclear Society, London, 1985.
14. F. P. Glasser and C. McCulloch, *Characterization of Radioactive Waste Forms, Progress Report for 1986*, EUR-11354, Commission of the European Communities, Luxembourg, 1986.
15. M. J. Angus and F. P. Glasser, "The Chemical Environment in Cement Matrixes," *Mater. Res. Soc. Symp. Proc.* **50**, 547-556, 1986.
16. R. D. Spence, W. D. Bostick, E. W. McDaniel, T. M. Gilliam, J. L. Shoemaker, O. K. Tallent, I. L. Morgan, B. S. Evans-Brown, K. E. Dodson, "Immobilization of Technetium in Blast Furnace Slag Grouts," pp. 1579-1596 in *Proc. 3rd International Conference on the Use of Fly Ash, Silica Fume, Slag and Natural Pozzolans in Concrete, Trondheim, Norway (June 19-24, 1989), Vol 2*, American Concrete Institute, Detroit, Michigan, 1989.
17. T. M. Gilliam, R. D. Spence, W. D. Bostick, and J. L. Shoemaker, "Solidification/Stabilization of Technetium in Cement-Based Grouts," *J. Hazardous Materials*, **24**, 189-197, 1990.
18. R. D. Spence, T. M. Gilliam, and A. Bleier, "Cementitious Stabilization of Chromium, Arsenic, and Selenium in a Cooling Tower Sludge," paper no. 95-RP130.03 in *Proc. 88th Annual Meeting of the Air & Waste Management Association in San Antonio, Texas, Vol. 15, Hazardous, Radioactive, and Mixed Waste, June 18-23, 1995*, Pittsburgh, Pennsylvania, 1995.
19. S. B. Clark and E. L. Wilhite, "Low-Level Liquid Waste Disposal at the Savannah River Site: A Large Scale Demonstration of Saltstone," pp. 603-609 in *Proc. Waste Management '91, Tucson, AZ, February 24-28, 1991*, WSRC-MS-90-210, DE92 009907, Westinghouse Savannah River Co., Savannah River Laboratory, Aiken, South Carolina, 1991.
20. C. A. Langton, M. D. Dukes, and R. V. Simmons, "Cement-Based Waste Forms for Disposal of Savannah River Plant Low-Level Radioactive Salt Waste," pp. 575-582 in *Proc. Materials Research Society, Scientific Basis for Nuclear Waste Management VII, 1983*, Elsevier Science, New York : North-Holland, 1984.

21. C. A. Langton, "Solidification of Low-Level Radioactive Waste at the Savannah River Site," pp 37–40 in *Proc. NIST and NRC Workshop Cement Solidification of Low Level Radioactive Waste*, NUREG/CP-0103, U.S. Nuclear Regulatory Commission, Washington, D.C., 1989.
22. C. A. Langton and P. B. Wong, "Properties of Slag Concrete for Low-Level Waste Containment," WSRC-MS-91-073, DE92 013194, Westinghouse Savannah River Company, Aiken, South Carolina, 1991.
23. D. W. Pepper, *Transport of Nitrate from a Large Cement-Based Wasteform*, DPST-85-963, DE88 004271, Savannah River Laboratory, Aiken, South Carolina, 1986.
24. C. H. Wolf, *Large-Scale Demonstration of Disposal of Decontaminated Salt as Saltstone Part I: Construction, Loading, and Capping of Lysimeters*, DPST-84-497, Savannah River Laboratory, Aiken, South Carolina, June 1984.
25. ASTM C 989-89, "Standard Specification for Ground Granulated Blast-Furnace Slag for Use in Concrete and Mortars," pp. 492–496 in *1991 Annual Book of ASTM Standards Section 4 Construction*, Vol 04.02, *Concrete and Aggregates*, American Association for Testing and Materials, Philadelphia, 1991.
26. ASTM C 204-91a, "Standard Specification for Fineness of Portland Cement by Air Permeability Apparatus," pp. 155–161 in *1991 Annual Book of ASTM Standards Section 4 Construction*, Vol. 04.01, *Cement; Lime; Gypsum*, American Association for Testing and Materials, Philadelphia, 1991.

A. APPENDIX: ROLES AND RESPONSIBILITIES

The performance of the hot demonstration of the use of the MPI process in closure of the OHF tanks is a cooperative effort between ORNL, BJC, BJC-subcontractors, and GES. The following sections provide a detailed description of the breakdown of roles and responsibilities of each major participant.

A.1 ORNL

A.1.1 Management

ORNL will manage the subcontract with GES for the design and fabrication of tooling, procurement of long-lead materials, cold-checkout of the systems needed for closure of the OHF tanks, and reporting to the EM-50 sponsor. ORNL will be responsible for funding the GES subcontract through an approved Technical Task Plan (TFP) by the DOE EM-50 TFA. ORNL will prepare and/or supervise the preparation of all reports and status updates associated with this effort that are required by the TTP. The actual on-site fieldwork for the hot demonstration of the MPI technology will be managed by BJC.

A.1.2 Environment, Safety, and Health (ES&H)

ORNL, through the ORNL project manager, will be responsible for overseeing the environmental compliance of the subcontractor(s) performing the MPI process during the preparatory efforts for which ORNL has contractual responsibility, including routine inspections of project activities. Oversight of on-site activities will be the responsibility of BJC. Any noncompliance directly attributable to on-site project activities will be the responsibility of BJC. The ORNL project manager will plan and conduct oversight of project activities to assess compliance with project and/or program requirements. Observations will be shared with the project team, and appropriate recommendation(s) to achieve or improve compliance will be provided to the project manager.

For on-site activities during the hot demonstration, the hazards, the requirements, and the responsibilities will be defined in a site health and safety plan, which will be developed by BJC and/or its subcontractor(s). This plan will define responsibilities for health and safety management and oversight and list basic hazards and controls, general requirements for health and safety training, personnel certification, site access, and personnel protection. ORNL will provide input to BJC during the development of this plan, as needed.

Health and safety requirements will be documented in the project health and safety plan and communicated to project participants through the contract documents and prejob training.

ORNL will provide input to the waste management plan, which is to be prepared by BJC and/or its subcontractor(s). ORNL and GES will review the plan and provide input, as necessary. Materials and wastes generated will be managed in compliance with project requirements. Management of excess materials and wastes that may be encountered during project activities will be addressed in the project waste management plan.

ORNL and GES will identify materials and equipment that may require demobilization, decontamination, or disposal at the end of the demonstration. BJC and/or its subcontractor(s) will have responsibility for decontamination, demobilization, and disposal of radiologically or chemically contaminated equipment that may be generated during the project that will be removed from the site. BJC and/or its subcontractor(s) will also have responsibility for disposal of noncontaminated excess materials (flush water, grout, industrial supplies, etc.). To the greatest extent possible, all excess grout and flush water will be placed in the OHF tanks. In general, all in-tank hardware; small, low-cost contaminated equipment, tools; and other items used in the implementation of the MPI process will be disposed of as contaminated waste and not decontaminated. ORNL will provide input for decontamination or disposal of anticipated contaminated and non-contaminated items as part of the waste management plan. Provision for unexpectedly contaminated equipment will also be made in the waste management plan.

A.1.3 Reporting

A.1.3.1 Monthly Progress Reports

Monthly reports will be prepared and submitted to DOE-ORO. The ORNL principal investigator responsible for this effort will compile and submit this report. Monthly reports will include the status of all milestones, summary of the work progress, and a brief financial summary. ORNL will prepare these reports based on observations of progress and submissions from participants. Financial information reports will only include those items specifically funded through ORNL and the TTP for the demonstration.

A.1.3.2 Weekly Reports

Highlight reports will be prepared and submitted to the TFA during the active portion of this project on a one- to two-week basis. The ORNL principal investigator responsible for this effort will compile and submit this report. The reports will describe the technical progress of the tank-closure effort and highlight success and/or problem areas.

A.1.3.3 Topical Report(s)

A topical report for external distribution will be prepared describing the observations and results from the tank-closure hot demonstrations. The ORNL principal investigator responsible for this effort will ensure the technical content and quality of all reports prepared for this project and funded under the TTP with ORNL.

A.1.4 Overview Camera

One or more overview cameras will be installed and used to monitor the progress of the grouting processes in one or more of the OHF tanks during the tank-closure operations.

A.1.4.1 Hardware

ORNL will specify and procure the necessary cameras and lights sufficient such as to view and videotape the progress of the tank closures. Small, inexpensive, and disposable cameras and lights will be used to minimize the potential for contamination. ORNL will provide all necessary monitors, cables, support poles, and recording equipment to view

the in situ grouting process. The camera and lighting hardware will be compatible with the grout injection-tool mounting flanges, which will be attached to OHF tank risers.

A.1.4.2 Personnel

ORNL personnel will procure and test all cameras and other monitoring equipment before installing the equipment in the OHF tanks. ORNL personnel will operate the camera and video-taping systems, as necessary, to capture the performance of the grout injection system in each tank.

A.1.5 Grout Formulation

ORNL personnel will specify the grout formulation to be used in the MPI process. The grout formulation will be similar to that used in past cold demonstrations of the MPI process.

A.1.6 Process Water

ORNL will provide the process water to be used for the MPI process. Process water will be obtained from a fire hydrant located near the OHF site. ORNL will provide an approved back-flow-preventer and necessary connections to the fire hydrant to supply water to storage tankers. It is estimated that up to ~100,000 gal of process water in batches of ~20,000 gal each will be required for the grouting operation.

A.1.7 Sampling and Analysis

Samples of the grouted waste will be taken from one or more of the OHF tanks and analyzed to assess the mixing effectiveness of the MPI process. ORNL will be responsible for obtaining these samples through contractual arrangements with Waste Management Federal, Inc., and in coordination with BJC. Samples will be taken after completion of the injection process and before the grout sets. ORNL will also be responsible for measuring the temperature of the monolithic grouted waste form inside the tanks using embedded thermocouples.

A.1.8 GES Contract Considerations

A.1.8.1 Sole-Source Procurement

GES has exclusive capability and holds several patents on MPI technology. This technology has been selected for hot demonstration for in situ tank waste immobilization in the OHF tanks at Oak Ridge. MPI technology has been successfully demonstrated in the immobilization of buried LLWs and in cold demonstration with simulated tank wastes in vertical and horizontal tanks. It is on the strength of the results from these tests that this technology has been selected for hot demonstration. The hot demonstration on the OHF tanks will prove the utility of MPI technology in a variety of horizontal tank-closure applications and allow comparison with low-pressure grout pours in tank-closure operations.

A.1.8.2 GES's Relationship with ORNL

GES will be a subcontractor to ORNL for the preparatory work associated with the closure of the OHF tanks. These efforts will include meetings, planning for the

demonstration, tool design and procurement, cold--checkout of the equipment, procurement of minor materials, delivery of equipment and supplies, and report preparation.

A.1.8.3 Patent Issues

GES holds all patents and patent rights to MPI technology. Ownership of any novel or patentable inventions or process developed as part of the OHF closure will be assigned to the inventor(s). Ownership of joint developments that are not included in the MPI patents will be shared between the inventors based on their documented contributions.

A.1.8.4 Proprietary Information Control

Any proprietary information received from GES during the course of this project will be handled according to the ORNL procedure, ORNL-TT-001, "Identification, Use, and Handling of Protected CRADA Information and Proprietary Information Acquired for or Related to Research and Development Activities at ORNL."

A.1.8.5 ORNL Interface with BJC

BJC will have oversight responsibility for all operations at the OHF site. ORNL will ensure that information necessary for equipment design and specifications are provided to GES and that the necessary interfaces with BJC are established. ORNL will ensure that GES is aware of all appropriate procedures and requirements for site access and performance of work at the site, as described and provided by BJC.

A.2 CONTRACTOR—GES

A.2.1 Personnel

A.2.1.1 MPI Deployment Coordinator

GES will provide an MPI deployment coordinator, who will be responsible for ensuring the proper deployment, installation, and operation of MPI systems in the OHF tanks. The coordinator will work under the supervision of BJC or its subcontractor. The deployment coordinator will also be responsible for providing the necessary work plans to BJC for implementation of the MPI process at the OHF site.

A.2.1.2 Grout-Pump Operators

GES will provide grout-pump operators who will work under the direct supervision of the MPI deployment coordinator. Operators will be trained and qualified for grout-pump operation according to GES policies. Pump operators will receive further specific training as required by applicable ES&H and security policies, as necessary.

A.2.1.3 Grout-Plant Operators

GES will provide grout-plant operators. These operators will work under the direct supervision of the MPI deployment coordinator. Operators will be trained and qualified for grout-plant operation according to GES policies. Grout-plant operators will receive further specific training as required by applicable ES&H and security policies, as necessary.

A.2.2 Site Interfaces

A.2.2.1 Tank-Riser Interface

GES will design and fabricate tank-riser interfaces based on information provided by ORNL and BJC. The interfaces will attach to the OHF tank risers and will include the necessary carrier casings for installation of the grout injection tooling as well as porting for an overview camera, lighting, sampling, and an off-gas connection. ORNL and BJC will approve GES' designs before fabrication and perform inspections and acceptance tests, as required, following fabrication and before installation. ORNL or BJC craft or BJC subcontract personnel will install the tank-riser interface hardware at the tank riser with oversight by GES to ensure proper installation.

A.2.2.2 Utilities, Services, and Facilities

Utilities, services, and facilities required for hot demonstration of the MPI process will be provided and maintained by BJC, as recommended by the MPI deployment coordinator. The utilities, services, and facilities are to include personal protective equipment, 120/240-V electrical service, and drinking water, break room, air conditioning, and restroom facilities. Process water for grout components will be provided by ORNL. GES will provide the necessary connections and interfaces to utilize BJC and ORNL supplied utilities, services, and facilities.

A.2.3 Pumping Equipment

GES will provide pumping equipment and connections necessary to transfer materials to the OHF tanks and between GES-provided support equipment.

A.2.3.1 Mobile Pumping Equipment

GES will provide the high-pressure mobile pumping equipment necessary for implementation of the MPI process. Location of the pumping equipment will be coordinated between GES, ORNL, and BJC. GES personnel will connect the pumping equipment to the MPI tooling with oversight by ORNL and BJC personnel.

A.2.3.2 Mobilization and Demobilization

ORNL and/or BJC will perform a radiological survey of the equipment before initiating work activities. A detailed health and safety plan will be prepared. The plan will summarize the identified chemical and radiological hazards present at the work site and identify control measures to reduce worker risk to as low as reasonable achievable (ALARA) levels. The plan will specify worker qualifications, personnel protective equipment, safety awareness, radiation work permits (if required), exposure control programs, decontamination procedures, and emergency coordination. The plan will be developed in compliance with 29 CFR Part 1910.120, and all workers will be required to show documentation of training and medical monitoring as required by this Occupational Safety and Health Act (OSHA) standard.

Equipment required for closure, including but not limited to high-pressure pumps, decontamination equipment, and waste, liquid, or grout transportation systems, will be mobilized or scheduled for mobilization.

Before startup of the project, GES will prepare a demobilization plan (listing all equipment items to be demobilized in place) for review and approval by ORNL and BJC. At the completion of the project, equipment will be demobilized according to this plan.

A.2.4 Grout-Injection Technology

A.2.4.1 Injection-Tool Fabrication

GES will be responsible for designing and fabricating grout-injection tools and hoses. ORNL will perform an inspection of the tooling and hoses before their installation within the tanks. GES will provide documentation of all pressure and acceptance test results before the equipment is installed.

A.2.4.2 Injection-Tool Installation

GES will oversee the installation of the injection tools at the site. GES will prepare an installation plan for approval by BJC prior to the start of installation. ORNL and/or BJC personnel will perform the installation.

A.2.4.3 Injection-Tool In-Tank Disposal

Before installation, GES will prepare a plan for in-tank disposal of the injection tools for ORNL and BJC approval. ORNL and/or BJC personnel or BJC subcontractors will perform in-tank disposal of the grout injection tools and hoses with oversight by GES.

A.2.5 Grout

A.2.5.1 Dry-Blend Materials

Dry-blend materials will be procured by GES, as necessary, to meet the requirements of the specified grout. The grout will be substantially equivalent or superior to a low strength Harrison Mix 80, consisting of 600-lb of Type F fly ash; 50-lb of portland Type II cement; and 2,400-lb of concrete sand (manufactured or river run).

GES will determine the quantity of dry-blend materials to be maintained on site. BJC and ORNL will determine storage locations and materials-handling procedures according to applicable BJC ES&H requirements and GES needs. GES will provide the necessary dry-blend storage facilities.

A.2.5.2 Blending Facilities

GES will provide the necessary blending facilities and services for mixing the dry blend ingredients. Storage and setdown space for the dry-blend materials will be determined jointly by ORNL, BJC, and GES.

A.2.5.3 Mobilization and Demobilization

Before the initiation of work activities, BJC or its subcontractors will survey the units. A detailed health and safety plan will be prepared. The plan will summarize the identified chemical and/or radiological hazards present at the work site and identify control measures to reduce worker risk to ALARA levels. The plan will specify worker qualifications, personnel protective equipment, safety awareness, radiation work permits (if required), exposure control programs, decontamination procedures, and emergency coordination requirements. The plan will be developed in compliance with 29 CFR Part 1910.120, and all workers will be required to show documentation of training and medical monitoring as required by this OSHA standard.

Equipment required for closure, including, but not limited to, high-pressure pumps, decontamination equipment, and waste, liquid, or grout transportation systems will be mobilized or scheduled for mobilization by GES.

Before completion of the project, GES will prepare for review and approval by BJC a demobilization plan listing all equipment items to be demobilized in place. At the completion of the project, equipment will be demobilized according to this plan.

A.2.6 Training Requirements

A.2.6.1 Site Access

All GES personnel assigned to the project who require site access will attend Site Access training as required by ORNL and BJC policies and procedures (i.e. General Employee Training). GES will be responsible for ensuring that this training has been successfully completed, that it is current, and that records have been provided to ORNL and BJC prior to starting on-site work.

A.2.6.2 Radiation Worker

All GES personnel required to enter a radiation control area will attend Radiation Worker II training as required by ORNL and BJC policies and procedures for workers at radioactively contaminated sites. GES will be responsible for ensuring that this training has been successfully completed, that it is current, and that records have been provided to ORNL and BJC prior to starting on-site work.

A.2.6.3 Hazardous Waste Operations

All GES personnel assigned to the project and performing work within the radiological area at the OHF site will attend 40 h of Hazardous Waste Operations (HAZWOPER) training as required by ORNL and BJC policies and procedures for hazardous waste operations site workers. GES will be responsible for ensuring that this training has been successfully completed, is current, and records have been provided to ORNL and BJC prior to starting on-site work.

A.2.6.4 Melton Valley Site Access

All GES personnel assigned to the project that require access to the OHF site within Waste Area Grouping 5 will attend training as required by BJC policies and procedures (i.e. Melton Valley Site Access Training).

A.2.6.5 Other

All GES personnel assigned to the project that require access to the site will attend other training as required by ORNL and BJC policies and procedures. This training will be specified in the contractual agreements between GES and ORNL and between GES and BJC.

A.2.7 Reporting

A.2.7.1 Status (Weekly/Monthly)

The GES MPI Deployment Coordinator will provide weekly and monthly status reports to ORNL. The reports will describe the technical progress of the tank-closure effort and highlight success and/or problem areas. Monthly progress reports will be prepared and will include the status of all milestones and summary of the work progress.

A.2.7.2 Performance

GES will prepare a performance report at the conclusion of the hot demonstration. The performance report will be submitted to ORNL for review by both ORNL and BJC and will include a description of the setup, operating conditions, quantities of grout delivered, equipment used, tooling descriptions, mobilization/demobilization activities, waste generation, disposal actions, and lessons learned. The report will be edited and issued by ORNL as an ORNL-TM with unrestricted distribution.

A.3 TANK OWNER—BJC

A.3.1 Oversight

BJC will have oversight responsibility for all operations at the OHF site. ORNL and GES personnel will follow all appropriate procedures and requirements for site access and performance of work at the site.

A.3.1.1 ES&H

Primary responsibility for ES&H resides with BJC. BJC will ensure that GES and ORNL personnel associated with the MPI hot demonstration adhere to the health and safety plan prepared by BJC and/or its subcontractor(s).

A.3.1.2 Radiation Control

BJC and/or its subcontractor(s) will characterize project areas and activities to assess radiation control requirements and identify the applicable radiological control requirements. Work plans and radiation work permits will be prepared and used during radiological work as necessary and will be reviewed and approved by BJC. GES will provide the necessary work plans for implementation of the MPI process.

A.3.1.3 Readiness Assessment

Prior to installation of equipment at the site, BJC will conduct a readiness assessment with input from ORNL and GES. The Operational Readiness Assessment should be used to ensure that hardware designs, operational procedures, and management controls are complete, accurate, and appropriate to the task. The review should determine whether all hardware and management systems are ready to proceed with the MPI demonstration.

A.3.2 Site Preparation

A.3.2.1 Tank Riser Access

BJC and/or its subcontractor(s) will provide access to the OHF tanks as necessary to perform the MPI Demonstration. Excess material, equipment, and other obstructions will be moved and/or removed from the site to provide adequate access to the OHF tanks.

A.3.2.2 Facilities

BJC and/or its subcontractor(s) will provide the facilities necessary to support project operations. The ORNL project manager will provide a list of requirements to BJC. The necessary facilities are anticipated to include drinking water, break room, air conditioning, and restrooms.

A.3.3 Logistics

A.3.3.1 Traffic Flow

BJC and/or its subcontractor(s) will identify traffic flow problems and provide a traffic flow plan in consultation with the ORNL project manager and the GES Deployment Coordinator. BJC will ensure compliance with the traffic flow plan.

A.3.3.2 Weight Limitations for Access

BJC will identify weight limitations for access to the site and will ensure compliance with weight limitations within the site.

A.3.3.3 Routes and Maps

BJC will identify routes for material delivery and provide maps to ORNL and GES and will ensure compliance with the route plan.

A.3.4 Site Access

A.3.4.1 Clearance and Badges

BJC will be responsible for oversight of site access, including clearance and badging. ORNL will be directly responsible for clearing and badging GES personnel.

A.3.4.2 Training

BJC will provide oversight of the required training for site access and operations. ORNL and BJC will determine the training requirements for site personnel (ORNL and GES). ORNL will be responsible for documenting training for ORNL and GES will be responsible for documentation of the training of all GES personnel working at the OHF site.

A.3.5 Utilities and Services

A.3.5.1 Water

BJC will provide potable water to the site as required by GES and ORNL. Process water for use in the grouting operations will be obtained from a fire hydrant located in the vicinity of the OHF site and will be provided by ORNL. ORNL will provide an approved back-flow preventer and be responsible for the connection to the fire hydrant. It is estimated that up to ~80,000 gal of process water in batches of ~20,000 gal each will be required for the grouting operation.

A.3.5.2 Electrical

BJC and/or its subcontractor(s) will provide electrical utilities to the site as required by GES and ORNL. The electrical service requirements will vary depending on the type of equipment selected for use. It is anticipated that 120 and 240-V electrical services will be required.

A.3.5.3 Air Handling System

BJC and/or its subcontractor(s) will provide the necessary air handling system (i.e. High Efficiency Particulate Air filters, hoses, and connections as necessary to couple to GES provided tank interface cover) for use by GES during the hot demonstration. GES and ORNL will work with BJC to provide the information necessary to properly size the air handling system and connections.

A.3.5.4 Air

BJC and/or its subcontractor(s) will provide pressurized air to the site as required by GES and ORNL. It is anticipated that air pressure in the range of 90 to 100 psig and intermittent airflow of 10 to 20 scfm will be required for the tank-closure operation.

A.3.5.5 Waste Disposal

Materials and wastes generated will be managed in compliance with project requirements. Management of excess materials and wastes that may be encountered during project activities will be addressed in the project Waste Management Plan, which will be prepared by BJC personnel or their subcontractors.

A.3.5.5.1 Personal protective equipment

BJC and/or their subcontractor(s) will provide all Personal Protective Equipment necessary for use in hot demonstration of the MPI process at the OHF site.

A.3.5.5.2 Excess grout

Flushing the grout pumping and mixing equipment at the conclusion of the MPI process or at the end of each day of operation will result in the production of excess grout. BJC will have oversight of the utilization/disposal of excess grout according to the project Waste Management Plan. ORNL and GES will assist BJC in identification of methods for utilizing and/or disposing of excess grout. Where possible the excess grout will be added to the OHF tanks.

A.3.5.5.3 Decontamination supplies

Decontamination supplies will be managed in compliance with project requirements. Management of excess and/or used decontamination supplies will be addressed in the project's waste management plan.

A.3.5.5.4 Contaminated equipment and materials

Contaminated equipment and materials will be handled in accordance to the project's waste management plan.

A.3.5.6 Maintenance

BJC and/or its subcontractor(s) will be responsible for maintenance of site access routes, utilities, services, facilities, and project infrastructure. ORNL will be responsible for the maintenance of the video inspection equipment. GES will be responsible for maintenance of all MPI equipment provided under the contracts with ORNL and BJC.

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