

ALTERNATIVE FIELD METHODS TO TREAT MERCURY IN SOIL TOPICAL REPORT/FINAL TECHNICAL REPORT

IT CORPORATION
With NUCLEAR FUEL SERVICES

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**IT CORPORATION
TOPICAL REPORT/FINAL TECHNICAL REPORT**

For:

**National Energy Technology Laboratory (NETL) and
Department of Energy (DOE)**



Contract No. DE-AC26-01NT41345
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ABSTRACT

U.S. Department of Energy (DOE) used large quantities of mercury in the uranium separating process from the 1950s until the late 1980s in support of national defense. Some of this mercury, as well as other hazardous metals and radionuclides, found its way into, and under, several buildings, soil and subsurface soils and into some of the surface waters. Several of these areas may pose potential health or environmental risks and must be dealt with under current environmental regulations.

DOE's National Energy Technology Laboratory (NETL) awarded a contract "Alternative Field Methods to Treat Mercury in Soil" to IT Group, Knoxville TN (IT) and its subcontractor NFS, Erwin, TN to identify remedial methods to clean up mercury-contaminated high-clay content soils using proven treatment chemistries. The sites of interest were the Y-12 National Security Complex located in Oak Ridge, Tennessee, the David Witherspoon properties located in Knoxville, Tennessee, and at other similarly contaminated sites. The primary laboratory-scale contract objectives were (1) to safely retrieve and test samples of contaminated soil in an approved laboratory and (2) to determine an acceptable treatment method to ensure that the mercury does not leach from the soil above regulatory levels. The leaching requirements were to meet the TC (0.2 mg/l) and UTS (0.025 mg/l) TCLP criteria. In-situ treatments were preferred to control potential mercury vapors emissions and liquid mercury spills associated with ex-situ treatments. All laboratory work was conducted in IT's and NFS laboratories.

Mercury contaminated nonradioactive soil from under the Alpha 2 building in the Y-12 complex was used. This soils contained insufficient levels of leachable mercury and resulted in TCLP mercury concentrations that were similar to the applicable LDR limits. The soil was spiked at multiple levels with metallic (up to 6000 mg/l) and soluble mercury compounds (up to 500 mg/kg) to simulate expected ranges of mercury contamination and to increase the TCLP mercury values.

IT/NFS investigated ambient temperature amalgamation/stabilization/fixation of mercury-contaminated soils to meet these objectives. Treatment ranged in size from a few ounces to 10 pounds. The treatability study philosophy was to develop working envelopes of formulations where reasonable minimum and maximum amounts of each reagent that would successfully treat the contaminated soil were determined. The dosages investigated were based on ratios of stoichiometric reactions and applications of standard sets of formulations. The approach purposely identified formulations that failed short or longer cure-time performance criteria to define the limits of the envelope. Reagent envelopes successfully met the project requirements one day after treatment and after greater than 30-day cures. The use of multiple levels of spikes allowed the establishment of reagent dosages that were successful across a broad range of mercury values, e.g., 50 to 6000 mg/kg mercury. The treatment products were damp to slightly wet material. Enough drying reagent, e.g., Portland cement or lime by-product, were added to some formulations to control the leachability of uranium and other hazardous metals and to ensure the product passed the paint filter test.

Cost analyzes and conceptual designs for four alternatives for full-scale treatments were prepared. The alternatives included two in-situ treatments and two ex-situ treatments. The cost estimates were based on the results from the bench-scale study. All four alternatives treatment costs were well below the baseline costs.

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ACGIH	American Conference of Governmental Industrial Hygienists
ALARA	As Low As Reasonably Achievable
APC	Air Pollution Control
ATG	Allied Technology Group
ATL	Applied Technology Laboratories
BJC	Bechtel Jacob Company
BNL	Brookhaven National Laboratory
cc	cubic centimeters
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
cf	cubic foot
CFR	Code of Federal Regulation
CHP	Certified Health Physicist
CLP	Contract Laboratory Procedure
CO	Contracting Officer
CVAA	Cold Vapor Atomic Adsorption
cy	cubic yards
D&D	Decontamination and Decommissioning
DeHg [®]	(Pronounced DEE-merc)
DoD	Department of Defense
DOE	Department of Energy
DOT	Department of Transportation
DQO	Data Quality Objective
EM	Environmental Management
EMWMF	Environmental Management Waste Management Facility
EOU	Envirocare of Utah
EPA	US Environmental Protection Agency
ES&H	Environmental Safety and Health
ESTCP	Environmental Security Technology Certification Program
g	gram
Hg	Mercury
Hg ⁰	Metallic or elemental or liquid mercury metal
Hg ²⁺	Mercury in the plus two valence state
HgO	Mercury (II) oxide
HgCl ₂	Mercury (II) chloride
H&S	Health and Safety
HTRW	Hazardous, toxic and radioactive wastes
ICP	Inductively Coupled Plasma
ID:	Idaho DOE Site
INEEL	Idaho National Environmental and Engineering Laboratory
IT	IT Corporation
ITSR	Integrated Technology Summary Report
kg	kilogram
Ksp	Solubility products Constant
L	liter
LA:	Los Alamos DOE Site
LANL	Los Alamos National Laboratory
LDR	Land Disposal Restriction

LTTD	Low Temperature Thermal Desorption
m ³	cubic meters
mg/kg	milligram per kilogram
mg/l	milligram per liter
MARSSIM	Multi Agency Radiation Survey and Site Investigation Manual
MLLW	Mixed Low Level Waste
MMES	Martin Marietta Energy Systems
MS	Matrix Spike
MTCLP	Modified toxicity characteristic leaching procedure
MTRU	Mixed Transuranic Waste
MVA	Mercury Vapor Analyzer
MWFA	Mixed Waste Focus Area
MWIR	Mixed Waste Inventory Report
NEPA	National Environmental Policy Act
NETL	National Energy Technology Laboratory
NFS	Nuclear Fuel Services
ng/m ³	nanogram per cubic meter
NIOSH	National Institute for Occupational Safety and Health
NPDES	National Pollution Discharge Emission Standard
NTS	Nevada Test Site
ORNL	Oak Ridge National Laboratory
ORR	Oak Ridge Reservation
OSHA	Occupational Safety and Health Administration
OSW	EPA-Office of Solid Waste
PCB	polychlorinated biphenyls
pcf	per cubic foot
pCi/g	pico curies per gram
PEL	Permissible Exposure Limit
PO	Portsmouth DOE Site,
PPE	personal protective equipment
ppm	Parts per million
PRDA	Program Research and Development Announcement
psi	pounds per square inch
QA/QC	Quality Assurance/Quality Control
RCRA	Resource Conservation and Recovery Act
REL	Recommended Exposure Limit
RFP	Request for Proposal
RF	Rocky Flat DOE Site
RL	Richland DOE Site
RMERC	Retorting of mercury contaminated materials
SAP	Sampling and Analysis Plan
SCFA	Subsurface Contaminant Focus Area
SOG	Standard Operating Guideline
SOW	Statement of Work
SRS	Savannah River Site
SW	Solid Waste
TAT	Turn Around Time

TC	Toxicity Characteristic
TCLP	Toxicity Characteristic Leaching Procedure
TCLP Hg	The TCLP for mercury
TDL	Technology Development Laboratory
TLV-TWA	Threshold Limit Values-Time Weighted Average
TSDF	Treatment, Storage and Disposal Facility
UEFPC	Upper East Fork Poplar Creek
µg/l	micrograms per liter
UTS	Universal Treatment Standard
UHC	Underlying Hazardous Constituent
USACE	United States Army Corps of Engineers
UST	Underground Storage Tank
UTS	Universal Treatment Standard
WAC	Waste Acceptance Criteria
WBS	Work Breakdown Structure
Wc	Units of Geotechnical Water Content using the water/dry basis
w/w	Weight over weight ratio
WWT	Water Waste Treatment

The U.S. Department of Energy (DOE), National Energy Technology Laboratory (NETL), awarded IT Corporation (now Shaw Environmental, Inc.) a contract for “Alternative Field Methods to Treat Mercury in Soil” (DE-RA26-01NT41030). The contract was for identifying and demonstrating remedial methods to clean up mercury-contaminated high-clay content soils at the Oak Ridge, Tennessee Y-12 National Security Complex, at the off-site David Witherspoon properties, and at other similarly contaminated sites. The treatment was to use proven treatment chemistries. Soil from the Y12 Complex Alpha 2 building was investigated in this study. The received soil TCLP mercury (TCLP Hg) values were below the cleanup goal (approximately 20 percent of the performance criteria). The soils were therefore spiked with liquid mercury and mercury (II) oxide to increase their TCLP leachability.

IT and its subcontractor, NFS, successfully identified treatment chemistries and processes to meet the performance criteria on the spiked soil. The TCLP Hg values from the selected formulas were $1/10^{\text{th}}$ or smaller than the performance criteria. Both companies used their own proprietary chemistry. IT developed two different successful treatments that met and well exceeded the performance criteria and did not show any aging affect. NFS applied its DeHg® process to also achieve the desired results. During the scale-up proving tests using the 5000 mg/kg liquid Hg spiked soil, small amounts of liquid mercury were smeared on the inner surfaces of the DeHg® reactor.

The NFS scale-up batch tests provides insight into a potential full-scale issue. The observation confirms that separation of larger drops of liquid Hg from soil is a potential design issue for the full-scale. The efficiency at which any mixing process will incorporate liquid mercury into the stabilized material depends on the size of the Hg droplets and the intensity (shear) in the mixing process. The NFS reactor is a low-shear, low rpm mixer. The reactors/mixers proposed for the optional Phase 2 study are a high shear pugmill or properly operated in-situ auger. These will apply more shear to breakup the liquid mercury and react it with the treatment reagents. Small amounts of liquid mercury will still potentially separate from the soil during operations. In the IT/NFS proposed process, this mercury will be collected and treated. The collected mercury may be treated using NFS or other’s technology. Using the NFS technology, the mercury treatment should cost less than \$300,000 (\$100,000 extra labor for collection and maintenance and \$200,000 for treatment.)

At the request of NETL and Bechtel Jacobs personnel, engineering cost estimates for four cases were reported. The cases are in-situ (IT and NFS chemistries) and ex-situ (IT and NFS

chemistries). These prices were compared with the baseline treatment technology (Low Temperature Thermal Desorption) cost estimate for treating 50,000 cy. The most expensive of the IT/NFS processes was roughly 30 percent of the baseline treatment technology.

It is our understanding that much of the area to be treated is filled with backfill, debris, pipes, or other underground utilities. IT/NFS thus suggest an ex-situ process since it has a wider applicability. In open areas, e.g., at the off-site properties, in-situ treatment should be considered. In addition, since the NFS chemistry is a more mature chemistry and has been successfully applied in more demonstration programs, the NFS chemistry is recommended for implementation at this time.

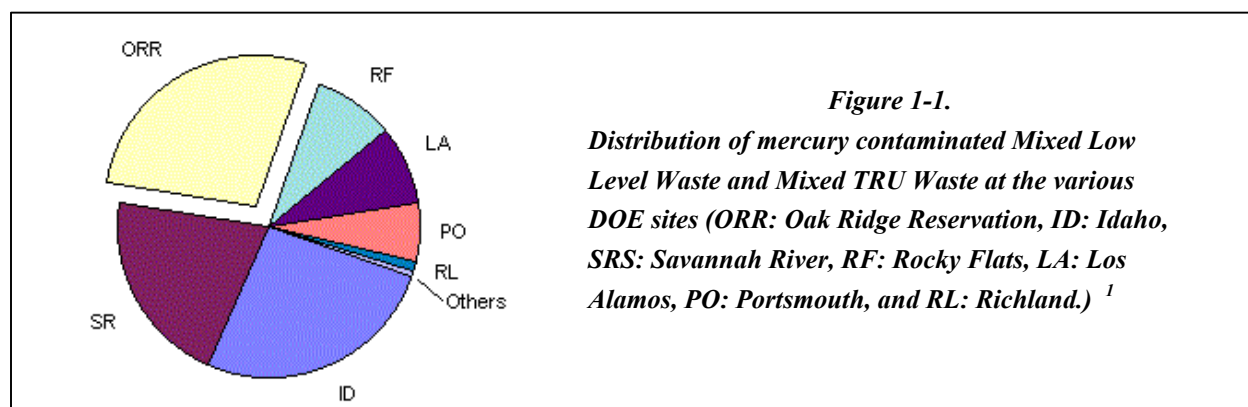
The key results of the project are as follows:

- The in-situ and ex-situ treatment costs are well below the baseline cost. The selected processes and their cost estimates are based on the results from the bench-scale study.
- IT and NFS both succeeded in demonstrating processes for stabilization of mercury-contaminated soil to meet the project performance criteria (TCLP 0.2 mg/l mercury).
- Treatment formulations were also determined which treated the soil to below the UTS-UHC treatment standard (0.025 mg/l in the TCLP.)
- The selected treatment formulations do not show any deleterious aging effect over a 30-day cure period.
- The volume increase was less than 27.5 percent for both the IT and NFS processes. This volume increase includes the addition of enough drying reagent for the product to meet the EMWMF WAC paint filter test requirement. The volume increase was larger than previously observed when using the DeHg® process. The larger volume increase was because of the soils' high clay content.
- The initial product is a damp to slightly wet material. Enough drying reagent, e.g., Portland cement or lime by-product, will be added to ensure the product meets the paint filter test.
- Approximately the same amount of drying agent will be used to dry the treated soil for the IT in-situ and ex-situ processes.

- Less water will need to be added in the NFS ex-situ process than in the in-situ process. The ex-situ product will require less drying reagent to meet the paint filter test requirement.
- The only secondary waste generated from the process will be personal protective equipment (PPE), general equipment cleaning solids, decontamination liquids from the last treatment, and potentially a small amount of collected metallic mercury. The collected metallic mercury will be treated to meet the performance criteria.

1.1 PROGRAM OVERVIEW

The Department of Energy (DOE) currently has mercury (Hg) contaminated materials and soils at the various sites. **Figure 1-1** (from ‘<http://www.ct.ornl.gov/stcg.hg/>’) shows the estimated distribution of mercury contaminated waste at the various DOE sites. Oak Ridge and Idaho sites have the largest deposits of contaminated materials. The majorities of these contaminated materials are soils, sludges, debris, and waste waters. ^{1, 2, 3, 18}



This project concerns treatment of mercury contaminated soils. The technology is applicable to many DOE sites, in-particular, the Y-12 National Security Complex in Oak Ridge Tennessee and Idaho National Engineering and Environmental Laboratory (INEEL). These sites have the majority of the soils and sediments contaminated with mercury. The soils may also be contaminated with other hazardous metals and radionuclides.

At the Y12 plant, the baseline treatment method for mercury contaminated soil is low temperature thermal desorption (LTTD), followed by on-site landfill disposal. LTTD is relatively expensive (estimated cost of treatment which exclude disposal cost for the collect mercury is greater than \$740/per cubic yard [cy] at Y-12), does not treat any of the metal or radionuclides. DOE is seeking a less costly alternative to the baseline technology.

As described in the solicitation (DE-RA-01NT41030), this project initially focused on evaluating cost-effective in-situ alternatives to stabilize or remove the mercury (Hg) contamination from high-clay content soil. It was believed that ex-situ treatment of soil contaminated with significant quantities of free-liquid mercury might pose challenges during excavation and handling. Such challenges may include controlling potential mercury vapors and containing liquid mercury beads. As described below, the focus of this project was expanded to include consideration of ex-situ treatment after award of the contract to International Technology

Corporation (IT). After award of the contract, IT became part of Shaw E&I. The company will be denoted as “IT” for the rest of the document since the original contract was awarded to IT.

This report details IT, Knoxville, TN and its subcontractor Nuclear Fuels Services (NFS) study to investigate alternative mercury treatment technology. The IT/NFS team demonstrated two processes for the amalgamation/stabilization/fixation of mercury and potentially Resource Conservation Recovery Act (RCRA) and radionuclide-contaminated soils. This project was to identify and demonstrate remedial methods to clean up mercury-contaminated soil using established treatment chemistries on soil from the Oak Ridge Reservation, Y-12 National Security Complex, the off-site David Witherspoon properties, and/or other similarly contaminated sites. Soil from the basement of Y-12 Plant Alpha 2 Building at the Oak Ridge Reservation was received at IT and NFS on December 20, 2001. Soils from the other locations were not investigated. The soil had background levels of radioactivity and had all eight RCRA metals well below the Toxicity Characteristic (TC) criteria.

This project addresses the new DOE Environmental Management³¹ Thrust 2 “Alternative Approaches to Current High Risk/High Cost Baselines”. Successful completion of this project will provide a step-change in DOE’s treatment ability.

The program consisted of two phases. The first phase was bench-scale and is the subject of this report. The second optional phase is a field demonstration. The bench-scale testing determined mix designs and treatment conditions using the Y-12 soil. The IT/NFS Phase 1 project duration was nearly 10 months and tested soil samples ranging in size from 45 grams to 21 kilograms (kg) per test. If this treatment method is selected for further use by DOE, work will continue in the field demonstration to treat many tons of contaminated soil. The estimated area to treat in IT’s proposed Phase 2 field demonstration is an area of 40 ft x 60 ft by 10 ft depth (889 cy, 1245 tons soil @ 1.4 ton/cy).

IT/NFS stabilized soil in this project to meet the RCRA Alternative Treatment Standard for soil leachability. IT and NFS have independently developed successful technologies to treat elemental mercury and its compounds. Both companies’ chemistries were investigated in this project. The chemistries have similarities but are different. They are based on amalgamation and fixation chemistries. The NFS chemistry is a more mature chemistry that has been demonstrated on widely different types of materials and is licensed for use at Envirocare to treat radiologically and mercury contaminated materials. The TCLP performance for the NFS treated

products has consistently improved over their three-year aging study on materials from previous projects.

The selected stabilization processes are very flexible and reagent mix and loading can be adjusted to meet a wide range of leachability requirements from the Universal Treatment Standard (UTS) criterion (0.025 milligrams per liter [mg/l]) to the Toxicity Characteristic (TC) level (0.20 mg/l) for mercury. This project was executed at the team laboratories in Knoxville TN (IT) and Erwin TN (NFS).

The team's chemistries are not only applicable to in-situ treatment of soil and sediments but are also applicable to ex-situ soil treatment. The companies' chemistries and process may be applied to most mercury-contaminated wastes within the DOE complex. Depending on the stream to be treated, the mixing operation can be adapted from an in-situ operation to in-drum stabilization or mixing in a pugmill. Since batch treatment processes were researched in this project, the bench-scale results are readily scaleable to larger projects.

This report summarizes the findings of IT's and NFS's bench-scale demonstration of their amalgamation/stabilization/fixation processes. Experiments were conducted at each company's facility using their own proprietary chemistries. The treatment batch sizes varied between (0.045 to 21 kg of soil. Successful formulations were identified by both companies.

1.2 DOE, STATE OF TN, BJC, AND COMMUNITY ACCEPTANCE

IT and NFS worked closely with DOE, NETL, and BJC personnel to develop the Statement of Work, Work Plan, and modifications of the program to best meet DOE's needs. The Workplan was reviewed and approved the NETL, DOE, BJC, and TN EPA personnel. At the Kickoff meeting, people from the same set of organizations provided input that was incorporated into the program. In addition, the state of TN requested additional information on the chemicals used in the treatment process. The state was concerned about possible hazards of materials being added to the ground during treatment and/or later stored in the on-site disposal cell. IT/NFS agreed that if the project went to field demonstration, the identity of the treatment chemicals would be given to the state under a business confidentiality agreement. There were monthly meetings with NETL and BJC personnel to ensure that NETL and the technology enduser were consistently updated and the program approach was acceptable.

The NFS chemistry (DeHg®) is a mature chemistry that has been accepted by multiple state agencies and by a mixed waste TSDF. NFS has demonstrated the DeHg® technology for three levels of regulatory agencies for acceptance. The following describes the interface between NFS and the pertinent regulatory agencies regarding the application of DeHg®.

1. State of Tennessee: DeHg® has been permitted in the State of Tennessee to treat a variety of mercury mixed wastes, including bulk radioactively contaminated mercury. Through this permitting and operating experience, NFS demonstrated that DeHg®:
 - Is protective of human health and the environment through both engineering and administrative controls,
 - Reduces the toxicity and mobility of material through treatment,
 - Can be implemented, with potential for deployment at other locations,
 - Has been accepted by the community as indicated in public hearings during the permitting process.

We envision that NFS's prior experience with the State of Tennessee in the area of mercury mixed waste treatment and disposition will facilitate discussions if the Y-12 soils project moves forward to Phase 3 and beyond.

2. EPA-Office of Solid Waste (OSW): NFS has made routine presentations since 1995 to the EPA-OSW (Crystal City) presenting new DeHg® application data as well as discussing continuing improvements to the DeHg® process. These processes have included both amalgamation of radioactively contaminated elemental mercury and amalgamation/stabilization of mercury-contaminated wastes. NFS has also participated in OSW programs to provide stabilized mercury waste forms for evaluation by EPA.
3. State of Utah: NFS has presented DeHg® to the State of Utah regulators, and as a result of this work, has qualified the process for application at the Envirocare of Utah (EOU) site. EOU has deployed DeHg® for treatment and disposal of 200 tons of mercury mixed waste, and plans to use it for treatment of other DOE mercury mixed waste streams during 2002 and beyond. EOU has also disposed of radioactively contaminated bulk elemental mercury that has been amalgamated by the DeHg® process.

1.3 REGULATORY STATUS AND PROJECT OBJECTIVES

1.3.1 RCRA Status of Received Soil

This study dealt with fixation of nonradioactive soil that was contaminated with mercury. The expected maximum mercury concentration was 0.7 percent (weight over weight ratio [w/w]). (The soil received had about 50 milligram per kilogram [mg/kg] total mercury.) Although the site soils may contain high levels of mercury, they have not been excavated for disposal and as such are not currently regulated under RCRA. IT/NFS thus considers the received soils as non-RCRA regulated samples. In addition, the in-house IT TCLP results showed that all RCRA metals were well below the TC criteria.

The Oak Ridge soil from the full-scale process will be sent to the on-site Environmental Management Waste Management Facility (EMWMF) for final disposal. As indicated in the solicitation, the soil once excavated is destined for the EMWMF and it must meet the RCRA Land Band criteria and the EMWMF Waste Acceptance Criteria (WAC). IT/NFS does not have a copy of the EMWMF WAC and therefore cannot compare their products to the WAC. Both companies assumed that the material must pass the paint filter test as part of the EMWMF WAC. All materials from the selected formulations met this criterion.

1.3.2 Goals and Objectives

The IT/NFS goals for these studies were to complete bench- and field demonstration-scale treatability tests which:

- Meet all the program objectives defined in the solicitation and Statement of Work,
- Maintain compliance with applicable health and safety criteria for worker and public exposure,
- Collect data needed to design a scaleable process to treat mercury-contaminated soils and sediments, and
- Demonstrate improved field technologies that can treat soils and sediments contaminated with metals and radionuclides at a cost below the baseline technology.

The global objectives for the study are:

- (1) To determine an acceptable treatment method to ensure that the mercury does not leach from the soil above acceptable levels,
- (2) Complete all work safely to protect the health of workers, public and the environment, and
- (3) To demonstrate that the method can be used in the field.

The third objective is for the Phase 2 optional field demonstration study and may be performed at DOE's Y-12 Plant or at the off-site properties.

The treatment technology objectives, as defined in the solicitation, was to reduce the contamination levels to render it below RCRA characteristic levels for the specified contaminant (DOE Solicitation DE-RA26-01NT41030). Specifically, an in-situ process was to render the treatment before excavation and an ex-situ process was to excavate the soil then treated it. For in-situ treatment, this meant that one or more of the following must be met:

- Soil less than the TC criteria for mercury (0.2 mg/l in TCLP leachate), or
- Ten times the Universal Treatment Standard (UTS) (0.25 mg/l) in TCLP leachate.

For ex-situ treatment, this means that one or more of the following must be met:

- Ninety (90) percent removal of mercury in the leachate from a TCLP test (for immobilization technologies)
- Ten (10) times the UTS (0.25 mg/l in TCLP leachate).

As well as the above treatment performance criteria for mercury, the treatment must document an estimated performance cost lower than LTTD baseline treatment when implemented on a volume of up to 50,000 cy, including all capital costs. The baseline cost ranges from \$35 to \$37 million. The baseline cost did not include the disposal costs for the collected mercury or any additional treatment for other RCRA constituents of concern.

As specified in the solicitation IT/NFS is presenting a cost comparison to the baseline. A comparison to other aspects of the baseline technology, e.g., the schedule, cannot be made at this time since IT/NFS do not have a copy of the baseline study.

1.4 BACKGROUND AND DOE NEEDS

DOE has many sites with mercury-contaminated materials and wastes. See References 1 and 18 for a more complete description of mercury-contaminated waste sites within DOE. Treatment of mercury contaminated radiological wastes, soils, and sediments may be divided into several different categories.¹⁸

- Amalgamation for treatment of elemental mercury,
- Stabilization for wastes with <260 mg/kg (parts per million [ppm]) of mercury to comply with the Universal Treatment Standard/Underlying Hazardous Constituent (UTS/UHC) TCLP Hg concentration of 0.025 mg/l. (See **Table 1-1** for TC and UTS limits for RCRA metals.),
- Separation and treatment of collected mercury with >260 ppm of mercury (i.e., separating by roasting, retorting, incineration, and chemical extraction; treatment by recycle, amalgamation, or stabilization),
- Petition EPA for a variance for mercury >260 ppm to treat to land band criteria, and for soils
- Meet the alternative treatment standard for soils (90% contaminant reduction capped by 10 times UTS values).

The last treatment category applies to the soil in this project.

1.5 PROJECT SPECIFIC NEEDS

This project deals specifically with mercury-contaminated soils which have not been excavated and are still under the control of DOE, or where DOE may be a principle in the clean up of a site. As such, the soil is not currently regulated by RCRA. DOE is evaluating treatment alternatives to control mercury concentrations in the groundwater and surface water.

The treatment must reduce the transportability

(particulate transport and leachability) of mercury to the creek and minimize potential exposure

Table 1-1 Toxicity Characteristics and Universal Treatment Standard for RCRA Metals		
Metal	TC (mg/l)	Phase IV UTS (mg/l)
Arsenic	5.0	5.0
Barium	100.0	21.
Cadmium	1.0	0.11
Chromium	5.0	0.6
Lead	5.0	0.75
Mercury	0.2	0.025
Selenium	1.0	5.7
Silver	5.0	0.14
Antimony	A	1.15
Beryllium	A	1.22
Nickel	A	11.
Thallium	A	0.2
Vanadium	A	1.6
Zinc	A	4.3
A TC limit not established		

to site workers. This cleanup action is under a Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) regulation.

Under this action, contaminated site soils and sediments at the Y-12 Plant are to be remediated to protect surface water, current and future industrial workers from exposure. These soils may be contaminated with mercury, other heavy metals and radionuclides (primarily uranium). The preferred remedy for roughly 130,000 cy of contaminated soils and sediments is excavation and disposal. It is expected that up to 50,000 cy of the excavated material will not meet the waste acceptance criteria (WAC) of the proposed on-site disposal location, EMWMF, because of failing RCRA LDRs. Although the excavated material is not anticipated to be a RCRA listed waste, it is likely that portions of the soils will not pass the TCLP screen and will be considered RCRA characteristic wastes for mercury. A soil or sediment fails the TCLP for mercury when the mercury concentration is greater than or equal to 0.2 mg/l in the TCLP extract.

This technology study is focused on needs identified by the DOE Nuclear Weapons Complex sites for technologies to treat, remove, or immobilize mercury in soil and sediment. The treatment of uranium and other radionuclides and heavy metals is of secondary interest. DOE hopes that significant cost savings for remediation could result in using improved technologies compared to the baseline technologies. The baseline technologies are as follows:

- Removal of contaminated soils and sediments (and possibly buildings) and treatment of the contaminated material using thermal desorption. The estimated cost for the treatment operation is \$740/cy.
- Capture of waters, which contain low levels of mercury bound for Upper East Fork Poplar Creek (UEFPC) and treatment via sorption with granular activated carbon and/or enhanced air stripping.

1.6 DOE, NETL, BJC MODIFICATION AND CLARIFICATIONS OF THE PROJECT

During the Kickoff meeting, DOE stated that it was known that an in-situ process would not be able to treat all the contaminated soil. There are areas that have significant quantities of debris and utilities. This will limit reasonable access. In addition, treatment under building will likely be limited. It was also stated that much of the soil would have to be excavated simply because of potential radiological contamination, irrespective of the level of mercury contamination. Since

the IT/NFS process is adaptable to both in-situ and ex-situ treatment, it was suggested that IT/NFS consider both in-situ and ex-situ treatments. Later during the project, IT/NFS was given specific directions from NETL and BJC to discuss in their report both in-situ and ex-situ treatments. Specifically, IT/NFS was to provide the following items on in-situ and ex-situ treatment processes.

- General discussions of the advantages/disadvantages of in-situ and ex-situ technology,
- Concepts of the treatment processes, and
- Engineering level price estimates for both types of technology.

For the engineering price estimates, IT/NFS was directed to make the estimate as comparable to the baseline study as reasonably possible. The following boundary conditions were placed on the treatment processes in the cost estimates.

- Both in-situ and ex-situ treatments. The total quantity of soil to be treated is 50,000 cy. Define the process with only one treatment location since it is not known how many different sites will be treated.
- In-situ treatment: DOE will define the location for treatment. IT/NFS is to excavate the soil after treatment and place it in a pile for DOE to transport to the EMWMF.
- Ex-situ: The pricing is based on a pile to pile process. IT/NFS was to assume that DOE would provide a pile of contaminated soil next to the treatment process. IT/NFS will transport the soil to the treatment operation and place it in another pile for DOE to transport to the EMWMF.

IT/NFS was also directed to spike the received Alpha 2 building soil with mercury since the TCLP leachability of the as-received soil was well below the Statement of Work performance criteria.

The key results of the project are as follows:

- The treatment cost is less than 30 percent the baseline cost.
- IT and NFS both succeeded in demonstrating processes for stabilization of mercury-contaminated soil to meet the project performance criteria (TCLP 0.2 mg/l mercury).
- Treatment formulations were also determined which treated the soil to below the UTS-UHC treatment standard (0.025 mg/l in the TCLP.)
- Treatment processes were determined which may be used in-situ or ex-situ.
- The selected treatment formulations do not show any deleterious aging effects over a 30-day cure period.
- The volume increase was less than 27.5 percent for both the IT and NFS processes. This volume increase includes the addition of enough Portland cement to dry the soil to meet the EMWMF WAC. The volume increase was larger than observed on previous projects because of the soils' high clay content.
- The initial product is a damp to slightly wet material where a drying agent may be added to ensure that the product passes the paint filter test requirement.
- Besides personal protective equipment (PPE), general equipment cleaning solids, decontamination liquids from the last treatment, and potentially a small amount of collected metallic mercury there are no secondary waste generations from the process. The collected metallic mercury will be treated to meet the performance criteria.

3.1 STATEMENT OF WORK FOR PHASE 1 AND OPTIONAL PHASE 2

The project was organized into a series of tasks. These tasks were defined in an agreed upon Statement of Work (SOW) for Phase 1 and the draft SOW for Phase 2. The major tasks from the Phase 1 SOW are described below. These tasks were modified from the original SOW as previously noted. The complete SOW is in **Appendix 1**. These Phase 1 tasks are as follows.

Task -- Coordination and Planning

This task consisted of necessary activities to ensure coordination and planning of the project with DOE/NETL, representatives from the site end-users.

Task -- Bench Scale Testing Work Plan

IT/NFS prepared and submitted for review a Work Plan for the bench-scale testing of mercury contaminated soil. The Work Plan was revised and approved by NETL, DOE, BJC and the state of TN.

Task -- Bench Scale Testing

IT/NFS conducted a bench-scale treatability study at each Contractor-provided facility. Testing was conducted in accordance with the approved Work Plan. Initial treatment tests were completed to identify reagent formulations for scale-up proving tests. Results were evaluated in respect to achieving TCLP criteria for mercury. Minimizing final waste volume increases was emphasized. Initial test results were evaluated and refinements designed to enhance operation of the technology system, and retesting was completed. Proving tests were conducted at NFS laboratory to verify process chemistries derived in the Scoping tests. These proving tests were called scale-up demonstrations. Technology performance and cost benefit analyses were completed for in-situ and ex-situ process using results from both IT and NFS bench-scale testing results.

Task -- Reporting

Attended and presented at the Annual Industry Programs Conference and the Subsurface Contaminant Focus Area Annual Midyear Review. IT/NFS prepared for review and comments, a draft Topical Report on the Phase 1 contract activities. After review and comment by the DOE and DOE selected representatives, the Contractor revised the report and submitted it to DOE. IT/NFS prepared and presented a briefing of Phase 1 results.

3.2 SUMMARY OF TASK COMPLETENESS

IT/NFS successfully completed all tasks defined in the SOW for Phase 1. **Table 3-1** lists each milestone and major event and the date that was started. **Figure 3-1** is a Gantt chart for the project showing the start and finish of all major tasks.

Table 3-1 Milestones And Major Events		
Milestone or Major Event Description	Baseline	Actual
NEPA Documentation	10/01/01	10/01/01
AIPC & SCFA Annual Midyear Review	10/30/01 and 3/15/02	10/30/01 and 03/05/02
Submit Permits & Licenses	11/01/01	10/18/01
Submitted final Bench-scale Testing Plans	12/21/01	12/21/01
Receive Soil Samples	12/21/01	12/21/01
Operation of Technology System (As Is Material)	12/31/01	01/08/02
Operation of Technology System (Treating "Spiked" Performance-Based Soil)	12/31/01	02/21/02 (Prepared Spike 1: 02/04/02, Spike 2; 02/11/02, Spike 3: 02/21/02)
Retesting of Technology System	3/13/02	04/08/02
Members of NETL, DOE-Oak Ridge, and BJC Visited NFS and IT Facility to Observe Treatment Process, Copies of Presentations sent to NETL, DOE, and BJC.	Spring 2002	04/11/02
Secondary Sampling and Analysis	04/02/02	04/10/02
Summary Presentation	Spring 2002	08/08/02
Final Report	07/01/02	08/14/02
AIPC – Annual Industry Programs Contractor SCFA – Subsurface Contamination Focus Area		

4.1 DEFINITIONS

Amalgamation, solidification, fixation and stabilization are terms that are often used to describe mercury treatment. The later three terms are often interchanged, sometimes inappropriately, when used to describe a process for the treatment of hazardous, toxic and radioactive wastes (HTRW). Definitions of each of these terms are given below.^{24-30, 35}

- Solidification treatments either convert a liquid material/waste to a solid form and/or encapsulate the wastes into monolithic solids of moderate to high structural integrity. Regulatorially, the solidified material must pass the paint filter test (SW-846 Method 9095) and sometimes the liquid release test where it is determined if liquid will be released when a load (typically 50 pounds per square inch [psi]) is applied (SW-846 Method 9096). Solidification treatments do not necessarily involve chemical reactions between the contaminants in the waste and the reagents; however, it may mechanically bind/encapsulate the contaminants into the monolith. Contaminant migration is restricted by the vast decrease in surface area available for leaching, isolation of the wastes within an impervious monolith, and often the lower permeability of the resulting waste form.
- Fixation refers to treatment processes that reduce the hazard potential of a waste by converting the contaminants into one of their least soluble forms thereby minimizing the mobility and toxicity of the hazardous material and their impact on the environment and human health. Fixation is typically achieved by the chemical reaction of hazardous constituents in the waste matrix with chemical additives or by absorption of the hazardous constituents onto specific additives (e.g., activated carbon, clays, etc.) Sometimes, the process may use multiple steps (e.g., the metal of interest is first converted to more soluble or reactive form, then reacted to form the low solubility/low mobility compounds).
- Stabilization treatment is combines fixation and solidification. Where the hazardous constituents are reacted with ingredients of the formulation and converted to minimal soluble forms or adsorbed to control leachability and converted to a solid form. This may be accomplished with single reagent or multiple reagents (e.g., addition of only Portland cement or the dual addition of phosphate to fixate lead and Class C fly ash to adsorb the liquid phase.) The process results in treated waste, which is a lower permeability solid where the contaminants are both chemically reacted and physically bound within the matrix.

- Solidification/Stabilization (S/S) treatment is a process that combines the attributes of solidification and stabilization.
- Amalgamation is the “reaction” of liquid mercury to form a solid solution with other metals. Mercury forms amalgams with most all metals except for a few (e.g., iron and aluminum.) This reaction solidifies the liquid mercury, lowers the vapor pressure and leachability of the elemental mercury. Since it solidifies the liquid and controls the leachability, the amalgamation process, is a stabilization process. By RCRA standards, the reaction of sulfur with liquid mercury is also amalgamation.
- In-situ Treatment: In-situ treatment methods treat the contaminated material in place by mixing reagents directly into the waste matrix. For this project, IT/NFS proposes to use an auger to mix the reagents with the contaminated soil. The first set of reagents would be added on one day and the second set added on a different day. The treatment processes are based on the experimental results from this bench-scale study. In-situ treatment is further described in the “Demonstration-Scale Concept” section of this report.
- Ex-Situ Treatment: As used in the context of this project, contaminated material is excavated and transported to a central treatment unit for processing to meet the performance criteria. The central processing unit may be adjacent to the area where the soil is excavated or located remotely. For this project, IT/NFS proposes to use a pugmill to mix the reagents with the contaminated soil. The IT and NFS treatment processes are different. The IT process will use a continuous pugmill to mix the first reagents on day one and then blend in the second reagent in another continuous pugmill on day two. The NFS chemistry will be conducted using batch treatments where various reagents are added, allowed to react, then more reagents added. Both IT and NFS processes are based on the experimental results from this bench-scale study. Ex-situ treatment is further described in the “Demonstration-Scale Concept” section of the report.

4.2 RELEVANT CHEMISTRY

4.2.1 Amalgamation

Amalgamation of mercury has been well described by DOE and other sources. DOE and others have experimented with liquid mercury amalgamations using zinc, copper, tin, gold and silver. The latter two are often considered too expensive for mercury remediation. Amalgamation with tin often does not meet the performance goals. Amalgamations with zinc or copper do not routinely lower the TCLP leachability to below the UTS limit. Combinations of zinc and copper solidify the liquid mercury and may lower the TCLP value to regulatory levels. Reviews on amalgamation and other Hg S/S technologies are in References.^{4,35} The DOE's 1997 Mixed Waste Focus Area (MWFA) review of amalgamation (Reference 4) states that

- Amalgamation often lowers the leachability of mercury by 100 to 1000 fold and
- Mercury is one of the most difficult hazardous materials to treat to meet regulatory requirements.

Amalgamation reagents used in this project may have included, but not limited to, those metals listed in Reference 4, i.e., copper, zinc, tin, gold and sulfur.

4.2.2 Chemical Fixation

IT/NFS not only relies on amalgamation, but also on chemical fixation of the mercury because of the difficulty meeting the TCLP leaching performance criteria using only amalgamation. Combining amalgamation and fixation ensures that the mercury TCLP leachability of is controlled. IT often used the addition of materials containing sulfides or phosphates to fixate the mercury. In the process, sulfur and sulfides fixate soluble forms of Hg, react with the small amounts of remaining metallic mercury (Hg^0) present after the amalgamation step, and fixate many other metals and radionuclides that may be present in the soil. The metals and radionuclides are converted to sparingly soluble metal sulfides or metal sulfide type complexes. Typical sulfur compounds that may have been used in this project include, but not limited to, powdered elemental sulfur, sodium sulfide (Na_2S), ferrous sulfide (FeS), trimercaptotriazine, diethyldithiocarbamate, and sodium or calcium polysulfide (CaS_4 , CaS_5 , etc., also called lime sulfur). A description of sulfide-based chemistry follows.^{4,5,6,18,24,35}

In the sulfur/sulfide process, mercury-contaminated soils or wastes are blended with fixation solutions containing sulfur compounds. These reactions convert the mercury and other metals to sparingly soluble metal sulfides or metal sulfide type complexes. These sparingly soluble salts have very small solubility product constants (K_{sp}). Examples of the solubility product for metal sulfides are shown in **Table 4-1**.

The solubilities of salts or minerals are often compared using their solubility product constant (K_{sp}) values. For a given empirical structure (e.g., C_xA_y where C and A are cations and anions and x and y are the number of ions), the solubility decreases with smaller K_{sp} values.

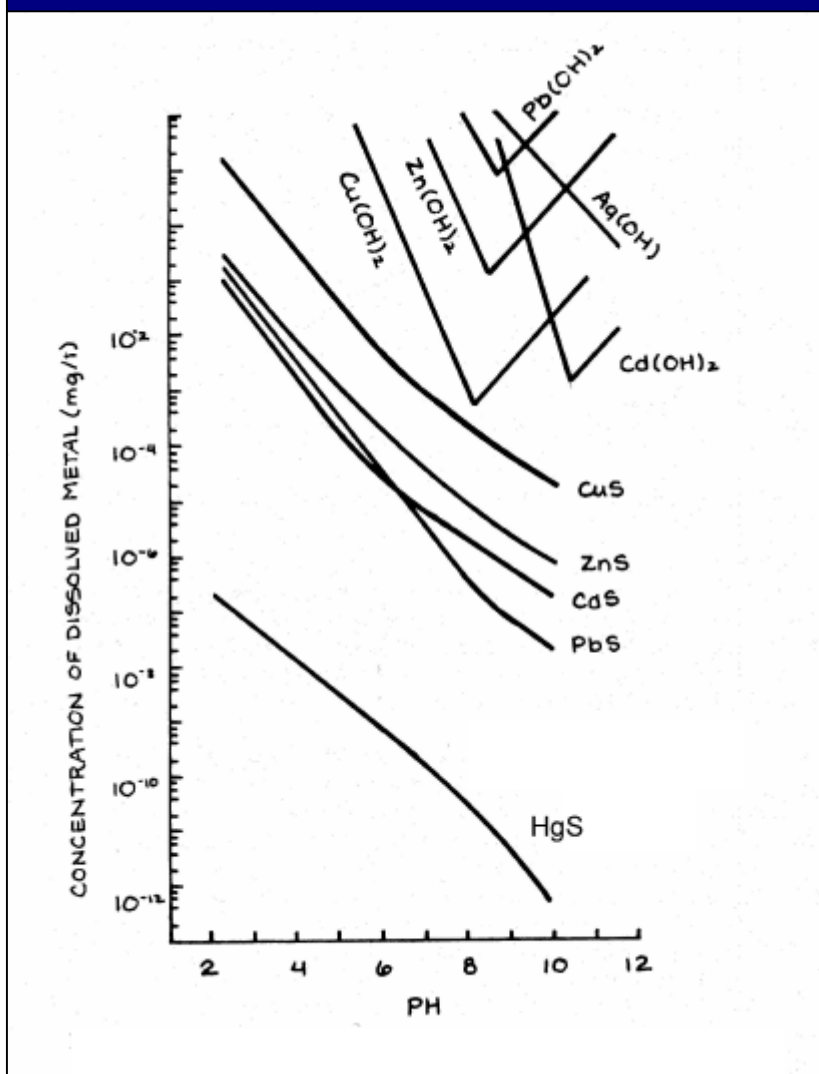
Mathematically K_{sp} for C_xA_y is defined as

$$K_{sp} = [C^{y+}]_x [B^{x-}]_y,$$

where [] indicate the molar concentration (mole/l) of ions in solution. Solubilities of most heavy metal sulfides are less than the corresponding hydroxide or carbonate salts. The small K_{sp} values for these salts show that these compounds have low solubility and that sulfur/sulfide addition are good candidate for the fixation of mercury. **Figure 4-1** (U.S. EPA, Federal Register. 52[155]: 29999 [Aug. 12, 1987]) shows the solubility of several RCRA metals hydroxides, carbonates and sulfides as a function of pH. Analysis of **Figure 4-1** graphically shows the solubilities of most heavy metal sulfides are less than the corresponding hydroxide or carbonate salts.

Table 4-1 Solubility Product for Common Metal Sulfides			
Compound	K _{sp}	Compound	K _{sp}
Al ₂ S ₃	2.0 x 10 ⁻⁷	MnS	1 x 10 ⁻¹¹
Bi ₂ S ₃	1 x 10 ⁻⁹⁶	HgS	3 x 10 ⁻⁵²
CdS	7.0 x 10 ⁻²⁷	Hg ₂ S	3 x 10 ⁻²⁰
CoS	5.0 x 10 ⁻²²	NiS	2.0 x 10 ⁻²²
CuS	8.0 x 10 ⁻³⁶	Ag ₂ S	8.0 x 10 ⁻⁵⁰
Fe ₂ S ₃	1.0 x 10 ⁻⁸⁸	Tl ₂ S	1.2 x 10 ⁻²⁴
FeS	3.0 x 10 ⁻¹⁷	SnS	1.3 x 10 ⁻²⁷
PbS	1.3 x 10 ⁻²⁸	ZnS	1.6 x 10 ⁻²³

Figure 4-1
pH versus Concentration of Dissolved Metal (mg/l)



The sulfur-based fixation reagents may have included, but not limited to, reagents listed in **Table 4-2**. The combination of amalgamation, fixation with sulfur/sulfide treatment and application of proprietary reagents, when needed, allowed IT and NFS to treat mercury to well below the performance criteria in the SOW.

Table 4-2 Typical Sulfur/Sulfide-Based Mercury Fixation Reagents	
Ferrous Sulfide	Slightly soluble sulfide salt. This salt slowly releases sulfide into aqueous matrices as it dissolves. As sulfide is removed from system by reactions with mercury and other metals, more sulfide is released by the dissolution of the reagent.
Sodium Sulfide	Very water soluble sulfide salt. Produces immediate "high" concentration of sulfide ions to react with mercury and other heavy metals.
Calcium Polysulfide	Soluble polysulfide releases reactive sulfur and sulfides to fixate metallic, ionized mercury, and mercury salts.
Degussa TMT	Commercial polymer that is very effective at precipitating many heavy metals, including mercury, and radionuclides; the chemical name is trimercaptotriazine.
Nalmet 8154	Commercial polymer that is very effective at precipitating many heavy metals, including mercury, and radionuclides; the chemical name is diethyldithiocarbamate.

4.2.3 Solubility and TCLP Values

The maximum concentration (mg/l) a constituent of concern (COC) can have in the TCLP is its total concentration (mg/kg) divided by 20. This assumes that all the material is solubilized and leaches into the TCLP extract. Some of the COC may be immobilized by interaction with the soil or waste or by the treatment process. This lowers the concentration in the TCLP extract. The concentration in the extract may also be limited by the solubility of the COC in the TCLP extract. Metallic Hg concentration is limited by its solubility. In screening tests with Hg^0 , the TCLP Hg concentrations were typically less than 0.035 mg/l. As the Hg^0 is allowed to oxidize to form more soluble Hg^+ or Hg^{2+} compounds, the TCLP Hg values increase.

The impact of this phenomenon is that the TCLP Hg concentration will not change once the TCLP solution is saturated in Hg^0 . As such, in IT/NFS experiments where Hg^0 spikes were used, there was no impact on the untreated TCLP Hg values whether the soil was spike with 1000-1500 or 5000-6300 mg/kg Hg^0 . The addition of more soluble Hg salt (i.e., mercuric oxide and mercuric chloride) to the soil did affect the TCLP Hg results. Obviously, the quantity of amalgamating reagent increased with the higher Hg^0 concentration in the spiked surrogate. (Note that in all engineering cost estimates, the reagent dosage needed to treat the largest concentration of Hg spiked to the soil was used.)

4.3 TECHNOLOGY SELECTION AND DESCRIPTION

Various existing in-situ and ex-situ treatment technologies have potential to treat the soils described for this project. The technologies are at various states of maturity. Some have been specifically tested for Hg treatment; others by their nature, should be expected to have potential to treat Hg in the site soils. IT did an abbreviated Feasibility Study (FS) level comparison of

several of these technologies. **Table 4-3** summarizes IT's analysis. The table provides a description of each technology, its advantages, disadvantages, and limitations. For the technologies proven to work with Hg, IT and NFS believe that their chemistries and processes provides the best benefit to DOE.

Table 4-3

Alternative Treatment Technologies

Technology	Description	Positives	Negatives	Limitation And Comment
IT/NFS Team Technology - In-Situ Treatment with Proprietary Chemicals	In-situ treatment designed to minimize mercury (Hg) vapor emissions, fixate Hg and other RCRA metals, and lower leachability of typical radionuclides with proven amalgamation and fixation chemistries.	<p>Elemental mercury is stabilized using one or more amalgamation agents. Chemical stabilization processes using reagents to break mercury complexes and allow removal of mercury as a precipitant.⁵²</p> <p>In previous studies, the mercury levels in the waste product meet the Envirocare Waste Acceptance Criteria.⁵²</p>	The treatment process is a multiple step process that uses proprietary reagents.	<p>The most significant limitation, as with all alternative stabilization technologies, is the need to achieve good mixing. In this project, extra water was added and the use of higher shear mixing was used to achieve the necessary good mixing.</p> <p>The volume of the treated soil is increased.</p> <p>Volatilization and emission of volatile organic compounds may occur during mixing procedures and emissions control may be warranted.³⁴</p>

Table 4-3
Alternative Treatment Technologies

Technology	Description	Positives	Negatives	Limitation And Comment
Solidification/stabilization (also referred to as immobilization)	Adding of pozzolanic reagents with or without additives to change the physical and chemical characteristics to immobilize contaminants. ^{33,50,52}	<p>The technology has been adapted to in-situ applications with various augers that provide reagent delivery and mixing.</p> <p>In situ treatment should have a cost saving over ex situ applications for larger volumes and for depths greater than 10 ft.</p> <p>Broadly applicable to a range of metals.</p>	Using conventional S/S technology, the process often does not treat Hg to meet the UTS criterion.	<p>This is a commercial technology. There are concerns with Hg treatment about the long-term integrity of the waste.³³</p> <p>Must consider, as with most in-situ treatments, the amount of debris in the soil and the depth of contamination.</p> <p>Performance is dependent on mixing efficiency. Soil having high clay content or significant debris may be difficult to mix.</p> <p>The volume of treated material will increase with the addition of reagent.³⁴</p> <p>Organics are usually not effectively treated using standard binding/stabilizing agents.</p> <p>Volatilization and emission of volatile organic compounds may occur during mixing procedures and emissions control may be warranted.</p>

Table 4-3
Alternative Treatment Technologies

Technology	Description	Positives	Negatives	Limitation And Comment
Cation Displacement and Soil Flushing ³³	<p>Injection and recapture of an extraction solution to flush heavy or radioactive metals from the subsurface. This method was originally based on in situ mining technology.</p> <p>Soil flushing is mostly used for organic contamination.</p>	<p>Water and chemical flushing solutions have successfully been used at the laboratory and larger scales on various metals (e.g., chromium, uranium and lead). There are on-going studies on other metals including Hg.</p>	<p>Cations that occur naturally, or that are present in the extraction solution, remain in the soil.</p>	<p>If necessary, a stabilizing solution is injected after soil flushing has been completed to react with the remaining contaminants to produce an immobile species and prevent further migration of residual metals.</p> <p>To determine the selection of the flushing solution and predict the effectiveness of soil flushing, soil hydrogeology and areal and vertical concentration gradients for the contaminants must be obtained.</p> <p>The water soil flushing is a commercial technology. The chemical reagent soil flushing has only been used in limited research. The addition of chelants may mobilize metals toward the site aquifer.</p>

Table 4-3
Alternative Treatment Technologies

Technology	Description	Positives	Negatives	Limitation And Comment
In-Situ Electrokinetics	In-situ treatment to lower metals concentration in soil. The process applies a low-intensity direct current (DC) or a combination of DC and alternating current (AC) through the soil between electrodes mobilizing charged species, ions and water to move toward electrodes. ³³	<p>The metals can be removed from the soil and concentrated on/around the electrodes with minimal intrusive operations. Non-ionic and some non-charged species will be transported along with the electroosmosis induced water flow.^{42-45,49,52}</p> <p>The process is stable over time.</p> <p>There has been a full-scale application in Europe.</p>	<p>Deposits that exhibit very high electrical conductivity, such as ore deposits, cause ineffectiveness.</p> <p>Oxidation/reduction reactions can form undesirable products.</p> <p>Metallic electrodes may dissolve and introduce corrosive products into the soil mass.</p> <p>Buried metal objects divert current from soil requiring remediation.</p> <p>Large concentration gradients in areas surrounding electrodes.</p>	<p>From the nonvendor open literature reports, the process is limited by soil moisture content, depth of contamination, and homogeneity of the soil. Water, salts, pH buffers, and potentially chelants may be added to the soil to maintain movement of the constituent of concern. The addition of chelants may mobilize metals toward the site aquifer. The process may produce a potentially hazardous material as the process concentrates hazardous, toxic, radioactive materials near/on the electrodes. The mercury collected at the electrodes must be treated before disposal. ^{21,22,23}</p> <p>The technology has recently been licensed in the U.S. Must consider the homogeneity of the soil and the moisture level in the soil.³³</p> <p>This technology is confined to sites contaminated with metals.B May raise the soil pH to levels that could inhibit or destroy microbial populations present.</p> <p>Chlorine gas may be formed from the reduction of chlorine ions near the anode.³⁴</p> <p>Electrical currents could concentrate in certain areas of the soil profile, bypassing other contaminated soil zones.⁴⁹</p> <p>Indigenous biological organisms could be killed.⁴⁹</p>

Table 4-3
Alternative Treatment Technologies

Technology	Description	Positives	Negatives	Limitation And Comment
In-Situ Injection of H ₂ S ⁵²	Injection of a low concentration of H ₂ S gas mixture (100-200 ppmv) into soils, where it reacts with oxidized metals such as Cr ⁺⁶ uranium, Hg followed by the extraction of gas.	Performed well on chromate-contaminated soils.	Potential Health and Safety (H&S) issues.	IT believes that performance of this technology in tight clay would be limited. The effectiveness of the technology is limited by subsurface heterogeneities, with channeling of the injected gases in the most permeable materials. (ex: sand)

Table 4-3
Alternative Treatment Technologies

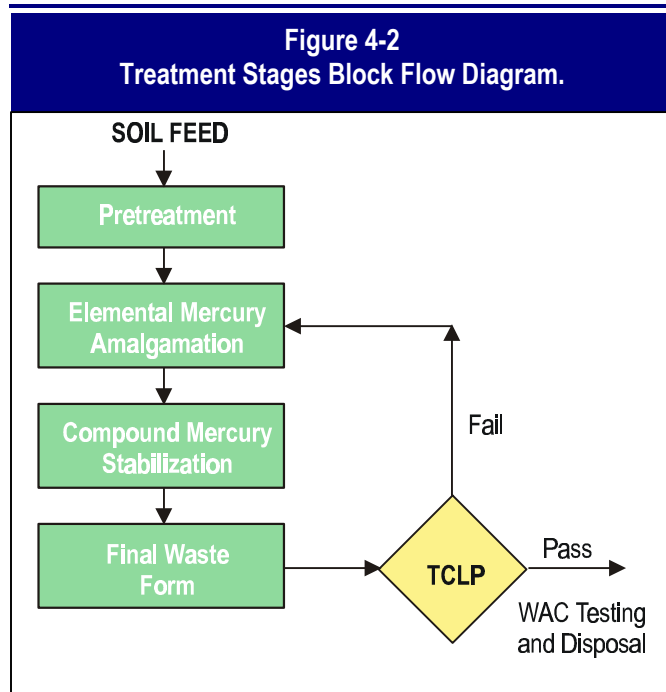
Technology	Description	Positives	Negatives	Limitation And Comment
In-Situ Thermal Removal with or without Vacuum Recovery ^{36,41,51,52}	A series of electrical heating rods are installed in the soil (typically 10 to 20-ft centers in hexagonal grid). These heating elements are used to heat the soil to temperatures sufficient to vaporize elemental mercury and mercury oxides (800 to 900 °F). Mercury vapors are pulled from the soil using soil vapor extraction wells. The mercury compounds are recovered in an offgas treatment system that can include condensation, wet scrubbing and adsorption on activated carbon or other media.	This technology can be carried out without excavating the soil, which may minimize migration of “veins” of elemental mercury.	Technology is not proven for Hg removal from soil. The results of bench-scale tests have been promising.	In the correct site location, the process has a high potential for effectively removing metallic Hg to below regulatory concern. The IT strongly considered this process to propose in this PRDA response as a second alternative treatment technology, but the process is not yet proven for Hg. Mercury emissions from the offgas cleaning system will be a concern. Elemental mercury and “Hg butter” will be produced in the offgas system and will collect on any cool spot, etc., in the off-gas equipment. It is unlikely the offgas system can be decontaminated at the end of the project. The process dries the soil under building and may cause subsidence and damage to building structure.

Table 4-3
Alternative Treatment Technologies

Technology	Description	Positives	Negatives	Limitation And Comment
Ex-Situ Sulfur Treatment in a Pugmill	S/S of mercury by combining liquid mercury with a sulfur mixture in a pugmill or similar mixing device. ⁵²	Processing is conducted at ambient conditions. ⁵² Treatment may pass Envirocare of Utah Waste Acceptance Criteria and TCLP limit for mercury. ⁵²	Air above the pug mill must be swept to remove mercury vapors and filtered through a HEPA filter and a sulfur-impregnated carbon filter to capture mercury. ⁵²	It is expected that it will be mechanically difficult to get elemental sulfur effectively in contact with Hg droplets in a high silty/clayey soil matrix. The chemical reaction between the sulfur and Hg is slow without application of heat. The process may not effectively treat many non-metallic forms of Hg.
Simple Amalgamation	Hg is treated by mixing amalgamation agents with elemental mercury. ^{4,18,52}	Treatment can be performed in an ambient hood. ⁵²	Before amalgamation, waste must be sorted, shredded, and slurried to create a homogeneous mixture. ⁵²	Mercury (I and II) oxides (Hg ₂ O and HgO) production must be optimized to ensure that at least 90 to 95% of the metallic Hg is amalgamated for the process to control the leachability. The process is often not effective at meeting LDR criterion for Hg (i.e., 0.025 mg/L in the TCLP.) Often need to optimize the combination of metals (e.g., use a combination of zinc and copper), optimize the pH, metal particle size range and distribution, and mixing intensity to get the chemical and physical properties necessary to successfully treat the elemental mercury.
Ex-Situ Molten Sulfur Treatment ^{7,37,38,39,46}	S/S of Hg contaminated materials or liquid mercury bearing medium. ^{7,37,38,39}	Product treated to meet LDR requirements. Solidified mass is very hard and its permeability is small.	Need to control the moisture content the material to be treated.	It may be mechanically difficult to get elemental sulfur effectively in contact with Hg droplets in a high silty/clayey soil matrix. The chemical reaction between the sulfur and Hg is slow without application of heat. As currently practiced, the process is a two step process where the waste or soil and sulfur materials are heated to approximately 40° C then the remainder of sulfur materials are added. The Hg volatilized have to be captured in the attached APC. ⁷

Table 4-3
Alternative Treatment Technologies

Technology	Description	Positives	Negatives	Limitation And Comment
Ex-Situ Low Temperature Thermal Treatment	Baseline Technology for the project.			Process is a proven technology that produces a condensed metallic Hg that DOE will likely have to treat before disposal. Potential problems with contamination and emissions from the APC system and production of "Hg butter" in any cool spot, etc., in the off-gas equipment. Due to potential for PCB and other organic compounds being present, the process may produce dioxins and furans during the processing of the soil.
Ex-Situ: IT/BNFL Sulfur Reducing Biological Process.				Ex-situ processes where final volume to be disposed is significantly less than original volume. IT/ has not yet proven the technology on soils with high concentrations of metallic Hg present. The Hg, as well as many other metals and radionuclides, are precipitated on the biomass as a metal sulfide. ⁵²



The amalgamation/fixation processes used in this project are based on more than ten years of research and demonstrations on mercury metal treatment processes. The commercial name for the NFS mercury treatment process is called DeHg[®] (pronounced “DEE-merc”).^{14, 15, 16, 17} IT’s process does not have a trademark name for its chemistry. Both the DeHg[®] and IT’s proprietary chemistries were investigated in this bench-scale project.

The processes used in this study consist of multiple stages. The IT and NFS processes used amalgamation, sulfur/sulfide-based reagent fixation, and several other proprietary compounds as the primary mechanism for reducing the leachability of the mercury, RCRA metals, and radionuclides. Portland cement, or similar materials, was used, as necessary, to increase the solid content of the treated materials to pass the paint filter test. By well-established processes, the addition of Portland cement and other conventional S/S reagents can also reduce the leachability of other metals and radionuclides in the final product.

The first stage of the treatment process involves amalgamation of the elemental mercury. Before amalgamation, it may be necessary to condition the contaminated soil by shredding, grinding, and/or slurring with water to improve the mixing characteristics. (It should be noted that size reduction was performed only prepare an appropriate feed to the mixing equipment used for this study. Size reduction is not a required element in order for the IT or NFS treatment chemistry to perform correctly.) In this bench-scale project, debris was removed and water was added to the make the soil more fluid to improve mixing. An overall flow diagram of the treatment scheme is shown in **Figure 4-2**.

The second stage of the process is stabilization/fixation of soluble or complexed mercury species using the proprietary DeHg[®] reagent or IT’s proprietary reagent. These reagents have the capability to free mercury from more soluble complexes and subsequently allow for its precipitation as a stable, nonleachable salt.

4.3.1 NFS DEHG® Process

DeHg® is an ambient temperature process that converts mercury in hazardous and mixed waste (waste containing both hazardous and radioactive components) to a RCRA non-hazardous material. If the contaminated material is mixed waste, it is converted to a low-level radioactive final waste form suitable for land disposal. The DeHg® reagent was originally developed in the early 1990's to treat solidified mercuric thiocyanate wastes when traditional mercury stabilization techniques failed to treat the strongly complexed mercury in this waste. DeHg® has enjoyed excellent success in demonstrating capability to stabilize a variety of DOE mercury mixed wastes. This has included radioactively contaminated elemental mercury (MER01), mercury (<260 ppm) and metal contaminated waste (MER02), various mercury species stabilization (MER02A), and mercury stabilization in DOE soils (MER03). The MER01 and MER02 projects have been previously reported in DOE EM Innovative Technology Summary Reports (ITSRs). DeHg® was also used in the MER04 program, a demonstration of capability to stabilize various mercury species in surrogate soil with total mercury concentration >260 ppm. The MER04 waste had similar total mercury levels as those defined in this project solicitation. The mercury concentrations were as follows:

- 1500 mg/kg Hg as elemental mercury
- 1200 mg/kg Hg as mercuric nitrate
- 1300 mg/kg Hg as mercury (II) oxide
- 1100 mg/kg Hg as mercury (II) chloride
- 500 mg/kg phenyl mercuric chloride.

In each of these important demonstration projects, DeHg® achieved all final waste form objectives, namely UTS for mercury and metals, and other applicable waste acceptance criteria. DeHg® has also addressed a variety of commercial mercury mixed wastes that have achieved acceptance criteria at licensed radioactive disposal sites. NFS has invoked a program of continuous improvement to enhance the capability of the process to address increasingly tighter regulations with regard to mercury stabilization. ^{2, 5, 6, 8, 9, 10, 11, 12, 13}

The treatment process is not limited by the amount of mercury in/on the materials to treat. The DeHg[®] process has successfully been demonstrated at the bench-scale for materials ranging from mercury-contaminated soil and debris to nearly pure Hg⁰. By the design of this project, the process is conducted at near ambient conditions of pressure and temperature. This lowers the potential for mercury volatilization.

4.3.2 IT's Mercury Stabilization Process

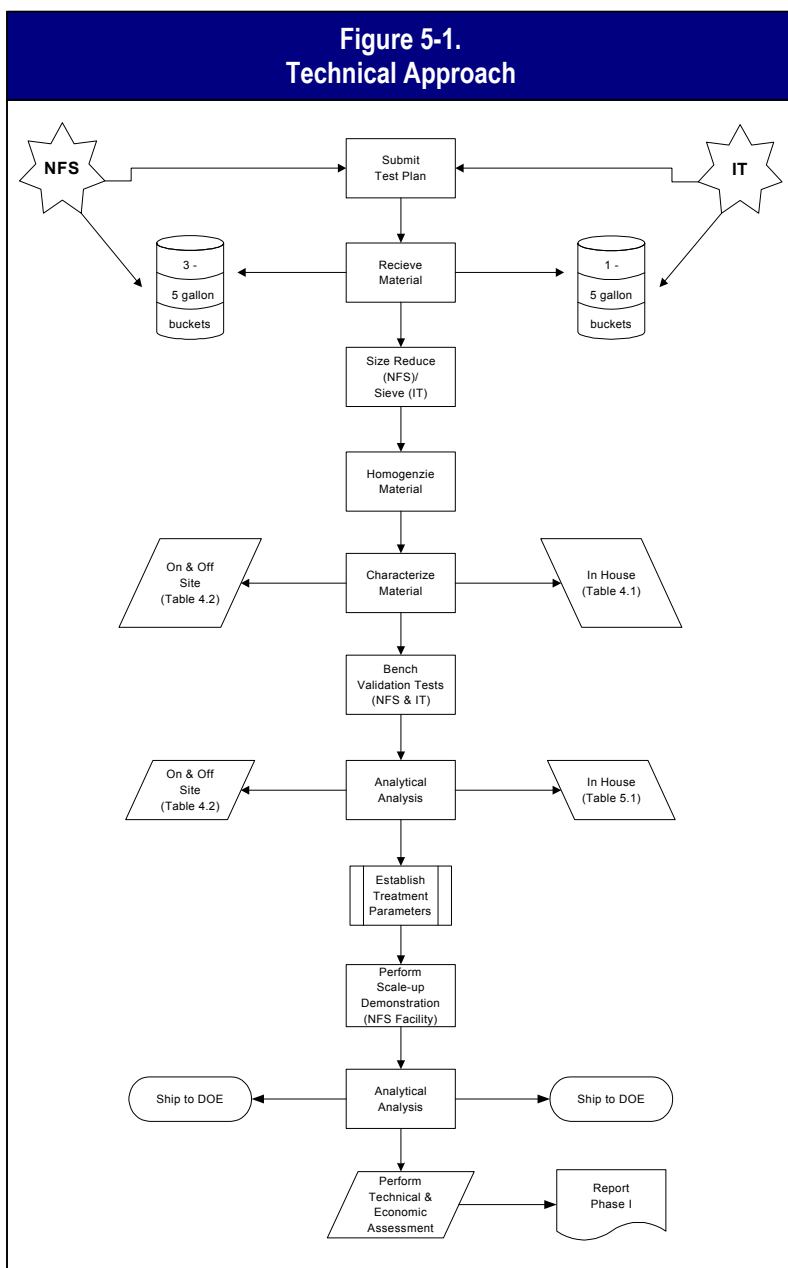
IT's process does not have a trademark name. It is an ambient temperature process that rapidly lowers the vapor pressure of mercury from above the contaminated material. The process chemistry has been applied to mercury and non-mercury contaminated materials to meet the performance requirements for all the RCRA metals and/or radionuclides of concern. It has been successfully applied to treat Hg⁰ condensed from LTDD pilot-studies, radioactive mercury from a confidential commercial client, and demonstrated on surrogate waste in the MER02 project. IT has improved the process to meet changing regulatory criterion.

5.1 INTRODUCTION

Bench-scale studies were conducted at IT's Technology Development Laboratory (TDL) in Knoxville, TN and NFS's Applied Technology Laboratory (ATL) in Erwin, TN. Small-scale treatment tests were conducted at TDL and ATL, while a series of larger size treatments (e.g., up to 21 kg) were conducted at ATL. The study followed the approved Workplan in **Appendix 3**. The Workplan was developed by IT/NFS, submitted for review and approved by DOE-selected technical personnel, and BJC personnel.

An overall flow diagram of the treatment scheme is shown in **Figure 5-1**. As the figure shows, the soil was received at both companies' facilities, size reduced by sieving to less than 0.5 (12.7 mm) (IT) or jaw-crushed to 0.236 (6 mm) (NFS) inch, homogenized, characterized, then subjected to treatments.

Although the general process flow was the same for IT and NFS, the experimental philosophies are different.



5.2 IT TREATABILITY STUDY PHILOSOPHY/APPROACH

IT philosophy is to determine the minimum and maximum amounts of each reagent that will successfully treat the contaminated material. IT experimental design develops the working envelop of formulations. The dosages investigated are based on both ratios of stoichiometric reactions and applications of standard sets of formulations. This determination of effective reagent envelop was for both the TCLP's of the freshly made and the >30-day cure samples. This philosophy ensured that IT could show there was an impact of reagent dosages. The approach purposely identifies formulations that fail one or both of the immediate or aged TCLP values for Hg. By spiking the "as is" soil with multiple levels of metallic Hg and moderately soluble HgO (about 153 mg/kg solution), IT was able to establish reagent dosages that were successful across a broad range of mercury values, e.g., 50 to 6000 mg/kg mercury.

5.3 NFS DeHg[®] TREATABILITY STUDY PHILOSOPHY/APPROACH

NFS has been conducting treatability studies on characteristic hazardous mercury waste using the proprietary DeHg[®] process for more than ten years. The DeHg[®] process was originally developed for a specific mercury mixed-waste that was generated at NFS. It was recognized shortly after the success of this process on the NFS waste that the chemistry of the DeHg[®] process would work on a wide variety of waste forms, and that it could be applied via a large assortment of processing equipment.

This is possible due to the unique ability of NFS to monitor the progress of the DeHg[®] chemistry, and apply only as much of the appropriate reagents as needed for the treatment of each individual waste batch. The effectiveness of this proprietary monitoring technique has been demonstrated repeatedly over a wide spectrum of waste forms, chemical mercury species, and processing scenarios. It has been demonstrated to be effective from beaker-scale laboratory tests all of the way up to 5000-pound full-scale treatment batches.

Thus the philosophy/approach of NFS to mercury waste treatability studies is more of a process validation/demonstration rather than that of a process development study. Since reagents are applied optimally in all tests based on prior experience, there is no need for reagent optimization experimental designs. Instead, the focus shifts to identifying the best processing equipment and the appropriate mixing and dwell times for the DeHg[®] reagents when a new waste stream is evaluated, particularly one with high clay content such as that found at Y-12.

5.4 IT PROCESS

5.4.1 IT Size Reduction, Homogenization, Hg Vapors

In a hood, the soil was hand sieved to remove debris greater than 0.5 inch. Soil clumps were broken by hand and sieved into the receiver container. The sieved soil was homogenized in an approximately 7-gallon Hobart Mixer. The cover to the mixing bowl was modified to attach a lid with an elephant hose vent. The vent was connected to the chemical hood vent system. The mixing system was located in front of a distillation hood. Before adding soil to the mixing bowl, the work area was smoke tested to ensure proper air flow. During the mixing operation, the mercury vapors were monitored at the opening of the mixer and in the breathing zones. The mercury vapor concentrations were normally below the detection limit (20 nanaograms per cubic meter [ng/m^3]) and were always below $30 \text{ ng}/\text{m}^3$.

The mercury vapor concentrations were also measured above the homogenized soil. Measurements were taken above the soil in the 5-gallon bulk sample container bucket and in the headspace of 150-milliliter specimen cups that had 45 grams of soil added to them. In the 5-gallon bucket test, the bucket top was removed and the material allowed to vent in the hood for nearly 10 minutes before taking the measurement. This test provides an indication of the mercury vapor release from a bulk non-confined source. In the specimen cup (headspace) tests, the containers were sealed to allow the vapors to build up. The top was removed and the vapor concentration immediately measured. This simulates a closed container situation. Similar type mercury volatilization experiments have previously been conducted at Oak Ridge National Laboratory (ORNL) on NFS, Brookhaven National Laboratory (BNL), and Applied Technology Group (ATG) treated wastes.¹⁹ There were significant mercury vapor concentrations measured above the soil. See **Section 6** for results.

5.4.2 IT Formulation Preparation

Aliquots of the homogenized materials were characterized using in-house analysis and subjected to treatments. The characterization and treatment results are in **Sections 6.1 and 6.2**. The treatments involved amalgamation and chemical fixation. As noted in **Section 4**, amalgamation is a solidification and fixation process (i.e., it converts the liquid mercury to a solid and decreases the leachability/solubility of the elemental mercury.) Chemical fixation treats the non-

elemental mercury. Before amalgamation, enough water was added to improve the mixing characteristics of the soil. The wetted soil had water content greater than its liquid limit and was a slightly fluid paste that would come off a large spoon in clumps when held at roughly a 75 to 80 degree angle from horizontal. The mercury vapor concentration above several mixes was measured to show that treatment significantly decreased the vapor emission rate.

Most tests were conducted on 45-gram samples. This allowed the total treated sample to be used in the MTCLP leachability procedure. In the 45-gram treatments, approximately 20 grams of water was added, the first reagent added on top of the water, then the water/reagent mixed in by hand. When there was a second reagent used, it was added to the first blend on the second day of treatment and mixed in by hand. No additional water was added with the second reagent. When the approximately one-kg sample was treated, it was processed in a Hobart planetary mixer using the same sequence and timing. The only significant differences were that all masses were scaled to larger values and the material was mixed with the Hobart mixer for about five minutes.

5.4.3 IT Spiking Soil with Mercury

During sample characterization, it was determined there was little leachable mercury in the soil. IT and NFS spiked the soil at three different levels as described below. All spiking procedures were reviewed and approved by NETL, BJC and their selected technical reviewers.

The soil was originally spiked with Hg^0 (1000 to 1500 mg/kg Hg). Metallic Hg was added to simulate an assumed maximum average concentration across a 10-foot column of soil at the ORR site. The TCLP Hg values for the Hg spike sample were similar to the unspiked soil. A second spike was made where 13 ± 0.5 mg/kg mercury (II) (Hg^{2+}) was added as mercuric (II) oxide (HgO) in addition to 1000-1500 mg/kg Hg^0 . HgO was added to these formulations to increase the quantity of “soluble” mercury and to represent aged Hg^0 after it has been partially oxidized. If all the added HgO leached from the soil in the TCLP, the TCLP Hg concentration would have been about 0.6 - 0.7 mg/l. This is three times greater than the performance criteria. The TCLP Hg value for this spiked soil was still below the performance criteria. A third spike was prepared. This final spike contained 100 ± 1 mg/kg Hg^{2+} as HgO and 5,000-6300 mg/kg Hg^0 . In this third series of spikes, the quantity of Hg^0 was increased to almost the maximum concentration reported in the solicitation and the HgO increased by an order of magnitude from the second spiked surrogate. The calculated TCLP Hg value for the third spike, if all the HgO

leached, was approximately 5 mg/l. These three spikes are described in the remainder of the document as

- Spike 1: 1000-1500 mg/kg Hg^0 ;
- Spike 2: 1000-1500 mg/kg Hg^0 combined with $13 \pm 0.5 \text{ mg/kg as } \text{HgO}$;
- Spike 3: 5000-6300 mg/kg Hg^0 plus $100 \pm 1 \text{ mg/kg } \text{Hg}^{2+} \text{ as } \text{HgO}$.

Results and further discussions are presented on the second and third spiked soils in **Section 6**. (The addition of Hg^0 had little impact on the TCLP Hg results; however, increasing the amount of Hg^0 did increase the amount of amalgamating reagent that was required. See the Relevant Chemistry for more details on mercury TCLP concentrations.)

There were three steps to prepare Spikes 2 and 3. (1) A sufficient quantity of HgO was added to the soil and well mixed to give the desired HgO concentration in the soil. The HgO solution had about 37 mg HgO/kg water. The resulting soil- HgO mixture had standing water on it and was well past the liquid limit. (2) The wet material was air dried for two days and the resulting soil clumps broken-up by hand. (3) The HgO spiked soil was then spiked with metallic Hg. In IT's procedure, very small drops (25-35 mg) of Hg^0 were added dropwise to each 45-gram or approximately 1-kg soil sample and well mixed. The 45-gram samples were mixed by hand while the approximately 1-kg sample was mixed in a four-quart Hobart planetary mixer.

Additional experiments were conducted to determine if liquid Hg would easily separate from the air-dried HgO spiked soil. In these experiments, Hg drops (25-35 mg each) were allowed to sit on top of the 45-grams soil samples without mixing for about 30 minutes. The Hg drops dispersed into the air-dried soil in less than 30 minutes. These containers were carefully inspected to locate any Hg. No metallic mercury could be found. Although no liquid Hg could be found in these experiments, all samples used for stabilization testing were well mixed to ensure dispersal of the liquid Hg throughout the sample before adding the treatment reagent(s).

5.5 NFS PROCESS

5.5.1 NFS Size Reduction, Homogenization, Hg Vapors

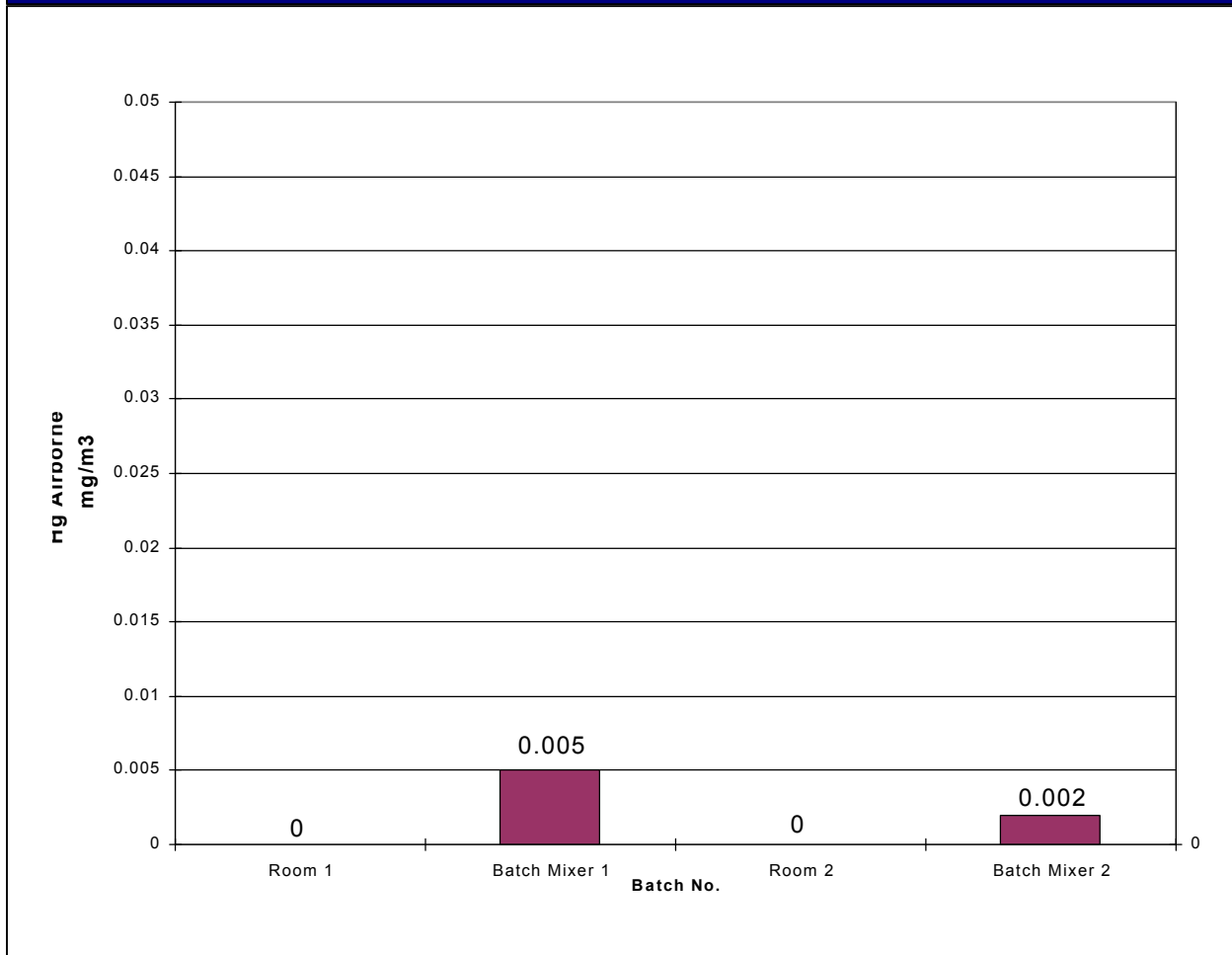
After receiving the three 5-gallon buckets of soil, NFS sized reduced the soil by crushing it in a jaw crusher to less than 6 millimeters (mm). Most all the crushed soil was less than 3 mm. All

15-gallons of size reduced soil was then homogenized in a drum-roller to present a single batch of material to test. All size reduction and homogenization operations were conducted in a specially designed containment area. This is the same containment area used by NFS in MER03 and MER04. It should be noted that size reduction was performed only in order to present an appropriate feed to the large-scale mixing equipment used for this study. Size reduction is not a required element in order for the NFS treatment chemistry to perform effectively.

Historical results from the MER03 and MER04 programs showed that the mercury levels in the containment area are well below Occupational Safety and Health Administration (OSHA) standards. In the NFS MER04 program, two batches of materials, each containing approximately 25-kg of untreated material, were run in the demonstration. Mercury levels throughout this study ran consistently lower than the federally mandated levels. The numbers reported in **Figure 5-2** are an average of the room readings (taken from the center of the containment) and an average of the batch mixer readings (taken directly over the batch mixer and in its opening).

The largest value in **Figure 5-2** is $1/5^{\text{th}}$ the American Conference of Governmental Hygienists (ACGIH) Threshold Limit Values-Time Weighted Average (TLV-TWA) criteria for mercury vapors. The results thus show that mercury vapors were safely contained within the working area and the treatment process controls the release of mercury vapors to the environment. These airborne mercury levels were used to estimate the quantity of mercury evolved during processing. Using a conservative flow-rate for the designated containment area, NFS estimates that a maximum of 0.05% of the initial inventory of mercury evolved during this demonstration. This is a negligible quantity released relative to the quantity of mercury input for the demonstration. Approximately 99.95% of the mercury input to the process was retained within the processing system.

Figure 5-2
Mercury Vapor Measurements Verses Batch Number During Processing



As described in **Section 4.3**, the MER04 soil had approximately the same concentration of elemental mercury as in the current project. Since the NFS process effectively controlled the mercury vapors in MER04, it is reasonable to expect that the mercury vapor emissions were effectively controlled by the process chemistry on this project.

5.5.2 NFS Formulation Development

NFS prepared the Spikes 1, 2, and 3 surrogates similar to those described previously in this report. Characterization results for the untreated spike soils are in **Section 6**. Treatment tests were conducted only on the Spike 3 surrogates.

NFS tested formulations using several types of mixers. Initial scoping tests were performed in a hood using a Kitchen Aid mixer and then a blender with approximately 0.50-kg specimens of Oak Ridge soil in each test. The last two scoping tests were performed in a blender in order to study the effects of higher shear mixing for dispersing the elemental mercury and treatment reagents throughout the clay-soil sample. Large-scale testing (i.e., the Scale-up demonstration tests) were conducted in the rotary DeHg[®] reactor capable of handling up to 45 kg of soil and reagents. In the scale-up testing, the nominal amount of feed soil was 21 kg. The test parameters and reagent dosages were selected based on NFS' extensive experience with mercury waste treatment and the results from the scoping tests.

5.5.3 NFS Spiking Soil with Mercury-Scoping Tests and Scale-up Demonstration

The as-received soil from Y-12 had TCLP leachate mercury concentrations below the UTS limit of 0.025 ppm. When 100% visually inspected, there was no evidence of elemental mercury in the soil. In order to provide a meaningful demonstration of the treatment technology, scoping tests were performed in which various combinations of elemental mercury and ionic mercury were added to aliquots of the soil prior to treatment. **Table 5-1** provides a description of the eleven scoping tests performed in this phase of the study.

TABLE 5-1: NFS SCOPING TESTS DESCRIPTION					
Test No.	Sample Wt., g	Spiked [Hg ⁰] ([Hg] as HgO), ppm	Objective	Result	TCLP (ppm)
1	200	5000 (0)	Initial test with Hg ⁰ spike	Mixing not acceptable. Larger sample needed for mixing vessel. Observed elemental Hg in treated waste.	0.713
2	500	1000 (0)	Duplicate of Test 1 using larger sample size.	Spiking with only Hg ⁰ not yielding high enough leach rate.	0.007
3	500	5000 (10)	Added 10 ppm Hg as HgO and Hg ⁰ spike.	Leaching rate still not high enough.	0.005
4	500	5000 (100)	Added 100 ppm Hg as HgO and Hg ⁰ spike.	Test 4 and 5 performed in the same day without test 4 results.	0.034
5	500	5000 (100)	Duplicate of test 4	Same as Test 4.	0.055

TABLE 5-1: NFS SCOPING TESTS DESCRIPTION

Test No.	Sample Wt., g	Spiked [Hg0] ([Hg] as HgO), ppm	Objective	Result	TCLP (ppm)
6	500	5000 (100)	Modified stabilization recipe.	Observed elemental Hg in treated waste.	0.082
7	500	5000 (100)	Modified amalgamation recipe.	Observed elemental Hg in treated waste.	0.005
8	500	5000 (100)	Re-treatment of test 6 residues.		0.029
9	500	5000 (100)	Modified amalgamation and stabilization recipes.	Observed elemental Hg in treated waste.	0.031
10	500	5000 (100)	Switched to higher shear mixed.	Better mixing, but need more water.	0.003
11	100	5000 (100)	Duplicate of Test 10 with additional water (smaller sample size needed to fit in blender).	Good. Ready to proceed to Large-scale tests.	0.001

The soil was spiked with elemental mercury in the Kitchen Aid® mixer used for the first nine scoping tests on a test-by-test basis. It was discovered that the Kitchen Aid® mixer bowl had a dead area in the bottom that allowed elemental mercury to collect and not be completely dispersed into the soil sample. This problem had not been encountered in past treatability studies using sandy soils. This problem persisted throughout the scoping tests despite efforts to add the mercury slowly using a transfer pipette while the mixer was running.

For the final two scoping tests, mixing was performed in a high shear blender. In these tests, the elemental mercury was well dispersed throughout the samples.

After the first two scoping tests, it was determined that spiking with elemental mercury alone did not provide a high enough mercury leaching rate for meaningful testing. For Tests 3 – 11, varying amounts of mercury as mercuric oxide were added to aliquots of soil prior to testing.

This was accomplished by first dissolving the mercury salt in water. A few milliliters of nitric acid were added to facilitate dissolution. The mercury-bearing solution was mixed into the soil using the Kitchen Aid® mixer, and then the soil was allowed to stand overnight to equilibrate.

Aliquots of these spiked soil samples were added to the Kitchen Aid® mixer, spiked with elemental mercury, and treated. **Table 5-2** provides the recipes used for spiking the soil samples for the scoping tests.

Table 5-2 – Mercury Spiking Recipes				
Soil Weight, (g)	HgO Added, (g)	Dissolution Water, (ml)	Solution [Hg], (ppm)	Final Soil [Hg], (ppm)
1200	0.0155	325	44	12
2000	0.216	1000	200	100
1500	0.164	800	190	101

5.5.4 NFS Scale-Up Demonstration

Nine scale-up experiments were conducted. All of the TCLP mercury leachate concentrations were well below the performance criteria (0.025 mg/l). In the first two tests, it was discovered that when preparing the surrogate and blending in the reagents that a small portion of the added liquid mercury separated away from the spiked soil. For the third test, the elemental mercury was pre-mixed on aliquots of the soil in a high-impact mixer before addition to the treatment reactor. Liquid mercury and mercury as mercuric oxide and mercuric chloride were only added to the soil in the first three batch tests. Test 4 was performed on aliquots from the first two tests to investigate re-treatment of the soil to amalgamate the un-reacted elemental mercury still contained in these samples. The metallic mercury that separated from the soil in the first two batches was partially trapped in the crevices of the reactor and continued to supply fresh elemental mercury to the next two batch tests. After Test 4, the reactor was disassembled and thoroughly cleaned out. Batches 5 through 9 were tests to determine the best mixing regime to amalgamate all the liquid mercury in the soil. The following paragraphs provide more details on these scale-up tests.

Larger-scale testing began with a 10-kg batch in the rotary mixer “DeHg® reactor”. All of these tests used the Spike 3 surrogate materials. In previous DeHg® treatability studies, amalgamation was performed in a dry setting. The need to add a mercury spiking solution to the material in this study made it necessary to perform the amalgamation step on wet samples for the large-scale tests in this study. Additional water was added to improve mixing and provide the fluidity necessary with in-situ treatment. Enough water was added to raise the water content to 55 percent. Once the waste slurry was homogenized, elemental mercury was added to the material

drop-wise using a transfer pipette, and mixing was continued. This first attempt revealed that most of the elemental mercury settled in the bottom of the reactor, so although the TCLP value was passing at 0.009 mg/l, visible mercury was dispersed throughout the sample. It was thought that pockets of material from the sample caked the bottom of the reactor and trapped the elemental mercury that had settled out and, therefore, resisted treatment. The trapped material and visible elemental mercury were cleaned from the reactor before initiating another batch.

The order of addition of material was changed for the second test (15 kg). In order to prevent caking of the material on the sides and bottom of the reactor, excess water was introduced first. Dry material and aqueous mercuric oxide were added gradually in order to reduce the amount of caking within the reactor. Elemental mercury was added drop-wise using a transfer pipette after sufficient mixing was achieved. Once treatment was complete, the material was analyzed and inspected. Although the TCLP value was 0.003 mg/l, trace elemental mercury was visible within the treated soil. It was unclear if this mercury was from the spiked soil, or from trace residuals left within the crevices of the mixer. The reaction chamber was again cleaned.

The previous two batches revealed that the bulk elemental mercury addition was a problem. Attempts to evenly disperse elemental mercury throughout the material in these tests failed, resulting in small pools of elemental mercury that were not contacted by reagent. Therefore, a different approach was used to better disperse the Hg during treatment.

Prior to the addition of elemental mercury to the third batch, the elemental mercury was premixed in three separate containers (2-liter TCLP bottles) with 5.5 kg of the test soil and mixed on the T2F Shaker-Mixer (Turbula) for dispersion. Once the mercury and soil were mixed, the TCLP bottle bottom was tapped on the countertop in an attempt to make the mercury settle out of the mixture. This was then allowed to sit overnight and inspected in the next morning for elemental mercury. No elemental mercury was visible, thereby validating this method of preparing the surrogate soil matrix without observing visible elemental mercury.

The order of addition for this test was the same as the second test. Mercuric chloride was used with mercuric oxide due to running out of mercury oxide. The spiking solution contained 2.26 g of mercuric chloride and 0.51 g mercuric oxide. Premixed elemental mercury in the soil was added after all other components had been mixed in the DeHg® reactor (total weight of soil, bulk and premixed, was 21 kg). The TCLP of the stabilized material had a passing TCLP mercury concentration of 0.004 mg/l. Elemental mercury was only barely perceptible within the stabilized soil at “smear” levels. Because of the barely visible smears of mercury, it is not

known whether this mercury was from the premixed elemental mercury or if there was residual elemental mercury from the previous batches. No elemental mercury was observed on the bottom of the mixer after removing the treated material.

Test 4 was performed using most of the treated material from Tests 1 and 2. The objective of this test was to establish a protocol for re-treating the soil to amalgamate the small beads of elemental mercury remaining in this material. The TCLP leachate mercury concentration for this test was 0.003 ppm, however the treated material still contained very small beads of un-reacted elemental mercury.

Because of the concern for hold-up of elemental mercury in the reactor, the reactor was disassembled and thoroughly cleaned out. Disassembly of the reactor is difficult and time consuming, and usually not performed until a project is completed. Addressing the concerns of mercury hold-up raised in the first four tests in this project warranted taking this drastic step.

In order to demonstrate that all elemental mercury could be reacted with the DeHg® amalgamation reagent, material from the four preceding batches was combined and subdivided into five new batches. These five batches were returned to the rotary DeHg® reactor for supplemental amalgamation. After processing, grab samples were acquired from each batch and submitted for in-house mercury TCLP analysis. All batches passed TCLP. Grab samples were sent to an off-site laboratory for independent analysis.

Before sending Batch 8 for off-site analysis, it was noticed that it contained smear levels of elemental mercury. This batch was subsequently re-amalgamated, and the free mercury was eliminated. Grab samples of the original Batch 8 and the retreated Batch 8 (designated B8R) were both sent for off-site analysis.

The off-site analysis of Batch 5 gave a TCLP leachate mercury concentration of 0.11 ppm. On-site re-analysis of this material confirmed the failing TCLP results for this material. The Test 5 material was subsequently re-treated using both the amalgamation and stabilization protocols to ensure successful treatment. The TCLP leachate mercury concentration of the re-treated material was 0.002 ppm. Stability of this, and of the other scale-up test material was confirmed through on-going aging studies. The results of the aging studies to date, and the off-site and on-site analytical data for the scale-up tests are provided in **Section 6**.

5.6 DEVIATION AND MODIFICATION FROM THE WORKPLAN

Since the soil was analytically shown by Bechtel Jacobs Company (BJC) and IT to not be radiologically contaminated, the total uranium concentration was not measured in the soil.

The as received soil TCLP mercury concentration was similar to the UTS-UHC value of 0.025 mg/l. This is well below the contract performance criterion for mercury. The soil was therefore spiked with Hg^0 and mercury salts in order to increase the TCLP mercury concentration.

6.1 RECEIPT AND CHEMICAL, RADIOACTIVITY, GEOTECHNICAL CHARACTERIZATION

IT and NFS received their Y-12 Alpha 2 building soil samples on 12/20/2001. Pictures taken during the Alpha 2 building soil collection by BJC are in **Figures 6-1, 6-2, 6-3**. The samples were shipped in plastic lined 5-gallon buckets in overpacks. IT and NFS characterization results are in **Tables 6-1 and 6-2a/b**.

6.1.1 IT Characterization and Spiking Soil with Mercury

At IT, after opening the inner bucket in the hood, the activity was check using a Ludlum 3/44-9 pancake G-M detector. The results were at background levels. The shipping container was smeared for removable contamination. Smears were counted for gross alpha and beta activity. Smear results were below the facility contamination limits of 20-dpm alpha and 200-dpm beta/gamma. Samples of the soil were taken and analyzed by gross alpha, beta and gamma

Figures 6-1, 6-2, and 6-3
BJC Sampling Alpha 2 Building Soil



spectroscopy. Both the gross alpha and beta activities for the soil were below the detection limit. The total gamma activity was approximately 29 picoCuries per gram (pCi/g) with most of the activity from isotope K-40. K-40 is a common naturally occurring isotope. Based on the Bechtel Jacob's supplied radiological and the IT analytical results, the samples were treated as non-radiological materials. NFS was informed of the IT and BJC radiological analysis results.

The soil looked like a dry reddish silty clayey soil with clumps in it. It had less than seven percent water in it (See **Table 6-1**.) The material in the bucket consisted of mostly soil and small amounts of debris and a white flaky material. The as-received material had about 3.5 percent debris (material >0.5 inch) in it. The debris was mostly rocks. The white flaky substance and the clumps of dirt were easily broken by hand to less than 0.5 inch. Rocks were removed by sieving through a 0.5-inch sieve. After sieving to remove the debris, the soil was homogenized in a 7-gallon Hobart mixer. The soil consistency was such that all remaining soil clumps easily broke down to sand or smaller size particles within the first two minutes of mixing.

Table 6-1 Characterization Analyses		
Parameter	Method	Result
Percent Solids/Moisture	ASTM D2216	93.3. % Solid
Liquid Limits	ASTM D4318	Liquid Limit: 38% Wc (73% solid) Wc soil achieved a cake batter consistency: 55% Wc (65% solid)
Bulk Density	ASTM D5057	93.0 pcf (wet weight basis)
Mercury Vapor Concentration in Open 5-Gallon Bucket	Lumex Multifunctional Mercury Analyzer, Model-915+	>20,000 ng/m ³
Headspace Mercury Vapor Concentration (45 g in 150 cc specimen cup)	Lumex Multifunctional Mercury Analyzer, Model-915+	28,000-38,000 ng/m ³
pH (1 part waste to 10 parts water) ^{1,2}	Solid Waste (SW) 846 Method 9045B, modified	<ul style="list-style-type: none"> IT: 6.79 EnviroTest: 7.7
Total Hg ^{1,2}	SW 846 Methods 7471A	<ul style="list-style-type: none"> IT: 55.6 and 46.1 Average 50.8 mg/kg EnviroTest: 52.1 mg/kg See Table 6-2a for Total RCRA metal results.
Modified TCLP for 8 RCRA Metals ^{1,2}	SW 846 Methods 6010A & 7470A and Modified 1311	<ul style="list-style-type: none"> IT: Hg: 0.0299 and 0.0332 mg/l Average 0.0316 mg/l NFS: 0.025 mg/l EnvioTest: 0.016 mg/l See Tables 6-2a and 6-2b for remaining metals TCLP results from IT and NFS samples.

Table 6-1 Characterization Analyses		
Parameter	Method	Result
Modified TCLP for 1 st Hg Spike (1000 –1500 mg/kg Hg metal) 4	SW 846 Methods 7470A and Modified 1311	IT: 0.0324 and 0.0492 mg/l Average 0.0408 mg/l
Modified TCLP for 2 nd Hg Spike (1000-1500 mg/kg Hg metal, 13 mg/kg Hg from HgO) 3	SW 846 Methods 7470A and Modified 1311	<ul style="list-style-type: none"> IT: 0.0846 and 0.0717 mg/l Average 0.0782 mg/l NFS: 0.107 mg/l
IT Modified TCLP for 3 rd Hg Spike (5000-6300 mg/kg Hg metal, 100 mg/kg Hg from HgO)	SW 846 Methods 7470A and Modified 1311	IT: 2.115 and 1.965 mg/l Average 2.040 mg/l
NFS TCLP for 3 rd Hg Spike (5000 mg/kg Hg metal, 100 mg/kg Hg from HgO + HgCl ₂) 3, 5	SW 846 Methods 7470A and Modified 1311	<ul style="list-style-type: none"> NFS: 0.759 and 0.788 mg/l Average 0.774 mg/l EnviroTest: 0.856 mg/l EnviroTest Total Hg: 3870 mg/kg
Gross Alpha/Beta	Standard Operating Guideline (SOG) TALRAD0012	Gross Alpha <4.1E-5 uCi/g Gross Beta <4.6E-5 uCi/g
Gamma Spectroscopy	Equipment manufacturer's method	29 Pci/g

Note 1: EnviroTest is a commercial analytical laboratory used to validate NFS internal analytical results.

Note 2: "IT:" and "NFS:" preceding a value indicates that the values are from the respective company's internal laboratory analyses.

Note 3: NFS numbers were derived from bench-scale material (0.5 kg). EnviroTest assays were performed on grab samples taken during pilot testing.

Note 4: See Relevant Chemistry Section for more details on TCLP Hg values for Hg⁰ and HgO.

Note 5: For these tests, a solution containing the equivalent soluble mercury as dissolved HgO and HgCl₂ was added to the large batches and mixed in for approximately 15 minutes. A sample was then pulled for analysis. No drying was allowed to occur. Elemental mercury was then added, mixing continued for approximately 5 minutes and then a grab sample was taken.

Table 6-2a IT Total Metal and TCLP Results for Untreated Soil			
Metal	Total Metal (mg/kg)	TCLP Sample 1 (mg/l)	TCLP Sample 2 (mg/l)
Aluminum	NA	<0.023	<0.023
Antimony	NA	<0.0893	<0.0893
Arsenic	<3.50	<0.107	<0.107
Barium	52.04	<0.0058	0.233
Beryllium	NA	<0.005	<0.005
Cadmium	4.023	<0.0098	<0.0098
Calcium	NA	<0.104	>500
Chromium	20.58	<0.0214	<0.0214

Table 6-2a IT Total Metal and TCLP Results for Untreated Soil			
Metal	Total Metal (mg/kg)	TCLP Sample 1 (mg/l)	TCLP Sample 2 (mg/l)
Cobalt	NA	<0.0236	<0.0236
Copper	NA	0.016	0.0156
Iron	NA	<0.0116	<0.0116
Lead	25.92	<0.117	<0.117
Magnesium	NA	<0.113	20.2
Manganese	NA	<0.0174	3.27
Nickel	NA	<0.0227	<0.0227
Selenium	<5.54	<0.135	<0.135
Silver	<0.345	<0.0196	<0.0196
Sodium	NA	>250	>250
Thallium	NA	0.428	0.274
Vanadium	NA	<0.0258	<0.0258
Zinc	NA	0.041	0.023
NA – Not Analyzed			

Table 6-2b NFS TCLP Analyses of Untreated and Treated Soils Excluding Hg ¹								
TCLP Metals	Untreated Soil ² (mg/l)	Scale-up Treated Sample TCLP Results ³						UTS Limits (mg/l)
		B5 (mg/l)	B6 (mg/l)	B7 (mg/l)	B8 (mg/l)	B8R (mg/l)	B9 (mg/l)	
Arsenic	<0.006	<0.006	<0.006	<0.006	0.006	<0.006	<0.006	5.0
Barium	0.222	0.097	0.060	0.055	0.071	0.052	0.058	21
Cadmium	<0.007	<0.007	<0.007	<0.007	<0.007	<0.007	<0.007	0.11
Chromium	0.18	0.035	0.03	0.03	0.025	0.125	0.055	0.60
Lead	0.041	<0.004	<0.004	0.005	<0.004	<0.004	<0.004	0.75
Selenium	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	5.7
Silver	<0.01	<0.01	<0.01	0.015	<0.01	<0.01	<0.01	0.14
Note 1: Analytical results measured at EnviroTest.								
Note 2: All TCLP values, except the untreated soil, are the average of two samples.								
Note3: Samples B5, B6, B7, B8, B8R and B9 denote Batches 5 through 9, respectively. These batches are described in Section 5 . Samples from Batches 1-4 were not sent to EnviroTest for analysis.								

There were no visible mercury droplets in or on the soil. The mercury vapor concentrations in the headspace of the received 5-gallon bucket from the homogenized soil after approximately 10 minutes of exposure to air in the laboratory fume hood was >20,000 ng/m³. The mercury vapor concentrations in 150-cubic centimeters (cc) closed containers, which were approximately 1/3rd full of soil, ranged from 28,000 to 36,000 ng/m³. These latter concentrations are greater than the American Conference of Governmental Hygienists (ACGIH) Threshold Limit Values-Time Weighted Average (TLV-TWA) level for mercury (25,000 ng/m³).²⁰

Although the mercury vapor concentrations were high, the total and soluble mercury values were small. See **Table 6-1**. The TCLP mercury was well below the contractual performance criteria and was similar to the UTS criteria (0.025 mg/l). The TCLP mercury concentration was similar to the solubility limit of metallic mercury in water. The IT TCLP results for TAL metals excluding Hg are listed in **Table 6-2a**. All eight RCRA metals were below the TC regulatory criteria. Most of the metals were at least an order of magnitude less the regulatory criteria.

Since the TCLP mercury concentration was well below the contractual performance criteria, the soil was spiked at three different levels. The spiking procedure and rationale were described previously. As shown in **Table 6-1**, the TCLP values for the “as is” and the three spike levels were 0.032, 0.041, 0.079, 2.0 mg/l respectively. If all the added HgO had leached from the spiked soil, the TCLP values for Spikes 2 and 3 would have been about 0.65 mg/l and 5 mg/l instead of the measured values. It is speculated that the high-clay content in the soil adsorbed most of the soluble HgO in Spike 2. In Spike 3, the quantity of soluble Hg overwhelmed the clay adsorption capacity.

Full-scale in-situ mixing with an auger requires that the water content (geotechnical water content [Wc] – water/dry basis) be greater than the liquid limit. This minimum amount of water allows the blades to move through the soil, disperse the mercury drops, and mix in the reagents. The liquid limit for the soil was 38 percent water content. IT felt that this minimum amount of water did not make this high-clay content soil fluid enough to mix well. In all experiments at IT and all scale-up experiments at NFS, enough water was added to raise the Wc to 55 percent. This is equivalent to adding about 444 grams water to 1000 grams soil at about 6.7 percent solid contents. The water was added to the soil before any reagents.

6.1.2 NFS Characterization

NFS also completed characterization of the received materials. The total and TCLP Hg results for both IT and NFS were similar. See **Table 6-1**. The “as is” soil TCLP mercury was again well below the contractual performance criteria. The TCLP for the RCRA TC metals excluding Hg are presented in **Table 6-2b**. All 8 RCRA metals were below the TC regulatory criteria. Most of the metals were at least an order of magnitude less the regulatory criteria. **Table 6-2b** also lists the non-Hg TCLP metals for the scale-up experiments, Batches 5 through 9.

The TCLP Hg value for the Spike 2 was similar to IT's result. With Spike 3, the results differ. This may be due to NFS using a combination of HgCl_2 and HgO for the soluble spike. As shown in **Table 6-1**, the TCLP Hg values for this spike exceeded the contract performance criteria but is less than the IT result.

As described in the previous section, in all scale-up experiments at NFS, enough water was added to raise the W_c to 55 percent.

6.2 TREATMENT RESULTS

In the scoping experiments, bench-scale tests (≤ 1000 -g samples), both IT and NFS successfully determined treatment formulations that met both the project performance criteria and the UTS criteria for Spike Level 3 soils. During the scoping experiments, IT conducted experiments to demonstrate the affect of various ratios of reagent(s) and mercury contamination on the process. In order to show the affect the level of mercury contamination has on the treatment process, "as is" and soil spiked at Spike Levels 2 and 3 were used.

6.2.1 IT Results

6.2.1.1 TCLP Results

The IT results using one or two reagents are in **Tables 6-3, 6-4 and 6-5** and **Figures 6-4 and 6-5**, respectively. The IT results are ordered in **Table 6-5** and **Figure 6-4** by increasing Reagent A dosage. Similarly, **Table 6-4** and **Figure 6-5** are ordered on the increasing Reagent B then the increasing Reagent A dose. "Percent Dose Reagent (w/w)" in these tables is defined as the grams of reagent per 100 grams of homogenized soil or air-dried surrogate. The column labeled "Spike Level" in **Tables 6-3 and 6-4** define the mercury concentrations in the soils (i.e., "as is": no spike; Spike 2: 13 mg/kg Hg^{2+} /5000-6300 mg/kg metallic Hg; Spike 3; 100 mg/kg Hg^{2+} /5000-6300 mg/kg metallic Hg). In **Table 6-4** there are comments added to show where duplicates were run and where acid (sulfuric) or base (sodium hydroxide) or table salt (NaCl) was added to the soil before adding the reagents to determine if they would have an affect on the results. At the reagent dosages investigated, the addition of acid, base, or table salt had no affect on the mercury leaching results. **Table 6-5** contains the results from a factorial designed experiment. These experiments were conducted to show if variations expected in the field operation would cause the treated product to fail the TCLP. In the experiment, each reagent dosage was varied by ± 30 percent from a base case.

6.2.1.2 Hg Vapor

Addition of the treatment reagents lowered the vapor pressure of mercury from the soil. The mercury vapor concentrations decreased after the addition of Reagents A, B, or B then A from the initial close cup values (28,000-38,000 ng/m³) to less than 53 ng/m³ (when first open sealed container) and to below the instrument detection limit (20 ng/m³ at a 20 second average).

Table 6-3 IT Single Reagent Process–Scoping Experiment Results				
Mercury Spike Level ²	Percent Dose Reagent A ¹ (w/w)	Total Hg (mg/kg)	TCLP Hg <24 hr Cure (mg/l)	TCLP Hg After >30 day Cure (mg/l)
"As Is"	2.0	50.8	ND (0.0015)	0.00270
Spike 2	2.0	1850	0.0725	0.292
Spike 3	2.0	5160	0.130	0.0842
"As Is"	4.7	50.8	ND (0.0015)	0.00250
Spike 2	4.7	1820	0.0365	--
Spike 3	4.7	5140	0.869	0.00304
"As Is"	7.3	50.8	ND (0.0015)	0.00124
Spike 2	7.3	1940	0.00183	0.00178
Spike 3	7.3	5070	0.00170	0.00298
"As Is"	12.2	50.8	ND (1.5)	--

ND – Below detection limit. The detection limit value is in the ().

Note 1: Percent Dose Reagent equates to grams of reagent per 100 grams of soil.

Note 2: Spike Level corresponds to the 2nd and 3rd spiked surrogate (See Table 6-1).

Table 6-4 IT Dual Reagents Process–Scoping Experiment Results						
Mercury Spike Level ²	Percent Dose Reagent B ¹ (w/w)	Percent Dose Reagent A ¹ (w/w)	Total Hg (mg/kg)	TCLP Hg <24 hr Cure (mg/l)	TCLP Hg after >30 day Cure (mg/l)	Comment
Spike 3	0.4	2.7	5180	0.525	--	--
Spike 3	0.7	5.1	5140	0.0615	--	--
Spike 2	2.2	2.0	1370	0.00156	--	--
Spike 3	2.2	2.0	5140	0.432	2.040	--
Spike 2	3.3	2.6	1340	0.00150	0.117	--
Spike 3	3.3	2.6	5160	0.00846	0.165	--
Spike 2	3.3	3.9	1360	0.00170	0.00544	--
Spike 3	3.3	3.9	5170	0.297	0.157	--
Spike 2	6.4	2.9	1350	0.00164	0.0420	--
Spike 3	6.4	2.9	5110	0.0360	0.127	--
Spike 2	6.4	5.0	1340	0.00197	0.00256	--
Spike 3	6.4	5.0	5090	0.00765	0.303	--
Spike 2	9.6	4.3	1300	0.00150	0.00302	--

Table 6-4 IT Dual Reagents Process–Scoping Experiment Results						
Mercury Spike Level ²	Percent Dose Reagent B ¹ (w/w)	Percent Dose Reagent A ¹ (w/w)	Total Hg (mg/kg)	TCLP Hg <24 hr Cure (mg/l)	TCLP Hg after >30 day Cure (mg/l)	Comment
Spike 3	9.6	4.3	5050	2.000	0.0280	--
Spike 2	9.6	7.3	1280	ND (0.0015)	0.00224	--
Spike 3	9.6	7.3	6270	ND (0.0015)	--	--
Spike 3	9.6	7.3	6270	ND (0.0015)	--	Duplicate Preceding Smp
Spike 3	9.6	7.3	6270	ND (0.0015)	--	Make Soil Acidic (H ₂ SO ₄) to pH 4.5
Spike 3	9.6	7.3	6270	ND (0.0015)	--	Make Soil Alkaline (NaOH) to pH 9.0
Spike 3	9.6	7.3	6270	ND (0.0015)	--	Make Soil Alkaline (NaOH) to pH 9.0
Spike 3	9.6	7.3	5180	0.00318	0.00480	--
Spike 3	9.6	22.2	6270	0.0203	--	--
Spike 3	21.1	7.3	6270	ND (0.0015)	--	--
Spike 3	32.7	7.3	6270	ND (0.0015)	--	--
Spike 3	32.7	7.3	6270	ND (0.0015)	--	Make Soil Acidic (H ₂ SO ₄) to pH 4.5
Spike 3	32.7	7.3	6270	ND (0.0015)	--	Add NaCl at approximately 1830 mg NaCl per kg soil
Spike 3	32.7	7.3	6270	0.00218	--	--
Spike 3	32.7	22.2	6270	0.00689	--	--

ND – Below detection limit. The detection limit value is in the ().

Note 1: Percent Dose Reagent equates to grams of reagent per 100 grams of soil.

Note 2: Spike Level corresponds to the 2nd and 3rd spiked surrogate (See Table 6-1).

Table 6-5 Affect Of Reagent Dose Variability on a Base Case Successful Formulation Using IT Dual Reagents Process–IT Scoping Experiment Results					
Percent Dose Reagent B ¹ (w/w)	Percent Dose Reagent A ¹ (w/w)	Reagent B Factorial Level	Reagent A Factorial Level	Spike Level ²	TCLP Hg <24 hr Cure (mg/l)
3.3	2.6	Base Case		2	ND (0.0015)
3.3	2.6	Base Case		3	0.0039
2.33	1.79	-1	-1	2	0.0038
2.33	1.79	-1	-1	3	0.0324
2.33	3.32	-1	1	2	ND (0.0015)
2.33	3.32	-1	1	3	0.0023

Table 6-5 Affect Of Reagent Dose Variability on a Base Case Successful Formulation Using IT Dual Reagents Process–IT Scoping Experiment Results					
Percent Dose Reagent B ¹ (w/w)	Percent Dose Reagent A ¹ (w/w)	Reagent B Factorial Level	Reagent A Factorial Level	Spike Level ²	TCLP Hg <24 hr Cure (mg/l)
4.33	1.79	1	-1	2	0.0017
4.33	1.79	1	-1	3	0.0326
4.33	3.32	1	1	2	0.0019
4.33	3.32	1	1	3	0.0033
ND – Below detection limit. The detection limit value is in the (). Note 1: Percent Dose Reagent equates to grams of reagent per 100 grams of soil. Note 2: Spike Level corresponds to the 2 nd and 3 rd spiked surrogate (See Table 6-1).					

Figure 6-4. IT: Single Reagent Addition: MTCLP Hg, Results Sorted by Spike Level, then by Reagent A

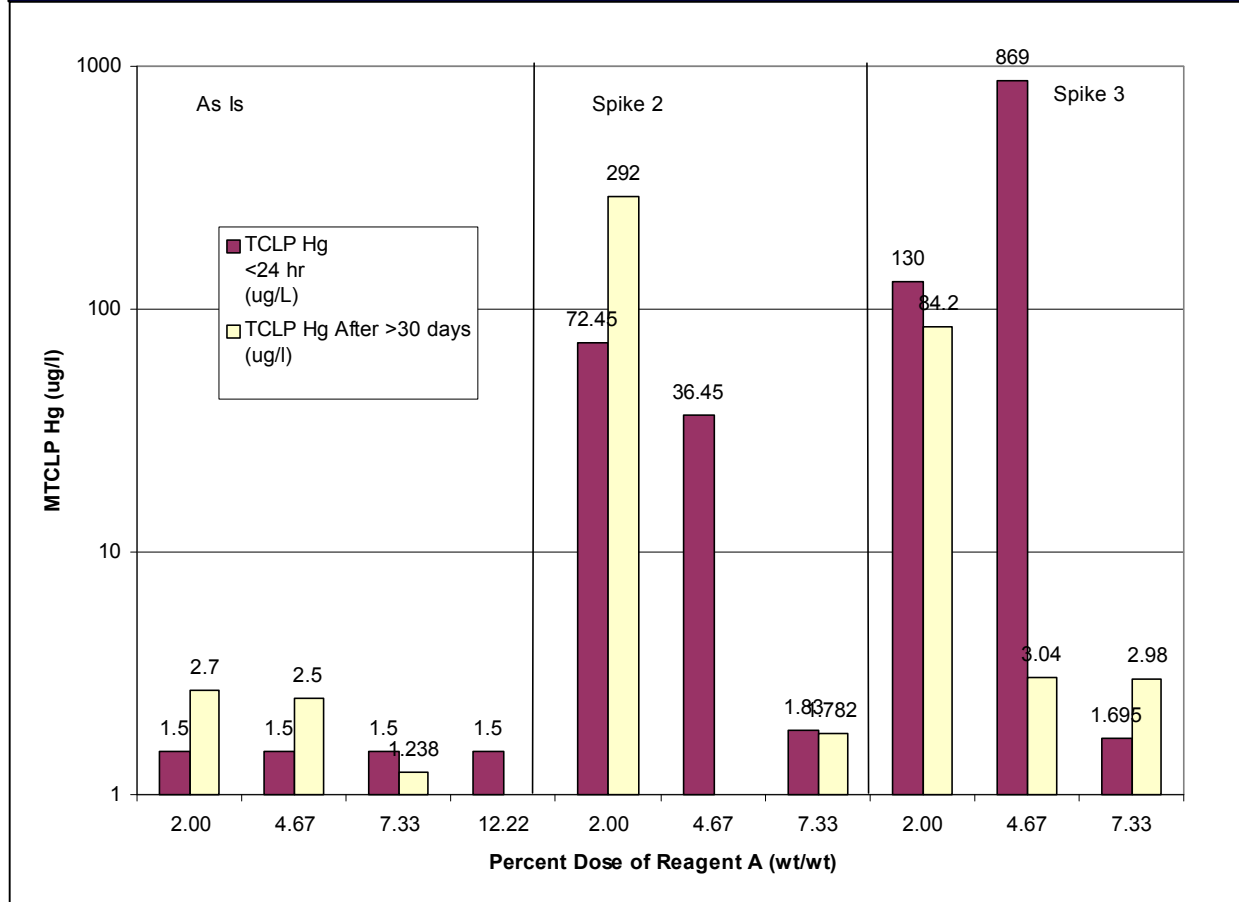
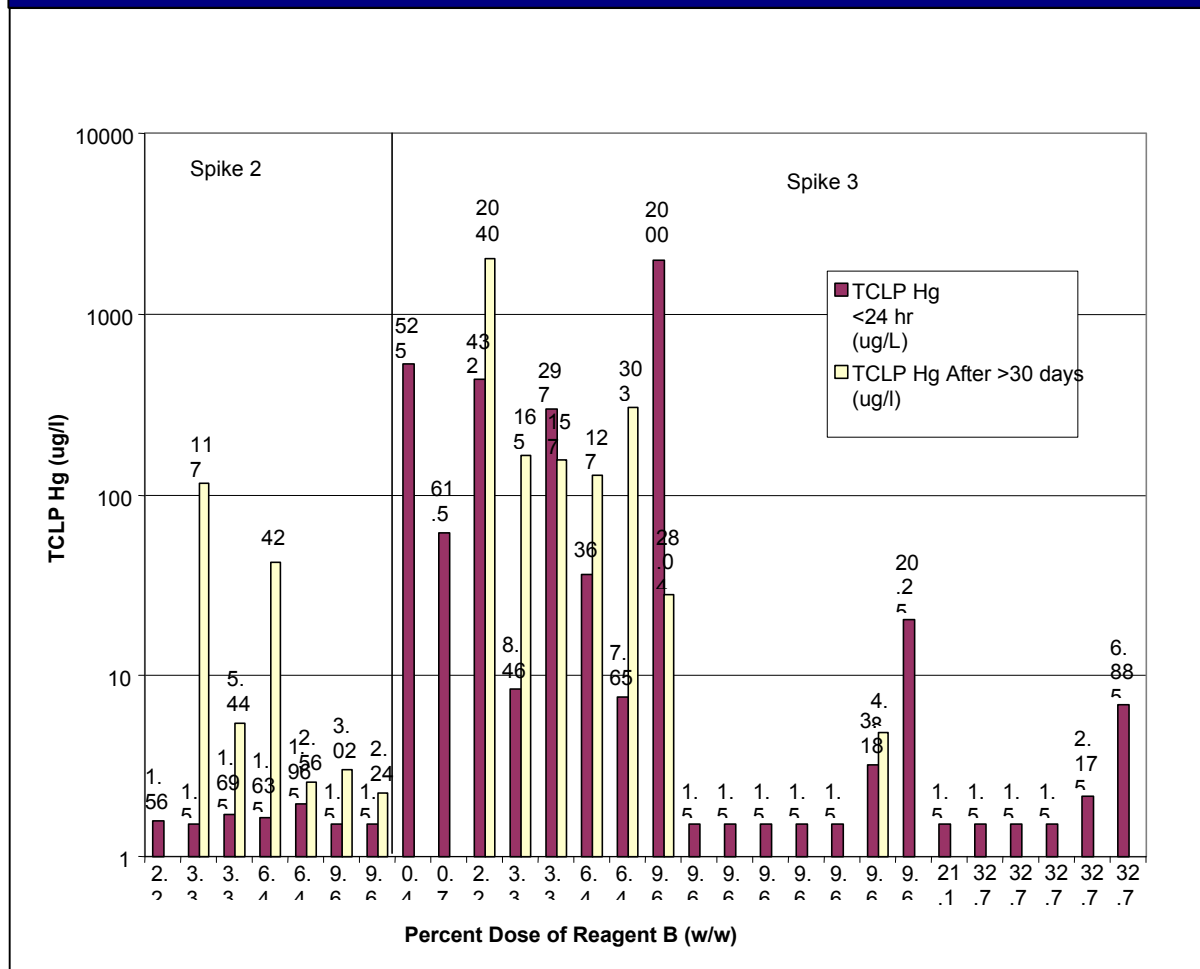


Figure 6-5.
IT Dual Regent Process- MTCLP Hg Results, Sorted by Spike Level, then by Reagent B



6.2.2 NFS Results

The TCLP results for NFS scoping and scaled-up demonstration are in **Tables 5-1, 6-6 and 6-7**. **Table 6-6** provides the internal TCLP results for Scale-up Tests 1 - 4. **Table 6-7** provides both the internal NFS and EnviroTest results. Cold Vapor Atomic Absorption (CVAA) analysis for mercury in TCLP fluid is the currently accepted method. Inductively-Coupled Plasma Mass Spectroscopy (ICP-MS) numbers are used for comparison. Total Hg measures the amount of elemental mercury contained within the soil. TCLP Hg is the amount of mercury that leaches out of the matrix and into an acidic aqueous solution.

TABLE 6-6 NFS INTERNAL ANALYTICAL RESULTS FOR SCOPING TESTS 1-4		
Test Number	TCLP [Hg], ppm	TCLP Leachate pH
1	0.009	8.92
2	0.003	9.88
3	0.004	9.62
4	0.004	9.48

The scoping test results (**Table 5-1**) were all below the performance criteria except for the initial test. Based on the results from the NFS scoping experiments, NFS scaled-up the successful formulation from the blender to the pugmill (approximately 10 to 20 kg soil). The NFS TCLP Hg values from all final product analyses were less than 0.025 mg/l which is smaller than the contract performance criteria (2.0 mg/l) and the UTS criteria. Grab samples were also sent out to the EnviroTest Analytical Laboratory for independent TCLP analyses. The in-house and EnviroTest TCLP results were similar except for Batch 5 and 8. The repeats of Batches 5 and 8 (i.e., 5R, 8R) gave similar results to the original NFS results.

In Batches 5 through 9 the moisture content and the intensity of mixing were varied to ascertain their affect on the process. It is believed the differences between the analyses for Batches 5 and 8 are the result of incomplete mixing of the amalgamation reagent into the soil. After a second treatment (Batches 5R and 8R), both materials TCLP were less than 0.004 mg/l mercury. The same result could have been obtained in one amalgamation step when the samples were better mixed (i.e. note the other batches 6, 7, and 9).

Table 6-7 NFS DeMerc® Process – Scale-Up Results							
Lab and Type of Assay	Batch 5	Batch 5R	Batch 6	Batch 7	Batch 8	Batch 8R	Batch 9
NFS –TCLP (mg/l)	0.004	0.002	0.002	0.002	0.007	0.001	0.001
EnviroTest –ICP-MS TCLP (mg/l)	0.121	N/A	< 0.006	< 0.006	0.096	< 0.006	< 0.006
EnviroTest – Cold Vapor TCLP (mg/l)	0.110	N/A	0.004	0.003	0.069	0.001	0.005
EnviroTest – Total Hg (ppm)	1,520	N/A	807	1,300	1,225	808	1,385
PH	10.4	9.44	10.5	10.4	10.4	10.5	10.5
N/A Not Analyzed at EnviroTest Laboratory							

As indicated previously (See **Section 5**) due to problems of mercury dispersion within the soil, all material was subjected to a second amalgamation step. This increased the volume of the material, and thereby diluted the original concentration of mercury from about 5000 to 1000 mg/kg. This dilution is described in more details in the following paragraphs.

The difficulty of blending the soil sample to obtain a homogenous dispersion of spiked elemental mercury and the difficulty of effectively mixing reagents into the soil for treatment of the mercury made it necessary to reuse the limited weight of soil sample provided for the demonstration several times. **Table 6-8 and Figure 6-6** provide a detailed analysis of the flow of the soil sample through testing. The mercury concentrations given are calculated values, not analytical results. They were derived by using the known inputs of soil, water, reagents, and added mercury. The purpose of these mercury numbers is to demonstrate accountability for all of the mercury input into treatment. This was accomplished by demonstrating that the calculated mercury concentrations correlate well with the measured mercury levels from the samples sent for off-site confirmatory analyses. **Table 6-7** results are related to **Table 6-8 and Figure 6-6**, by the following:

- Mass Balance No. 11 is the composite of Batches 1, 2, 3
- Mass Balance No. 16 is Batch 4
- Mass Balance No. 22 is the composite of Batches 5 through 9
- Mass Balance No. 27 is the composite of Batches 5R and 8R.

The 46.4 kg of untreated soil used for the large-scale tests (Mass Balance No. 7) was spiked to 5100-ppm total mercury by the addition of a total of 236.3 g of mercury in the three tests. The resulting residues from these tests had total mercury concentrations of about 3600 ppm on a dry basis and 2500-ppm on an “as is” wet basis (Mass Balance No. 11). As re-tests were required to develop the most effective treatment parameters, the addition of reagents and water roughly quadrupled the final total weight of the residues (Mass Balance No. 29).

The calculated value of 1219-ppm total mercury (Mass Balance No. 29) for the “as is” composite material after treatment is in very close agreement to the average value of 1175 ppm total mercury for the five samples sent for off-site analysis (only 4% higher). This is well within the typical uncertainty for the analysis of total mercury in samples containing these levels of mercury (10 – 15%).

It is important to note that these final residues contain greater than 40% by weight water. This will not be the case in full-scale treatment. It is expected that the proposed full-scale treatment process will produce residues containing about 20% water by weight. There will also not be the need for the same degree of reagent usage on the proposed single-pass treatment. Based on the interpretation of the results of the treatability tests by NFS, reagent loading to treat this waste in an appropriate mixer will only add about 25 to 45% by weight reagents to the final treated waste.

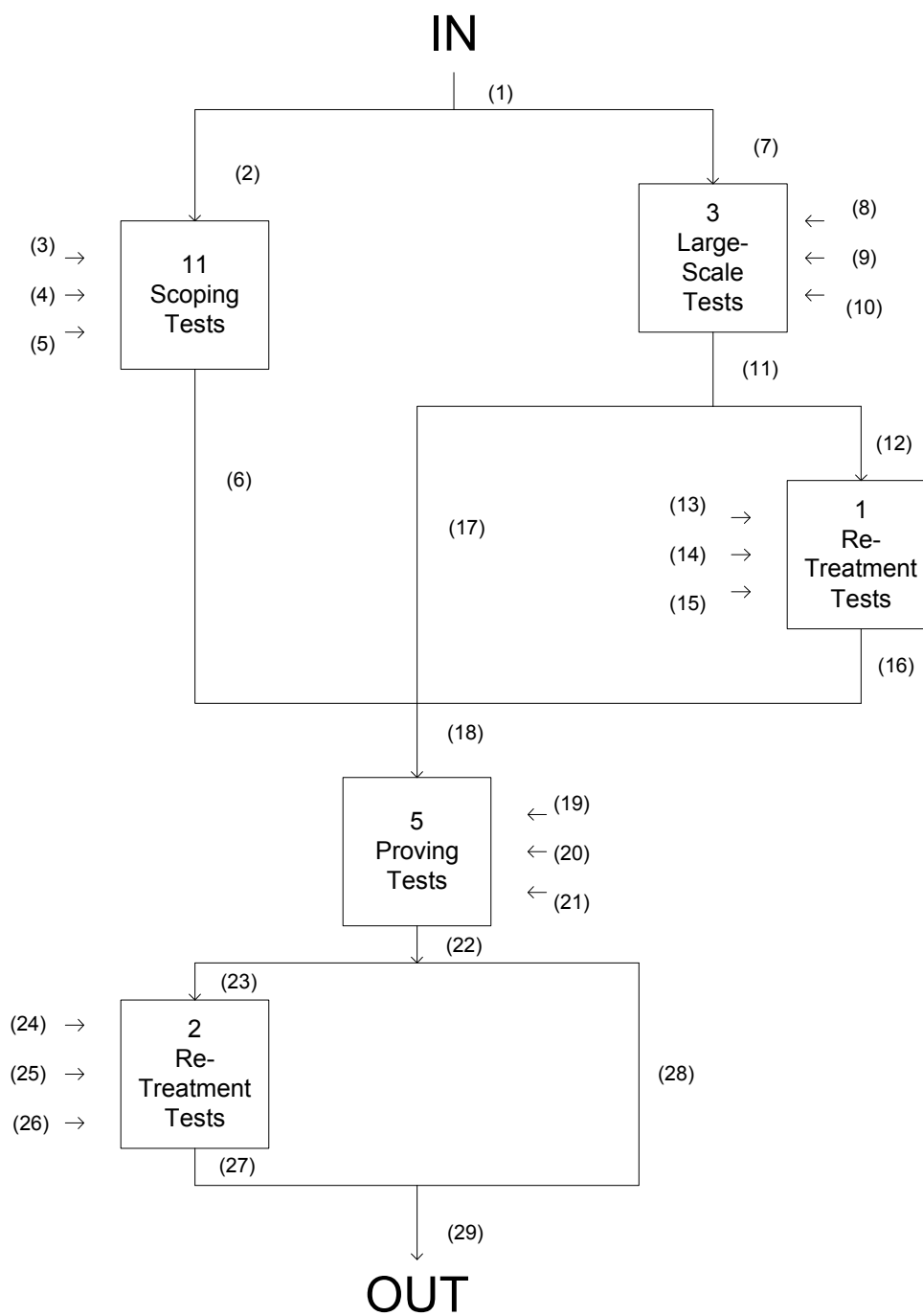


FIGURE 6-6: NFS MASS FLOW DIAGRAM

Table 6-8 Mass Balance for Treatability Sample Testing Flow Diagram (Figure 6-6)										
ID Number	1	2	3	4	5	6	7	8	9	10
Untreated Sample, kg	51.3	4.9					46.4			
Treated Sample, kg (dry)						6.9				
Treated Sample, kg (wet)						12.4				
Percent Solids						55.6				
[Hg], ppm (dry basis)						2986				
[Hg], ppm (wet basis)						1661				
Water Added, kg			5.5					28.5		
Reagents Added, kg				2.0					19.1	
Hg Added, g					20.6					236.3
ID Number	11	12	13	14	15	16	17	18	19	20
Untreated Sample, kg										
Treated Sample, kg (dry)	65.7	15.5				22.9	50.2	80.0		
Treated Sample, kg (wet)	94.3	22.3				41.6	72.0	126.0		
Percent Solids	69.7	69.7				55.0	69.7	63.5		
[Hg], ppm (dry basis)	3597	3597				2435	3597	3211		
[Hg], ppm (wet basis)	2506	2506				1343	2506	2039		
Water Added, kg			12.0						31.5	
Reagents Added, kg				7.4						30.0
Hg Added, g					0.0					
ID Number	21	22	23	24	25	26	27	28	29	
Untreated Sample, kg										
Treated Sample, kg (dry)			110.0	31.2				41.5	78.8	120.3
Treated Sample, kg (wet)			187.5	53.1				76.3	134.4	210.7
Percent Solids			58.7	58.7				54.4	58.7	57.1
[Hg], ppm (dry basis)			2335					1755		2135
[Hg], ppm (wet basis)			1370					953		1219
Water Added, kg					13.0					
Reagents Added, kg						10.2				
Hg Added, g		0.0					0.0			

NFS maintains an ongoing program in which samples from all treatability studies are archived and tested for long-term stability. Currently, there are samples in this program that have been under evaluation for four years. Samples for the Y-12 treatability study have also been retained for evaluation in this program. **Figure 6-7** provides a graphical display of the performance of the Y-12 material over the three-month time period since its initial treatment. For comparative purposes, the graph also provides aging stability data for samples from the MER03 and MER04 treatability studies. These programs were selected for comparison because, like the Y-12 sample, they were also soils or soil-like surrogates containing elemental mercury at similar

levels. Samples from the MER03 and MER04 studies have been in the testing program for 2 years and 1 year respectively.

NFS has drawn a couple of conclusions from tracking the stability of treated soils that contained elemental mercury in the initial waste form:

1. Soils in which all of the elemental mercury is not completely amalgamated are not stable over time with regards to TCLP leachability. A final waste form that contains visible un-amalgamated elemental mercury should not be land disposed even if it initially passes TCLP criteria. Test 8 for this study provides an example. The initial internal TCLP for this test's material was 0.007 ppm. When the sample was sent for off-site analysis, it was known that it contained small beads of un-amalgamated mercury. The material from this test was re-amalgamated, and the sample from the re-treatment submitted for off-site analysis at the same time as the original Test 8 sample. The initial internal TCLP for the Test 8R sample was 0.001 ppm. In the few weeks that elapsed while the samples were in queue for off-site analysis, the Test 8 sample's TCLP increased to nearly 0.1 ppm. However, the Test 8R TCLP remained stable at 0.001 ppm.
2. There is a common trend for correctly treated samples in which the TCLP mercury leachate concentrations rise from the initial low levels to levels sometimes approaching 40 – 50 ppb. Then the TCLP mercury concentrations return to their original, and oftentimes lower values. This equilibration period typically occurs within the first 90 – 120 days post-treatment for materials treated using the NFS process. **Figure 6-2** provides an excellent visual depiction of this trend for the three mercury-in-soils studies.

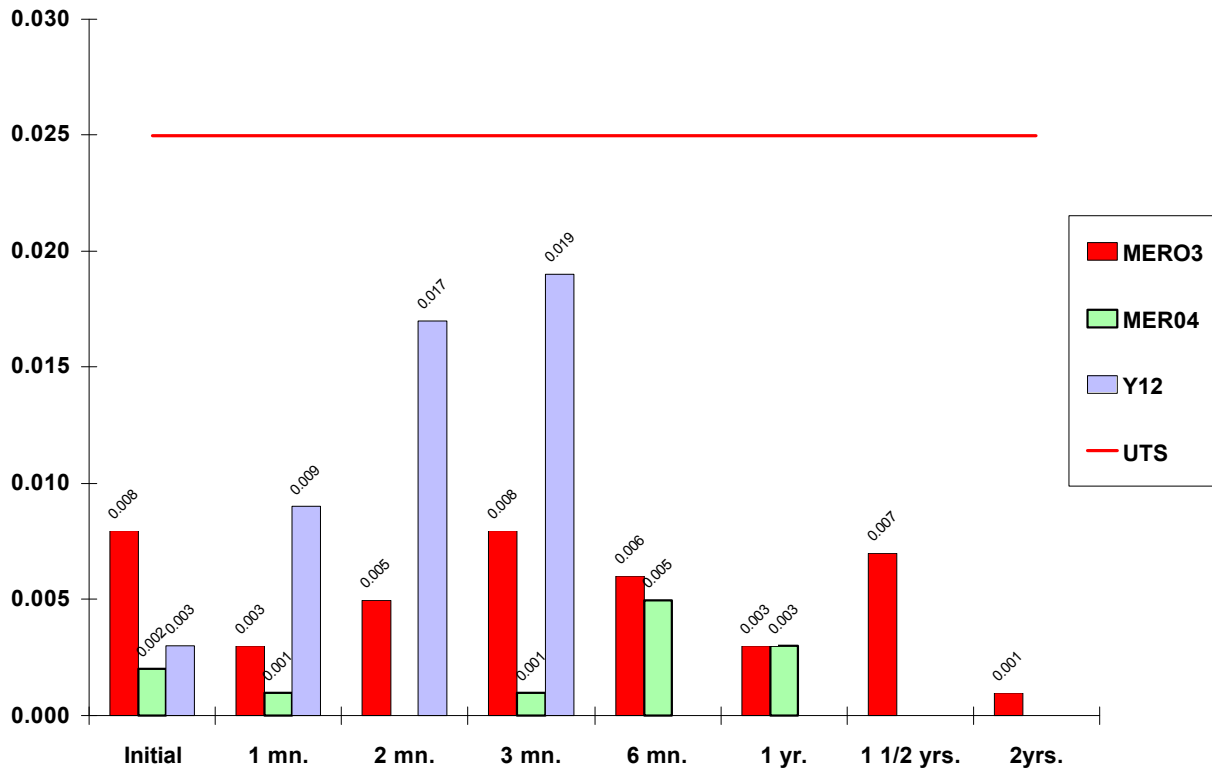


Figure 6-7 – Aging Studies for the Y-12, MER03, AND MER04 PROJECTS

6.3 DISCUSSION OF RESULTS

Both companies successfully used amalgamation and fixation to treat the mercury-spiked soil. Their proprietary reagents have similarities and differences. Although the chemical reagents were different, both companies were successful at meeting the contract performance criteria and meeting the LDR TCLP criteria. The NFS process uses multiple reagents while the IT process uses either one or two reagents.

6.3.1 Discussion of IT Results

Three different levels of mercury contamination were investigated in the single reagent evaluation. “As is” soil and two surrogates (Spike 2 and Spike 3) were investigated to address the range of expected mercury contamination at the site. The following conclusion may be made from analysis of the results in **Table 6-3** and **Figure 6-4**.

- All treatment dosages met the performance criteria.
- As the untreated TCLP Hg value increased, more Reagent A was needed to lower the treated TCLP Hg value to below the UTS level (0.025 mg/l) value.
- When the Reagent A dosage was 7.33 grams Reagent A per 100 gram soil, there was no significant aging affect.

Two surrogates (Spike 2 and Spike 3) were investigated in the dual reagent evaluation. The “as is” homogenized soil was not treated in this part of the study. The following conclusion may be made from analysis of the results in **Table 6-4** and **Figure 6-5**.

- Most treatment dosages met the contractual performance criteria.
- Changing the total amount of reagents added and the ratios of the reagent dosages affected the leachability of mercury.
- Generally increasing the dosage of Reagent B lowered the TCLP Hg value. The variations within this trend were due to varying the dosage of Reagent A.
- In order to address potential environmental issues, the pH of the surrogate was change to pH 4.5 or 9 by addition of sulfuric acid or sodium hydroxide before addition of Reagent B. Changing the pH of the sample prior to treatment had no affect on the efficiency of the treatment. Also, sodium chloride was added to one surrogate at about four times the molar concentration of total mercury. At the dosage investigated, there was no impact of the salt addition.
- When the Percent Reagent B and A dosages were 9.6 and 7.3, respectively, there was no significant aging affect for either the Spike 2 or Spike 3 surrogates.
- At the same Percent Reagent Dosage (i.e., 9.6/7.3), both TCLP Hg concentrations were decreased below the UTS criteria for both the Spike 2 and Spike 3 surrogates.

A statistical factorial design experiment was conducted to determine if variations in reagent dosages that could be expected in the field operation would cause the treated product to fail the TCLP. A base case was selected where the TCLP Hg value was less than the UTS criteria. The base case formulation had 3.3 and 2.6 percent dose for Reagents B and A, respectively. The reagent dosages were varied by ± 30 percent from a base case. A higher and lower concentration of total mercury concentrations was superimposed on the 2^2 factorial for Reagents A and B to yield eight (2^3) experiments plus center points. The higher and lower mercury concentrations correspond to Spike 2 and 3 surrogates. **Table 6-5** contains the results from this experiment. Analysis of the results show that at the lower total Hg concentration, all TCLP values remain approximately constant, i.e., less than 0.004 mg/l. At the higher mercury concentration, the

TCLP Hg increased when the Reagent A concentration was decreased to its lower value. The TCLP Hg concentrations were approximately 0.034 mg/l. These TCLP Hg concentrations are still well below the performance criteria. This set of experiments shows that reasonable variations in the ratio of Reagent A and B from the base case formulation can be tolerated to yield products that consistently meet the performance criteria.

Analyzing the combined results from **Tables 6-3** and **6-3** shows that the IT process is robust for treating elemental and ionic mercury compounds in high clay content soils. IT showed that for a wide range of mercury contamination (i.e., 50 mg/kg to 6300 mg/kg mercury) the treatment formulation maintained the TCLP mercury concentration below the LDR criteria. The TCLP met this criteria at both one and then at greater than 30 days cure. In addition, it was shown that the IT process is not excessively sensitive to variations that may be seen in actual production. When the relative ratios of reagents were varied ($\pm 30\%$ from the base case values), the process chemistry maintained the TCLP mercury well below the contract performance criteria and normally well below the LDR value.

The treatment process, not only lowered the impact on human, health, and the environment by making the mercury less leachable, the process significantly lowered the potential for exposure to the mercury vapors. In the experiments where Hg vapors were measured in the sample container's headspace, over 99.8 percent (worst case) of the mercury vapor was arrested by the treatment. This worst-case mercury vapor concentration is 0.1 and 0.2 percent of the current National Institute for Occupational Safety and Health (NIOSH) Recommended Exposure Limit (REL) and ACGIH TLV-TWA values, respectively. The calculation of the worst case uses the largest Hg vapor headspace concentration for the successful treated samples (49 ng/m³). In addition, using the headspace analysis simulates the mercury concentration in a confined area. In a more open environment, the Hg vapor concentration would be less.

Based on the preceding discussions, the Hg vapor concentrations will not be an issue in an enclosed or non-enclosed lay down areas for the treated soil stockpiles. Most likely, there will not be a regulatory issue with the Hg vapor concentration for the untreated soil in a non-enclosed laydown area. The measured mercury vapor concentrations were less than the PEL and REL values. However, in IT's closed cap test of the untreated soil, the Hg vapor concentrations exceeded the ACGIH TLV-TWA. Also, when BJC personnel collected soil from the Alpha 2 building basement, the Hg concentrations were above the sites acceptable Hg concentration. As shown in **Figure 6-1 – 6-3**, the BJC personnel wore respirators. Thus, in an enclosed area, the Hg vapor concentration of the untreated soil may be an issue. IT/NFS proposes to measure the Hg vapor concentrations during the Phase 2 project to better define this issue.

6.3.2 Discussion of NFS Results

There are several conclusions that may be drawn from the NFS study. They are as follows.

- Over 50 kg of as-received soil spiked to ~5000 mg/kg elemental mercury and ~100 mg/kg soluble, ionic mercury were treated to produce residues having an average TCLP leachate mercury concentration of 0.002 ppm (ranging from 0.001 to 0.002 ppm over the five samples analyzed). There was no visible, un-amalgamated mercury in these treated materials. On-going aging studies confirmed the stability of the treated materials over the 90-day post-treatment time period. The residues from this treatment are following a typical aging profile for elemental mercury bearing soils treated using the NFS process.
- Experience from treatability studies performed on other elemental mercury-bearing soils has shown that mercury vapors from the treatment of these types of soils should not be an issue. The greatest release of mercury vapor is expected to be during pre-treatment handling of the contaminated soils. Mercury vapor has been shown to decrease to nearly non-detectable levels following amalgamation by the NFS process.
- The clayey nature of the sample tested provided a challenge to the mixing equipment available for the large-scale testing performed at NFS. Excellent treatment results were achieved, but unusually long mixing times and reagent loadings were required. However, excellent results were achieved in the two scoping tests performed using a high-shear blender. The elemental mercury and reagents were thoroughly dispersed throughout the sample in reasonable mixing times.
- The NFS reagents have previously been shown to successfully treat many types of waste, (e.g., debris, ion exchange resins, and sandy soils). The process has consistently met the UTS requirements (i.e., TCLP Hg less than 0.025 mg/l.) It is key to realize that this current study shows that the NFS chemistry and process have now been proven successful on soils with high clay contents.

The NFS scale-up demonstration tests provide insight into a potential full-scale issue. The DeHg® reactor operators observed small smears of liquid mercury on the bottom of the mixing chamber during the first four scale-up tests. This observation confirms that separation of larger drops of liquid Hg from soil is a potential design issue for the full-scale system.

The detection of free elemental mercury within the stabilized matrix was unusual because it had not occurred in prior NFS scale-up demonstration work performed in MER03 and MER04. The soils from MER03 and MER04 contained elemental mercury levels that were very similar to those spiked into the Y-12 surrogate soils. Only a single amalgamation was required in each of the MER demonstrations. The IT/NFS team will address the challenge of complete and intimate blending of elemental mercury-bearing claylike soils with amalgamating reagents through both the proper selection of mixing equipment design, and the appropriate strategy for managing elemental mercury pockets that exist within raw Y-12 soils. With this approach, as well as with the experience of the IT/NFS team, it is envisioned that a single amalgamation followed by a single stabilization step will suffice for the proposed pilot study at the Y-12 site. The envisioned approach will be robust enough to address fluctuations in both total elemental mercury and total soluble mercury content. This is possible because of the ability to monitor the progress of the NFS process chemistry and only add levels of reagents needed to effectively treat the amount of mercury contained in each treatment batch.

The efficiency at which any mixing process will incorporate liquid mercury into the stabilized material depends on the size of the Hg droplets and the intensity (shear) in the mixing process. The 25-50 mg drop size used in the IT bench-scale tests were readily dispersed by hand mixing or in the Hobart ® planetary mixer. For the NFS 21 kg tests, the liquid mercury was added to the soil in slugs of 1-2 cc for the first two tests and mixed in a Turbula three-dimensional mixer for the third test. It is believed that, with the high-clay content soil, this mixer did not have sufficient shear to completely disperse the Hg droplets when the mercury was added directly to the large-scale reactor. Although the spiked soil was visually inspected for the Hg drops, experience shows that the small drops would have been covered with soil fines and virtually impossible to detect. When this spiked soil was added to the NFS's low-shear, low rpm, DeHg® reactor, a small portion of the liquid Hg was smeared on the mixer wall. This smearing process may be visualized as spreading a clump of mayonnaise (Hg analog) on a piece of bread with a flat flexible knife. However, this phenomenon was minimized in the third test where the elemental mercury was pre-blended in the Turbula mixer prior to addition to the large-scale reactor. Collection and treatment of this "free" Hg must be addressed by the operation plans for the treatment process.

Based on the IT (easy dispersion of Hg) and NFS (slight smearing of Hg) results, IT expects that when there are more significant sized pools of liquid mercury in the soil, up to 10 percent of the mass may separate away from the bulk of the soil during operations. This will likely occur to some extent with both the in-situ and ex-situ operations. The large-diameter horizontally

rotating auger, in the in-situ process, provides higher shear and more intense mixing than the pugger did in the Scale-up tests. The in-situ auger should break-up most of the larger Hg beads and laterally disperse and mix them with the reagents. It is likely, however, that some amount of the liquid Hg may work its way downwards during the mixing operation. During the excavation phase of project following the amalgamation/fixation, the field crew will be visually looking for liquid mercury. When Hg is observed, a mercury vacuum will be lowered down to the area of contamination and the liquid Hg collected. The liquid mercury may be treated using a small mobile treatment unit or sent to the Broad Spectrum Contractor. The IT/NFS price estimate assumes that liquid Hg will be treated using the mobile unit utilizing NFS chemistry. In the ex-situ operation, some liquid Hg will probably separate from the soil during the excavation and sieving operation before the pugmill. When there are obvious pools of liquid Hg forming during the excavation, the field crews will collect the liquid Hg using the same equipment described for the in-situ process. The laydown area under the treatment unit will be a sloped concrete pad with a sump in it. The concrete pad will be sealed to keep any mercury from adsorbing into the concrete. Any liquid mercury falling on this pad will be collected with a mercury vacuum. The pugmill will be a dual shaft high shear mixer that will disperse the mercury and mix it with the treatment reagents. The pugmill will be inspected periodically to determine if there is any liquid mercury in it. This mercury will also be collected with a mercury vacuum. After enough Hg is collected from all sources, it will be treated to meet the performance criteria.

Successful bench-scale treatments were effected in this study. All demonstration- and full-scale descriptions are based on the results from the bench-scale study. The process and process chemistry can be scaled-up to treat soil at the demonstration- and at the full-scale process. The process can be implemented, at commercial scale, by both in-situ and ex-situ techniques. In either case, reagent mass ratios (reagent to initial untreated soil) and reagent addition order will be the same as for the bench-scale tests. Reagent mass ratio may, in some cases, be increased to account for variations in the efficient of mixing at the full-scale. With minor modification, the ex-situ process may also be applied to non-soil matrices. Since a batch process was analyzed in this project, the results are readily scaleable to larger batch sizes for the optional demonstration phase project.

As initially defined in the SOW, the demonstration phase was to be conducted in-situ. After NETL, DOE, and BJC personnel visited both IT and NFS facilities, they recommended that both in-situ and ex-situ processes should be considered for the field-demonstration phase. It was also recommended that pricing for both the in-situ and ex-situ option using both IT and NFS process chemistries be supplied in the report. In order for the various vendors prices to be consistently compared to the baseline technology, NETL and BJC personnel provided directions on limiting what items were to be included in the price estimate. The following boundary conditions were placed on the treatment processes in the cost estimates.

- Both in-situ and ex-situ treatments. The total quantity of soil to be treated is 50,000 cy. Define the process with only one treatment location since it is not known how many different sites will be treated.
- In-situ treatment: DOE will define the location for treatment. IT/NFS is to excavate the soil after treatment and place it in a pile for DOE to transport to the EMWMF.
- Ex-situ: The pricing is based on a pile to pile process. IT/NFS was to assume that DOE will provide a pile of contaminated soil next to the treatment process. IT/NFS will transport the soil to and from the treatment operation and place it in another pile for DOE to transport to the EMWMF.

We propose a treatment process using commercially available equipment slightly modified for site-specific details. This allows an easier scale-up from the bench, through field demonstration testing, and into full-scale waste treatment operations. While the waste is complex, our treatment approach is straightforward and geared specifically toward full-scale operations.

7.1 IN-SITU versus EX-SITU OPERATIONS

Operationally, stabilization of soil or waste materials can be employed in place (in-situ) or the soil or waste can be excavated and transported to a central treatment plant (ex-situ).

In-situ treatment methods treat the contaminated material in place by mixing reagents directly into the soil or waste matrix. The area of contaminated material to be treated is surveyed to determine the depth of contaminated material. Based on the density and volume of the contaminated material, the amount of contaminated material is determined. The treatment formulation, determined by treatability testing, is then used to determine the amount of reagent to be added to the volume of contaminated material. The reagents can be pneumatically conveyed onto the area to be treated, water added, slurried and pumped onto/into the contaminated material as it is being mixed. As will be discussed, the choice of how the reagent is added depends largely on the type of mixing equipment employed and the level of dust emissions permitted.

The reagents can be mixed in-situ into the contaminated material using augers, rototillers, excavations, and tillers. In this project, augers are proposed for the in-situ process.

For ex-situ stabilization, the contaminated material is excavated and transported to a central treatment unit. Often, the contaminated material is screened to remove debris and oversized (>2 inch) material and stockpiled to await treatment. The contaminated material is fed into a feed hopper that places the contaminated material on conveyors for transfer to the mixing chamber. These conveyors often have belt scales to determine the amount of contaminated material entering the mixing chamber. The treatment formulation, determined by treatability testing, is then used to determine the amount of reagent to be added to the amount of contaminated material. The reagents can be conveyed into the mixing chamber or slurried and pumped. The choice of how the reagent is added depends largely on the type of mixing equipment employed and the level of dust emissions permitted. Mixing chambers employed for ex-situ stabilization includes pugmills, mullers, paddle extruders, mixing screws, and mixing pits. In this project, a pugmill is the recommended mixing unit. After treatment the treated material is conveyed from the mixing chamber to a stockpile or transported to its final destination.

The factors which influence the choice of in-situ or ex-situ treatment include, but are not limited to:

- Physical properties of the waste material (including the volatility of Hg);
- Presence of debris;
- Regulatory impacts of excavation and stockpiling;
- Treatment criteria; dust and odor emissions;
- Cost; and
- Public and regulatory acceptance.

The physical properties that favor in-situ treatment methods include high moisture content, low interparticle cohesion, and low clay content soil or easily mixable sludges. When augers are used, enough water must be present to exceed the liquid limit for the auger to turn and mix the soil. The presence of underground piping and greater than 5 percent by volume of debris will favor ex-situ treatment methods over in-situ treatment methods, as screening and separation steps can be added to the ex-situ treatment methods.

In certain circumstances, the excavation and stockpiling of waste materials prior to treatment can trigger additional regulatory requirements and lower treatment levels for waste treatment. The presence of such regulations favors the use of in-situ treatment methods. Typically, lower treatment standards require increased homogenization of the treated material to ensure compliance with these low treatment levels. Since ex-situ treatment methods offer better process control and better homogeneity of the treated material, low treatment standards favor ex-situ treatment methods. The requirement for low or no dust emissions favor ex-situ treatment methods because of the reagents, soil, waste, etc., being combined and mixed in a mixing chamber. However, low odor and mercury vapor emissions favor in-situ treatment methods due to less disturbance of the soil or waste material at any one time. Most waste materials can be treated with either in-situ or ex-situ treatment methods, though the project-specific treatment costs may not be equal and the low-cost alternative will be favored. Ex-situ treatment methods typically have higher public and regulatory acceptance than in-situ, since most of the in-situ treatment occurs hidden from view. This perceived lack of control leads to lower acceptance for in-situ treatment methods. In the United States, this lack of acceptance for in-situ treatment methods has largely disappeared.

7.2 COMPONENTS OF IN-SITU OPERATION

The reagents must contact the mercury, other metals, and radionuclides to fixate the metals, so an effective distribution system or mixing operation is needed. In the optional in-situ field demonstration study (Phase 2), IT/NFS proposes to use a combination of an auger and a long-stick excavator to ensure thorough mixing.

As the largest remediation firm in the United States, IT routinely addresses excavation of hazardous, radioactive, and mixed waste materials and the contamination control issues surrounding that activity. From this experience base IT downselected its in-situ treatment processes. Some of the alternatives for soil excavation, mixing, and handling considered were:

Excavation Equipment

- Hydraulic excavator
- Backhoe
- Bucket loader
- Remote controlled bucket loader
- Auger systems

Dust Control

- None
- Water sprays
- Foam
- Wind screens
- Temporary enclosure

IT chose the auger system combined with a long-stick excavator to maximize mixing efficiency. Dust and mercury vaporization control will be maintained by controlling the moisture content of the soil/sediment and the type of reagent added. After selecting the type of mixing operations, IT developed its process. The major operations details for the field demonstration phase are as follows.

1. Conduct an abbreviated Multi Agency Radiation Survey and Site Investigation Manual (MARSSIM)-like historical site assessment of the selected site for the field demonstration.
 - Interviews with site personnel to better understand the previous use of the land (e.g., what chemicals and radionuclides may be present and if there are utilities and piping below ground.) If there are utilities and piping below ground level, IT will check to ensure that they are de-energized and disconnected, respectively.
 - Conduct a geophysical sweep to locate any large piping and drums that may be underground.
2. Contain the contaminated area with sheetpiling to form a rectangular box with dimensions to allow easy access by the crane manipulating the auger system and a long-stick excavator. The proposed dimensions are 40 by 60 by 10 feet deep. Total volume

will be approximately 24,000 cubic feet (cf) (at an average set density of 110 per cubic foot (pcf), this is about 1320 tons). Based on previous work, a <27.5% volume increase is expected. The sheetpile will therefore extend one to two feet above ground level to contain all treated material and allow some freeboard.

3. Collect six initial characterization samples. The samples will be collected with a hand-held mechanical auger. The material from each borehole will be composited.
4. Add one or more different stabilization/fixation additives and water, as needed, to the soil within the cell. (In the proposed process, the soil will be allowed to react for at least 12 hours before adding the second set of reagents. See items 5 and 6. Longer reaction times are acceptable.) During this field study, it is anticipated that 100 to 200 cy of soil will be treated each day. (In the full-scale operation, it is anticipated that approximately 400 to 500 cy per day per unit will be processed.) During the demonstration, enough reagents will be added to treat the 100 to 200 cy of soil each day.
5. Add additional water as necessary to provide a soil with the desired workability, to provide a partial seal to minimize mercury vaporization, and to minimize dust emissions.
6. Thoroughly mix the fixation additive into soil using an auger system. Either a liquid or slurry will be injected through the auger.
7. Repeat steps 4, 5, and 6 until all the soil has been mixed in the cell or cells that are being treated. As shown at the bench-scale, the mercury emission rate will have been greatly diminished by this first pass of treatment with the auger. Over 99.8 percent (worst case) of the mercury vapor has been arrested by the treatment and the mercury concentration is 0.1 percent of the current NIOSH REL.
8. Repeat steps 6, and 7 with the next reagent. A damp soil-like material will result.
9. Collect soil samples the next day for TCLP analysis. In the IT/NFS Team cost basis, 10 to 15 composite samples will be analyzed for the test plot.
10. Using the long-stick excavator, add a drying agent to the treated soil as needed to meet on-site disposal landfill waste acceptance criteria. It is understood that for treated material to meet the EMWMF WAC's, the soil must pass the paint filter test. IT has significant experience on controlling liquid bleed from soil and wastes. In addition, IT is well versed in designing treatment processes where the product is compactable to 95 percent standard proctor density if this will be required by the EMWMF WAC.
11. Excavate the treated soil with a long-stick excavator and stockpile for DOE. In IT/NFS cost basis, the soil excavated will be placed on a non-lined area near the treatment site. As directed by NETL, this soil may also be loaded into trucks for shipment to the on-site

landfill, or place the soil onto a plastic lined and bermed holding area awaiting shipment to the on-site landfill.

12. Any liquid mercury visible from ground level can be collected using a mercury vacuum and stored awaiting disposal by DOE. IT/NFS has the technology to treat this liquid mercury to meet the performance criterion.
13. Sheet piling will be left in place at the end of the field demonstration. At NETL directions, holes created during excavation operations can be filled with appropriate materials. In the IT/NFS Team cost basis, NETL or its designee will supply backfill material. In full-scale operations, sheet piling will be pulled and reused for the next treatment cell.
14. All equipment will be decontaminated. It is assumed that all equipment can be easily decontaminated to meet free release criteria. The wash liquid will be collected. In the IT/NFS Team cost basis, the decontamination water will be accepted at and treated at the site wastewater treatment (WWT) facility. The IT/NFS Team can treat the decontamination water. As an example, the water may be in-drum stabilized in 55-gallon drums and analyzed. In the process, cement-based stabilization will be used to convert the liquid to a non-hazardous material. Drums containing radioactive material will be given to the site for disposal.

Analytical will be similar to the bench-scale (Phase 1) except that only full-scale TCLP will be conducted unless otherwise directed by NETL. In the IT/NFS Team costs basis, since treated materials are to be disposed in an on-site landfill, the analytical required will be TCLP for the eight RCRA metals, isotopic uranium and thorium, gamma spectroscopic analyzes, and total metal analysis for the eight RCRA metals. TCLP analyzes will be conducted on all samples, and the other analyses will be conducted on six or less samples. For RCRA metals, all analytical will be at data quality objective (DQO) level 3 or less. There will be no contract laboratory procedure (CLP) level analyses. This level of analytical can be modified to provide all required data for disposal in the on-site EMWMF.

7.3 COMPONENTS OF EX-SITU OPERATIONS

As with the in-situ process, the reagents must contact the mercury, other metals, and radionuclides to fixate the metals, so an effective distribution system or mixing operation is needed. In the optional ex-situ field demonstration study (Phase 2), IT/NFS will use a pugmill to mix in all reagents thoroughly.

For an ex-situ approach, IT/NFS proposes a pugmill-based stabilization system. These batch systems scales almost directly from the bench test procedures. The volume of the pugmill and batch cycle times will be based on the treatment formulation and past experience with similar soils. For the site demonstration, it is assumed that DOE will excavate and move the soil near to the location of the pilot study.

Ex-situ stabilization with on-site disposal of the treated material should be considered for treatment of these mercury-contaminated soils at the demonstration- and full-scale processes. In the ex-situ alternative the contaminated soils would be excavated (by DOE) and mixed with treatment chemicals in a pugmill (IT/NFS). As in the in-situ process, the additives will react with the metals to form insoluble, non-leachable compounds that are less mobile than the untreated contaminants. Ex-situ treatment provides the performance and effectiveness of the treatment by stabilization and, in addition, the contaminants are removed from the current location, in the ground, to the on-site RCRA-like landfill.

7.3.1 General Full-Scale Concepts

The stabilization system will consist of various power screens, feeders, conveyors, and a series of pugmill mixers integrated into a complete system for the continuous mixing of soils and reagents. There will be a bermed plastic sealed asphalt pad underneath the major unit operations to catch potential emissions of liquid mercury. This is especially important under the sieving operations which is use to remove debris.

The soil will be screened to remove debris (>2 to 4 inch) and stockpiled. Soil from the stockpile will be fed to a 4 to 8 cy surge bin with a live bottom feeder that conveys the soils into the pugmill for blending with the stabilization reagents. The pugmill mix box is typically 4 feet by 8 to 12 feet containing two mixing shafts and is rated at 100-200 tons per hour capacity at 90 pounds per cf. The pugmill may be optimized for more thorough mixing by using the extended length configuration coupled with closely placed paddles. Paddles are bolted onto structural steel shafts with replaceable shafts flanged on both ends for ease of maintenance. The paddles will be high carbon steel and heat-treated welded at both ends.

In this process, the untreated soil mass will be measured on a belt scale as it enters the pugmill. The stabilization reagent(s) will be introduced from reagent feeder(s) attached to the pugmill. The reagent feed rate will be correlated with the soil feed rate to ensure the appropriate ratio of stabilization additive is delivered to the pugmill in a consistent manner. There will be less water

added in the ex-situ operation than with the in-situ operation. The treated soil will exit the pugmill on a conveyor, and will be placed in temporary stockpiles until daily sampling is completed. The treated material exiting the pugmill will have a moist soil-like consistency.

Dust from the stabilization operation will be minimized by periodically wetting the product stockpile, if needed. In addition, if necessary more dust control will also be achieved in the stabilization treatment system by the use of spray bars in the inlet and exit of the pugmill. This will create a mist curtain to minimize dust emissions from the pugmill.

7.3.2 General Demonstration-Scale Concepts

The major operations details for the field demonstration phase are as follows.

1. As with the in-situ alternative, conduct an abbreviated MARSSIM-like historical site assessment of the selected site for the field demonstration. Unlike the in-situ operation, a geophysical sweep will not be conducted since DOE is providing the soil to IT/NFS.
2. Mobilize equipment, pour the concrete pad, and if necessary, erect the IT/NFS sprung structure. It is assumed that a sprung structure or its equivalent will not be necessary during the demonstration program.
3. DOE will excavate the soil, transport the soil to a containment area (e.g., bermed asphalt pad, which if necessary will be in a containment, e.g., ventilated sprung structure)
4. Collect six initial characterization samples. The samples will be collected with a hand-held auger into the side of the feed stockpile.
5. The soil will be sieved to remove material >2-inch diameter. Any mercury that collects under the sieving operations can be collected using a mercury vacuum and stored awaiting disposal by DOE. IT/NFS has the technology to treat this collected liquid mercury to meet the performance criterion.
6. Blend one or more different stabilization/fixation additives and water, as needed, to the soil within the pugmill. If needed, the pugmill and screening operations can be enclosed in a plastic tent to minimize mercury emission on the site. During this field study, it is anticipated that 80 to 120 cy of soil will be treated each day. (In the full-scale operation, it is expected that about 400 cy per day will be processed.) During the demonstration, enough reagents will be added to treat the 80 to 120 cy of soil each day. Unlike the in-situ process, if a drying agent is needed for the treated soil to pass the paint filter test, the drying agent will be mixed into the soil using the pugmill instead of the long-stick excavator. In addition, if a dryer product is needed to make it compactable to 95 percent

standard proctor density, IT has significant experience achieving this criteria, if it is required by the EMWMF WAC.

7. Repeat step 6 until all the soil has been mixed. As shown at the bench-scale, the mercury emission rate will have been greatly diminished by the treatment with the first reagent. The treated product Hg emission rate will be well below the permissible exposure limit (PEL), REL, or TLV levels.
8. The pugmill will be inspected periodically to determine if there is liquid mercury pooling in the pugmill. If there is any liquid mercury in the pugmill, it can be collected with a mercury vacuum. IT/NFS has the technology to treat this collected liquid mercury to meet the performance criterion.
9. The product will be stockpiled and stored under a plastic tarp. Soil samples will be collected each day for TCLP analyzes. As in the in-situ cost basis, IT/NFS expects to analyze 10 to 15 composite treated samples.
10. All equipment will be decontaminated. It is assumed that all equipment can be easily decontaminated to meet free release criteria. The wash liquid will be collected. In the IT/NFS Team cost basis, the decontamination water will be accepted at and treated at the site WWT facility. The IT/NFS Team can treat the decontamination water. Drums containing radioactive material will be given to the site for disposal.

Analytical will be similar to the bench-scale (Phase 1) except that only full-scale TCLP will be conducted unless otherwise directed by NETL. In the IT/NFS Team costs basis, since treated materials are to be disposed in an on-site landfill, the analytical required will be TCLP for the eight RCRA metals, isotopic uranium and thorium, gamma spectroscopic analyzes, and total metal analysis for the eight RCRA metals. TCLP analyzes will be conducted on all samples, and the other analyzes will be conducted on six or less samples. For RCRA metals, all analytical will be at DQO level 3 or less. There will be no TCLP level analyzes. This level of analytical can be modified to provide all required data for disposal in the on-site EMWMF.

8.1 IN-SITU ASSUMPTION

- As stated in the solicitation, the cost analyzes are to use 50,000 cy-contaminated soil as its basis for treatment. IT/NFS assumes the soil has an average bulk density of 1.4 ton/cy. The treatment depth averages between 10 to 12.5 feet.
- The amount of debris in the soil does not preclude the use of an auger system.
- A two-step process will be used. The first reagent will be added and allowed to react overnight; the second reagent will be added on the second to fifth day.
- All equipment except the auger, which will have had extensive contact with the soil, can be easily decontaminated for free release.
- Decontamination will be conducted in order of decreasing usage: simple hand wiping, wiping or power washing with soap and water, wiping with dilute nitric acid, wiping with chelant solution, e.g., Radwash.
- According to directions from NETL and BJC, this is a “stockpile to stockpile” cost analysis, as such the price consist of the following unit operations:
 - Mobilization
 - Shakedown of equipment
 - Treatment of contaminated soil with commercially available auger
 - Decontamination and Demobilization
- Price does not include the following unit operations:
 - Grubbing of trees and shrubs during site preparation
 - Excavation and stockpiling of treated soil
 - Transportation to landfill
 - Disposal Cost of treated soil
 - Transport, treatment, or disposal of decontamination waters
 - Filling in the hole from which the untreated soil was excavated
 - Revegetation of the area where the soil was removed.
 - Treatment of any liquid mercury from the bottom of the excavation area.
- Surround area to be treated with sheetpiles. Tiebacks will not be used on the sheetpiles.

- Will reuse sheetpiles from one area to the next. Each treatment zone (i.e., sheetpile array) will consist of five 40 wide by 60 foot long sheet pile rectangles. A total of 10 sheetpile arrays will be used.
- Major electrical power will be supplied to the production site. IT will connect to these utilities during the mobilization/set-up stage.
- Treated products will be considered non-hazardous, after confirmation by analytical results, and can be stockpiled on unimproved land supplied by DOE and its contractors.
- Major Schedule Elements
 - Write Plans and Permits (6 months)
 - Mobilization and Shakedown (20 working days)
 - Treatment (145 working days)
 - Decontamination and Demobilization (20 days)
- Labor and Personnel Assumptions:
 - It is assumed that IT/NFS will submit one draft work plan, QA/QC plan, etc., one set of comment response documents, and one final set of plans. It is also assumed that there will be one draft readiness review and one final review.
 - The labor rates are FY 2001 escalated to 2003.
 - There will be a full-time H&S (E10 grade level) on-site 10 hr a day during the field operation.
 - Each person working on the site will receive 40 hours of on-site radiological/H&S/security training.
 - A full-time HP technician will be supplied by DOE.
 - Excluding the supplied HP technician, the project will be staffed by non-union IT personnel.
 - There is per diem for all non-Knoxville employees working on-the-job.
 - The duration of schedule will not be impacted by the location of the site and the production operations will not be lengthened because of security issues.
 - PPE includes four change outs of poly-coated Tyvek® and booties and, if needed, one change out of respirator cartridge per day per person.
- Will take 25 analytical characterization samples for the following:
 - TCLP RCRA 8 metals
 - Total RCRA metal

- Total U and Th
 - Alpha Isotopic
 - Gamma Spectroscopy
 - All sample analyzes will be performed using a 14-day Turn-around-time (TAT)
 - Due to the samples being potentially radioactive, there will be a 1.25 radiological multiplier on all TCLP samples.
- Will take 200 samples for TCLP of treated product and 40 samples for the remainder of analyzes. This is two samples per day of treatment for TCLP analyzes. The samples will be analyzed for:
 - TCLP RCRA 8 metals (7-day TAT)
 - Total RCRA metal (14-day TAT)
 - Total U and Th (14-day TAT)
 - Alpha Isotopic (14-day TAT)
 - Gamma Spectroscopy (14-day TAT)
 - Due to the samples being potentially radioactive, there will be a 1.25 radiological multiplier on all TCLP samples.

8.2 EX-SITU ASSUMPTION

- As stated in the solicitation, the cost analyzes are to use 50,000 cy-contaminated soil as its basis for treatment. IT/NFS assumes the soil has an average bulk density of 1.4 ton/cy. The treatment depth averages between 10 to 12.5 feet.
- A two-step process will be used. The first reagent will be added and allowed to react overnight; the second reagent will be added on the second to fifth day.
- In order to decrease the duration of the project, the IT process will use two treatment trains and the NFS's process will use three.
- The pugmills will use disposable liners. The cost analysis assumes that each pugmill will consume five liners over the course of the project. The pugmill liners enclosures, axles, and blades will be too contaminated to decontaminate. It is therefore assumed that the enclosures, axles, and blades will be disposed in the on-site landfill.
- The concrete pads used in this project will be broken down during the demobilization phase. It is assumed that this concrete will also be disposed in the on-site landfill.

- All equipment, except the pugmill liners and original set of twin augers, which will have had extensive contact with the soil, can be easily decontaminated for free release.
- Decontamination will be conducted in order of decreasing usage: simple hand wiping, wiping or power washing with soap and water, wiping with dilute nitric acid, wiping with chelant solution, e.g., Radwash.
- Major electrical power will be supplied to the production site. IT will connect to these utilities during the mobilization/set-up stage.
- Unlike the in-situ operation, a geophysical sweep will not be conducted since DOE is providing the soil to IT/NFS.
- Treated products will be considered non-hazardous, after confirmation by analytical results, and can be stockpiled on unimproved land supplied by DOE and its contractors.
- Labor and Personnel Assumptions:
 - It is assumed that IT/NFS will submit one draft work plan, QA/QC plan, etc., one set of comment response documents, and one final set of plans. It is also assumed that there will be one draft readiness review and one final review.
 - The labor rates are FY 2001 escalated to 2003.
 - There will be a full-time H&S (E10 grade level) on-site 10 hr a day during the field operation.
 - Each person working on the site will receive 40 hours of on-site radiological/H&S/security training.
 - A full-time HP technician will be supplied by DOE.
 - Excluding the supplied HP technician, the project will be staffed by non-union IT personnel.
 - There is per diem for all non-Knoxville employees working on-the-job.
 - The duration of schedule will not be impacted by the location of the site and the production operations will not be lengthened because of security issues.
 - PPE includes four change outs of poly-coated Tyvek® and booties and, if needed, one change out of respirator cartridge per day per person.
- According to directions from NETL and BJC, this is a “stockpile to stockpile” cost analysis, as such, the price consist of the following unit operations:
 - Mobilization

- Shake-down of equipment
- Treatment of contaminated soil with commercially available equipment
- Decontamination, demolition, and transporting concrete pads to the on-site landfill
- Demobilization
- Price does not include the following unit operations:
 - Excavation and stockpiling of untreated soil
 - Backfilling the hole from which the untreated soil was excavated
 - Revegetation of the area where the soil was removed
 - Preparation of lay down area for DOE's feed stockpile
 - Transportation of the treated soil to the landfill
 - Disposal Cost of treated soil
 - Transport, treatment, or disposal of decontamination waters
 - Treatment of any liquid mercury collected from under the sieving operations or from the pugmill.
- Major Schedule Elements
 - Write Plans and Permits (6 months)
 - Mobilization and Shakedown (20 working days)
 - Treatment (IT) (110 working days)
 - Treatment (NFS) (187 working days)
 - Decontamination and Demobilization (30 working days)
- Will take 25 analytical characterization samples for the following:
 - TCLP RCRA 8 metals
 - Total RCRA metal
 - Total U and Th
 - Alpha Isotopic
 - Gamma Spectroscopy
 - All sample analyzes will use a 14-day Turn-around-time (TAT).
 - Because of the samples being radioactive, there will be a 1.25 radiological multiplier on all TCLP samples.
- Will take 500 samples for TCLP of treated product and 100 samples for the remainder of analyzes. This is one TCLP sample per every 100 cy of treated product. The samples will be analyzed for:

- TCLP RCRA 8 metals (7-day TAT)
- Total RCRA metal (14-day TAT)
- Total U and Th (14-day TAT)
- Alpha Isotopic (14-day TAT)
- Gamma Spectroscopy (14-day TAT)
- Because of the samples being radioactive, there will be a 1.25 radiological multiplier on all TCLP samples.

8.3 SUMMARY OF ENGINEERING COST ANALYSES

The engineering cost analyzes in this report are based on the bench-scale results. They use the IT or NFS reagent dosages which successfully treated the largest total Hg concentration material (i.e., Spike 3 surrogate.) The cost for the proposed in-situ process includes treating each column of soil twice. The soil column will be treated with reagent set one on the first day, then on a later date, the remainders of the amalgamation/stabilization reagents will be added. The drying reagent will be added during the excavation or loading into transport truck step. In the ex-situ Shaw/IT processes, the soil will be treated in a continuous feed pugmill using the first set of reagents. On a subsequent day, this soil will be picked up, moved and fed into a second continuous feed pugmill that mixes in the remainder of the reagents. In the ex-situ NFS chemistry process, the reagents will be mixed using a series of batch pugmill process. Unlike the ex-situ process using IT's chemistry, a 12-hour reaction time between addition of sets of reagents is not needed. Each batch step is less than 15 minutes.

At the request of NETL and BJC personnel, four different cost analyzes were performed. The cost analyzes are bounded by the assumptions provided in **Sections 8.1 and 8.2** and the NETL/BJC directives in **Section 7**. **Table 8-1** is a summary table providing costs for the four IT/NFS alternatives. It is our understanding that much of the area to be treated is filled with backfill, debris, pipes, or other underground utilities. IT/NFS thus recommends that of the alternatives, an ex-situ process be used since it will have a wider applicability. In open areas, e.g., at the off-site properties, an in-situ treatment should be considered. Further, the NFS chemistry is recommended since it is a more mature chemistry and has several years of aging studies showing that the TCLP leachability decreases with time.

Costs breakdowns for the recommended alternative are in **Tables 8-2 and 8-3**. These tables divide the cost between major schedule or work breakdown structure (WBS) elements (**Table 8-2**) and other cost elements defined by DoD ²³ (**Table 8-3**).

Analysis of the costs in **Tables 8-1 through 8-3** shows the costs for IT/NFS processes are less than 30 percent the baseline costs. The IT/NFS processes thus met the solicitation objective of being less expensive than the baseline cost.

The cost estimates using the NFS chemistry in this document are less than price estimates provided by NFS or others in previous documents. The lower costs are mostly due to the economy of scale for this project. The production rates for the full-scale project are about 200 times faster than those used in previous cost estimates. This allows for a more efficient usage of labor and equipment. In addition, the fixed costs are divided over a reasonably large volume of soil instead of a comparatively small volume. This lowers the cost per cubic yard value.

Table 8-1 Summary Engineering Cost Analysis for the Four Alternative Treatments for 50,000 cy Soil in a Cost Plus Fix Fee Type Contract ¹			
In-Situ		Ex-Situ	
Alternative 1A Shaw/IT Chemistry	Alternative 1 B NFS Chemistry	Alternative 2 A Shaw/IT Chemistry	Alternative 2 B NFS Chemistry
\$8,141,897	\$9,633,394	\$6,433,887	\$9,966,425
\$163/cy soil	\$193/cy soil	\$129/cy soil	\$199/cy soil
Note 1: Add up to \$300,000 to each total cost for the collection and treatment of elemental mercury collected during the treatment operations.			

Table 8-2 Engineering Cost for Recommended Alternative Treatment for Treating 50,000 cy Soil in a Cost Plus Fix Fee Type Contract Schedule Driven Cost Categories		
Schedule or WBS Cost Element	Cost (\$)	Cost for Calculating Unit (\$)
Technology Application		
Planning, Preparation, and Permitting	648,136	
Mobilization, Setup, and Demobilization	250,458	
Site Preparation and Technology Setup and Shakedown	305,230	
Treatment of Waste by Ex-Situ Reagent Mixing	7,478,090	
Final Reporting and Effectiveness Evaluation	128,892	
Engineering, Management, and Administration	742,278	
Total Technology Application Costs		9,553,083
Other Technology-Specific Costs		
Compliance testing and analysis	413,341,	
Soil, sludge, and debris excavation, collection, and Control	0	
Disposal of residues	0	
Total Other Cost		413,341
Total cost for calculating unit cost		9,966,425 ¹
Quantity treated (cubic yards)		50,000
Calculated unit cost (\$/cubic yard)		199
Note 1: Add up to \$300,000 to the total cost for the collection and treatment of elemental mercury collected during the treatment operations.		

Table 8-3
Engineering Cost for Recommended Alternative Treatment for
Treating 50,000 cy Soil in a Cost Plus Fix Fee Type Contract
(Categories in Table 8-3 are from Reference 23)

Cost Category/Element Cost	Cost (\$)	Cost for Calculating Unit (\$)
Capital Cost for Technology		
Technology mobilization, setup, and demobilization	157,354	
Planning and preparation (regulatory permitting)	697,451	
Site work (utility installation)	200,708,	
Equipment and appurtenances (Process equipment and Appurtenance/construction)	0	
Startup and testing	108,129	
Other (Pre-deployment treatability testing)	0	
Total Capital Costs		1,163,642
O&M for Technology		
Labor	1,111,040	
Materials	3,962,022	
Utilities and fuel	Incl. In Mat'l.	
Equipment ownership, rental, or lease	1,983,796	
Performance testing and analysis	408,335	
Other	472,076	
Total O&M Costs		7,937,269
Other Technology-Specific Costs		
Compliance testing and analysis	0	
Soil, sludge, and debris excavation, collection, and Control	0	
Disposal of residues	0	
Other (Reporting, Meetings, and Project Management)	865,172	
Total Other Cost		865,172
Total cost for calculating unit cost		9,966,425 ¹
Quantity treated (cubic yards)		50,000
Calculated unit cost (\$/cubic yard)		199
Note 1: Add up to \$300,000 to the total cost for the collection and treatment of elemental mercury collected during the treatment operations.		

This section presents the results of quality assurance/quality control (QA/QC) measures implemented during sampling and analysis activities conducted for this project. Quality indicators from every aspect of data collection have been reviewed and an assessment of the data, with regard to project-specific objectives, is presented. Successful execution of project-specific objectives and procedures provides a strong indication that data produced are adequate for the purpose of evaluating the results of this treatment study.

Part of this treatability study was performed by IT Corporation (IT) and part was performed by Nuclear Fuel Services, Inc. (NFS). IT performed the analytical portion of the project in-house, while NFS subcontracted the analytical to EnviroTest Laboratories L.L.C. in Casper, Wyoming.

These data were evaluated against specific criteria to verify the achievement of all precision, accuracy, representativeness, completeness, and comparability goals established in the work plan to meet the project objectives. The results of this review are presented in the following sections.

9.1 ANALYTICAL PROGRAM AND QC ACTIVITIES

Each analytical method requires that method-specific QA/QC protocols be followed during sample analysis. These protocols are a critical part of the methods employed and were followed the laboratories during sample analysis. Examples of specific measures include detailed record keeping procedures, initial and continuing instrument calibrations, and analysis of instrument blanks and matrix spikes/matrix spike duplicates (MS/MSD).

9.1.1 Calibration Blanks

Calibration blanks are samples of analyte-free water or solvent used to verify the instrument "zero" during the progress of a run, or following analysis of samples or standards having high analyte concentrations. The purpose for analyzing calibration blanks is to identify contaminants that could be introduced into the sample as a result of any part of the analytical process.

Whenever target compounds are detected in calibration blanks, there is greater uncertainty regarding the positive identification of the same constituents in associated samples. For IT Corporation, the only element detected in the blank was zinc at a level of 0.006 milligrams per liter (mg/L). This element was not being evaluated within the objectives of the project and is qualified as non-detected using the 5X/10X rule.

9.1.2 Data Accuracy

Data accuracy is an indication of how close a measurement is to the actual value. Data accuracy is determined by spiking a blank with a known value and measuring the percent recover (%R) of the spike. **Table 9-1** shows the %R for the spike.

Table 9-1 Percent Recovery for Spiked Blank Solutions				
	Blank	Spiked Blank	Spike Value	% R
Element	mg/l	mg/l	mg/l	
Aluminum	<0.023	2.24	2.22	101
Antimony	<0.0893	2.26	2.22	102
Arsenic	<0.107	2.3	2.22	104
Barium	<0.0058	>2.00	2.22	No Calculation
Beryllium	<0.005	2.47	2.22	111
Cadmium	<0.0098	2.16	2.22	97
Calcium	<0.104	2.22	2.22	100
Chromium	<0.0214	2.25	2.22	101
Cobalt	<0.0236	2.11	2.22	95
Copper	<0.0156	2.22	2.22	100
Iron	<0.0116	2.31	2.22	104
Lead	<0.117	2.33	2.22	105
Magnesium	<0.113	2.04	2.22	92
Manganese	<0.0174	2.13	2.22	96
Nickel	<0.0227	2.06	2.22	93
Selenium	<0.135	2.72	2.22	123
Silver	<0.0196	0.021	2.22	1
Sodium	<0.368	2.06	2.22	93
Thallium	<0.259	1.73	2.22	78
Vanadium	<0.0258	2.26	2.22	102
Zinc	0.006	3.3	2.22	149

The %R was not calculated for barium due to barium not being a constituent of concern. The samples analyzed did not contain barium. Zinc is outside the %R guidelines but it was not a constituent of concern so the sample was not reanalyzed. All other constituents were within the qualified limits.

9.1.3 Data Precision

Precision is an indicator of the repeatability of a measurement, and is expressed as the relative percent difference (RPD) of two analytical measurements. Two grab samples of the original sample were analyzed and compared. The result are shown in **Table 9-2**.

9.1.4 Data Completeness

Data completeness is defined as the percentage of useable data points from the set of total data points that are required to obtain a specified confidence level. All samples required for this project were analyzed and results for all elements obtained. The data completeness for this project is one hundred percent (100%).

Table 9-2 Percent RPD TCLP on Duplicate Grab Samples of Untreated Soil			
	Sample 11094302	Sample TDL3766	%RPD
Element	mg/l	mg/l	%
Aluminum	<0.023	<0.023	Undefined
Antimony	<0.0893	<0.0893	Undefined
Arsenic	<0.107	<0.107	Undefined
Barium	<0.0058	0.233	200
Beryllium	<0.005	<0.005	Undefined
Cadmium	<0.0098	<0.0098	Undefined
Calcium	<0.104	>500	Undefined
Chromium	<0.0214	<0.0214	Undefined
Cobalt	<0.0236	<0.0236	Undefined
Copper	<0.0156	0.0156	0
Iron	<0.0116	<0.0116	Undefined
Lead	<0.117	<0.117	Undefined
Magnesium	<0.113	20.2	200
Manganese	<0.0174	3.27	200
Nickel	<0.0227	<0.0227	Undefined
Selenium	<0.135	<0.135	Undefined
Silver	<0.0196	<0.0196	Undefined
Sodium	>250	>250	Undefined
Thallium	<0.259	0.274	5.6
Vanadium	<0.0258	<0.0258	Undefined
Zinc	0.006	0.023	117

Most of the values are undefined due to values being below the detection limit. RPD's were calculated for remaining metals, excluding Zn; the detection limit value was used in the calculations. All of the metals in this table are not elements concern for this project since their concentrations are well below the RCRA criteria.

9.1.5 Mercury Results

The constituent of concern for this project was mercury. The TCLP of the as-received sample was analyzed in duplicate. The soil was spiked with metallic mercury and HgO (Spikes 1, 2, and 3) to increase the Hg TCLP leachability. Matrix spikes refer to spikes applied to the sample matrix and are often analyzed in pairs. The results are in **Table 9-3**.

Table 9-3 Percent RPD for TCLP of Untreated and Spiked Soil				
Analyte	Spike Level ¹	Total Mercury (mg/kg)	TCLP Result (ug/l)	% RPD
Mercury	“as is”	50.8	29.9	10.5
Mercury	“as is”	50.8	33.2	
Mercury	1	1250.8	32.4	41.2
Mercury	1	1333.0	49.2	
Mercury	2	1629.0	84.6	16.5
Mercury	2	1857.9	71.7	
Mercury	3	6273	2115	7.4
Mercury	3	6273	1965	
Note 1: Spike Level corresponds to the 2 nd and 3 rd spiked surrogate (See Table 6-1).				

Table 9-4 Percent RPD for TCLP of Treated Soils				
Analyte	Spike Level ³	Total Mercury (mg/kg)	TCLP Result (ug/l)	% RPD
Mercury	As is	50.8	ND (1.5) ¹	19.8
Mercury	As is	50.8	1.83 ²	
Mercury	2	1719	1.78 ¹	2.8
Mercury	2	1943	1.83 ²	
Mercury	3	5070	1.70 ¹	54.7
Mercury	3	5139	2.98 ²	
Mercury	3	6273	ND (1.5)	Undefined
Mercury	3	6273	ND (1.5)	
Note 1: Immediate TCLP initiated after less than 24 hr cure time				
Note 2: Aged TCLP initiated after >30 day cure time.				
Note 3: Spike Level corresponds to the 2 nd and 3 rd spiked surrogate (See Table 6-1).				

The % RPD for the spiked soil is less than 25 percent for all samples except spike 1. The RPD for the treated samples were all based on non-detected values or use the TCLP for the initial and aged samples. Except for one "duplicate", the percent RPD is less than 25 percent. IT also conducted MS on a TCLP for treated material. The MS recovery was 102 percent.

9.2 NFS SAMPLE RESULTS

The RPD results for the TCLP lead and mercury are all above the acceptance criteria. The duplicate is a second aliquot of the same sample which was subject to the same procedure. The samples were analyzed on an as received basis with efforts made to mix the sample before taking the aliquots for analysis. The matrix spike recovery for silver was below the acceptance criteria due to interference. One MS/MSD was performed for TCLP mercury. It showed low recovery. However, the corresponding RPD was good at 1.4, indicating good reproducibility of the results. The second MS/MSD performed was within the acceptance criteria. The analytical results of the NFS work is in **Appendix 2**.

IT/NFS received soil from the Alpha 2 building basement for testing. These soils had minor-levels of leachable mercury. The TCLP mercury concentrations were about an order of magnitude less than the performance criteria. The mercury vapor concentration in the headspace of the received bucket of soil after being opened for 15 minutes was above the TLV-TWA for mercury. Thus although the leachable mercury was low, there was enough metallic mercury in the soil to provide a substantial concentration of mercury vapors. The soil was spiked with metallic and soluble mercury compounds to simulate ranges of mercury contamination and to increase the TCLP mercury values. The spiked soils were treated using IT and NFS chemistries.

Important conclusions and lessons learned during this project were as follows:

- At the request of NETL and BJC, four different pricing scenarios are presented in this report. They include the prices for:
 - in-situ using IT chemistry,
 - in-situ using NFS chemistry
 - ex-situ using IT chemistry,
 - ex-situ using NFS chemistry.

The estimated price for all four alternatives are less than 30 percent the baseline cost of \$700-\$740 per cubic yard (cy) of soil. The treatment of the 50,000 cy of contaminated soil range from \$130 to \$200/cy. The IT/NFS processes do not produce any hazardous soil from non-hazardous soil or hazardous secondary waste that must be treated before disposal. Comparatively small amounts of liquid mercury may separate from the soil during soil excavation, in-situ treatment, manipulations of the soil, and in the pugmill. This liquid mercury will be collected and treated to meet the performance criteria. The estimated additional cost for the project for this treatment is less than \$300,000.

- Four different pricing scenarios were presented in the report. At this time, although the NFS ex-situ process is the highest price per cubic yard process, IT recommends that NFS ex-situ process be implemented. The NFS process chemistry has the longest proven track record and better than three years long-term testing to show that the treated samples maintain their low TCLP mercury concentrations. The chemistry has also been applied to many types of waste and now high-clay content soil. As a third point, the NFS process chemistry has previously been applied to treat >200 tons of soil that was mercury and radiologically contaminated at a major mixed waste TSDF. The ex-situ process is suggested since there are numerous underground obstructions at the ORR site. This will

limit or impede the in-situ application. In addition, the ex-situ process typically has better process control. In more open areas with fewer underground obstacles, the application of in-situ treatment has merit and should be considered.

- Mercury in the soil may be treated to meet the contract and UTS performance criteria using both IT and NFS chemistries.
- The selected IT formulation was shown to have no measurable deleterious aging affect between one to >30 days cure. This applies to soil samples contaminated with 50, 1500, and >5000 mg/kg mercury.
- Aging studies from this project, MER03 and MER04, show that the NFS treated materials treated to meet the UTS standard maintain their TCLP leachabilities less than the UTS levels for extended periods.
- The volume increase was less than 27.5 percent for treatment processes. This includes the volume increase from addition of drying reagents to dry the product to meet the EMWMF WAC. The percent volume increase from the NFS process is somewhat smaller than the IT process. The volume increase for this project was larger than previously observed due to the need to add more reagents to effectively treat the high-clay content soil. NFS believes, with their final optimized process, that the volume and weight increase measured in the project can be reduced in future operations.
- Treatment processes were determined which may be used in-situ or ex-situ.
- The IT/NFS process uses commercially available equipment that has previously been used successfully to treat HTRW in full-scale production jobs. The chemical reactions and processes are well founded in proven chemistry.
- Using the in-situ process, the initial product is a damp to slightly wet material. In the proposed process, Portland cement will be added to dry the product to pass the paint filter tests.
- Initial soils received from Oak Ridge contained insufficient levels of mercury and resulted in TCLP mercury concentrations that were similar to the applicable LDR limits. The soil was spiked with liquid mercury and moderately soluble mercury salts to increase the TCLP mercury leachability. Attempts to elevate levels of elemental mercury contamination introduced additional challenges. Questionable dispersion of spiked

material added uncertainty to the treatment protocol and may not be a true validation of the DeHg[®] technology for *in-situ* use.

- The soils used for this demonstration were nearly dry and had high clay content. The presence of such a large amount of clay complicated both the spiking of the sample with mercury and the subsequent treatment of this matrix. The high clay content in the soil mandates adding larger doses of water to make the soil fluid enough to mix *in-situ*. The pugmill design needs to be selected to ensure good mixing ability to ensure the reagents mix well with the elemental mercury. IT proposes the use of a high-shear dual shaft pugmill for this project.
- The presence of elemental mercury after the scale-up demonstration was eliminated by application of additional reagents and applying a more vigorous mixing regime to contact all the visible elemental mercury. The results of this testing show that the IT/NFS process can eliminate problems with amalgamation of elemental mercury on the soil matrix of interest using optimal chemical dosages and enhanced mixing of components. The optimized process adds more amalgamating reagent in the first step and increases the shear in the mixer to ensure conversion of the elemental mercury.
- It is expected that less than 10 percent of the mercury that is currently in larger pools may separate out from the bulk of the soil during mixing operations (*in-situ* or *ex-situ*) or sieving (*ex-situ*) operations. Elemental mercury that separated from the soil during these operations will need to be collected and disposed according to established regulations. The estimated cost for containment and treatment of the collected elemental mercury is less than \$300,000.
- A possible alternate protocol could be used during excavation for *ex-situ* treatment to enhance the separation of liquid mercury from the bulk of the soil.
- Although various problems were apparent with this treatability, the study did prove that UTS levels for mercury could be achieved in a scale-up reactor using the DeHg[®] technology even under the most challenging conditions.

1. Mercury “<http://www.ct.ornl.gov/stcg.hg/>”
2. Demonstration of the NFS DeHg® Process for Stabilizing Mercury (<260 ppm) contaminated mixed (TMS2229) ITSR Report from MWFA – MER02. DOE/EM-0468.
3. P. A. Taylor, K. T. Klasson, S. L. Corder, T. R. Carlson, K. R. McCandless Chemical Technology Division, Mercury Separation From Mixed Wastes: Annual Report, Ornl/Tm-13121, “<http://www.ct.ornl.gov/eds/rtg/tm13121.pdf>”
4. Mixed Waste Focus Area, Technology Development Requirements Document, Mercury Amalgamation, March 1997, INEL/EXT-97-00314
<http://tmfa.inel.gov/Documents/mercamal.pdf>
5. DOE/EM-0472 Mercury Contamination – Amalgamate (contract with NFS and ADA) Stabilize Elemental Mercury Wastes Mixed Waste Focus Area Prepared for . . . URL: http://apps.em.doe.gov/ost/pubs/itsrs/itsr1675_2.pdf
6. DOE/EM-0471 Mercury Contamination – Amalgamate (contract with NFS and ADA) Demonstration of DeHgSM Process Mixed Waste Focus Area Prepared for U.S. . . . URL: <http://apps.em.doe.gov/ost/pubs/itsrs/itsr1675.pdf>
7. P.D. Kalb, D. Melamed, M. Fuhrmann, J.W. Adams, M. Sapanara, C. DeTello, Sulfur Polymer Stabilization/Solidification of Elemental Mercury Mixed Waste, <http://www.dne.bnl.gov/ewtc/waste/hgpaper.htm>
8. “Stabilization Demonstration for Mercury-Contaminated Mixed Waste”, Gregory A. Hulet, Thomas B. Conley, Michael I. Morris, paper presented at the AIChE Spring Meeting, March 15-18, Houston, Texas (1999).
9. Mercury Contamination – Amalgamate (contract with NFS and ADA) Demonstration of DeHg® Process (TMS 1675) ITSR report from MWFA – MER01 project.
10. Demonstration of the Nuclear Fuel Services, Inc. (NFS) DeHg Stabilization Process for Treatment of Radioactively Contaminated Wastes Containing >260 PPM Mercury (MER03) – September 2000.
11. Demonstration of the Nuclear Fuel Services, Inc. (NFS) DeHg® Stabilization Process for Treatment of Various Mercury Species in Surrogate Waste Containing >250 ppm Mercury (MER04) – September 2001.
12. TMFA Documents “Mercury Contamination – Amalgamate (contract with NFS and ADA) Stabilize Elemental Mercury Wastes (TMS 1675). . . . Permit Procedures for Mixed Waste Treatment (99). . . . URL: See documents at: tmfa.inel.gov/Documents/AllDocs.asp
13. DOE/EM-0471 Mercury Contamination – Amalgamate (contract with NFS and ADA) Demonstration of DeHgSM Process Mixed Waste Focus Area Prepared for U.S. . URL:

<http://apps.em.doe.gov/ost/pubs/itsrs/itsr2229.pdf> or URL: See documents at:
tmfa.inel.gov/Documents/AllDocs.asp

14. DOE demonstration Projects: Mercury Amalgamation (MER01; Mercury Stabilization <260 ppm (MER02; Mercury Stabilization >250 pm (BNL Soils). . . . URL:
<http://web.aed.anl.gov/TechCon/Projects/mercury/resources/docs/nfs.ppt>
15. Stabilization of Wastes Contaminated with >260 ppm Hg. . . . LMES MER02 ORNL Poplar Creek Hg Amalgamation. Commercial projects with wastes containing mercury and other metals. . . . URL:
web.ead.anl.gov/TechCon/Projects/mercury/resources/docs/itcorporation.ppt
16. TechCon Organization Capabilities Questionnaire “. . . a variety of matrices. NFS permitted DeHg® in the State of Tennessee and operated a treatment system to convert more than 50m3 of mercury-mixed waste to a . . .” URL:
web.ead.anl.gov/TechCon/Public2/ShowCap.cfm?GetWhat=NUCLFUELHG&Interest+ORHG
17. TechCon Organization Capabilities Questionnaire “. . . a variety of matrices. NFS permitted DeHg® in the State of Tennessee and operated a treatment system to convert more than 50m3 of mercury-mixed waste to a . . .” URL:
web.ead.anl.gov/TechCon/Public2/ShowCap.cfm?GetWhat=estine
18. Michael I. Morris, Irvin W. Osborne-Lee, Greg A. Hulet, Demonstration of New Technologies, Required for the Treatment of Mixed, Waste Contaminated with >260 ppm Mercury, ORNL/TM-2000/147, January 2002, URL:
<http://tmfa.inel.gov/Documents/TM2001-147final.pdf>
19. C. H. Mattus , Measurements of Mercury Released from Solidified/Stabilized Waste Forms, ORNLITM-2001/17, April 2001
URL:<http://www.ornl.gov/~webworks/cpr/v823/rpt/109832.pdf>
20. OSHA Regulations: URL: “<http://www.osha-slc.gov/SLTC/healthguides/mercuryvapor/recognition.html>”
21. FRTR Report on Electrokinetic Separation, URL: <http://www.frtr.gov/matrix2/section4/4-4.html>
22. USACE Report “In Situ Electrokinetic Remediation for Metal Contaminated Soil” URL:
<http://aec.army.mil/prod/usaec/et/restor/insitu.htm> and
<http://aec.army.mil/usaec/technology/cleanup04a.html>
23. ESTCP Report “Final, In-Situ Electrokinetic Remediation of Metal Contaminated Soils Technology Status Report” US Army Environmental Center, Report Number: SFIM-AEC-ET-CR-99022, July 2000, URL:
http://www.estcp.org/documents/techdocs/ISERMCS_Report.pdf

24. J.R. Conner, *Chemical Fixation and Solidification of Hazardous Waste*, Van Nostrand Reinhold, New York, New York, 1990.
25. J.R. Conner, *Method for treating wastes by solidification*, US Patent 4,518,508, May 21, 1985.
26. Method 1311, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, U.S. Environmental Protection Agency, U.S. Government Printing Office, Washington, DC, 1999; EPA Publication SW-846.
27. *Guide to the Disposal of Chemically Stabilized and Solidified Waste*, U.S. Environmental Protection Agency, Washington, DC, 1982; EPA Publication SW-872.
28. *Handbook for Stabilization/Solidification of Hazardous Waste*, U.S. Environmental Protection Agency, Washington, DC, 1986; EPA Publication No. 540/2-86-001.
29. *Stabilization/Solidification of CERCLA and RCRA Waste*, U.S. Environmental Protection Agency, Washington, DC, 1989; EPA Publication No. 625/6-89/022.
30. *Treatability Study Manual, Solidification/Stabilization*, The PQ Corporation, 1993.
31. *A Tool to Support Strategic Planning in the Subsurface Contaminants Focus Area*, WSRC-RP-2002-00077, Rev. 0, Edited by Brian Looney, Savannah River Technology Center, Westinghouse Savannah River Company, Savannah River Site, Aiken, South Carolina.
32. *Remedial Technologies Screening Matrix and Reference Guide*, Version 4.0
<http://222.frtr.gov/matrix2/section4/gov.html>,
http://www.frtr.gov/matrix2/section3/table3_5_upper.html,
http://www.frtr.gov/matrix2/section3/table3_5_lower.html.
33. *Recent Developments for In Situ Treatment of Metal Contaminated Soils*, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response,
<http://www.clu-in.org/download/remed/metals2.pdf>.
34. *Treatment of Lead-contaminated Soils*, U.S. Environmental Protection Agency, Washington, DC, 1991; EPA Publication No. 540/2-91-009.
35. *Waste Specific Evaluation of RMERC Treatment Standard*, U.S. Environmental Protection Agency, Washington, DC 1998;
<http://www.epa.gov/epaoswer/hazwaste/ldr/mercury/rmerc2.pdf>.
36. *In Situ Thermal Treatment and Thermal Enhancement Technologies: Notes on an Engineering Forum Roundtable Discussion*, U.S. Environmental Protection Agency Technical Support Project Meeting, 1998,
<http://www.epa.gov/tio/tsp/download/stamens.pdf>.

37. Kalb, P.D., D. Melamed, M. Fuhrmann, J.W. Adams, M. Sapanara, C. DeTello, 1998, ***Sulfur Polymer Stabilization/Solidification of Elemental Mercury Mixed Waste***, [URL:hkhttp://www.dne.bnl.gov/ewtc/waste/hgpaper.htm](http://www.dne.bnl.gov/ewtc/waste/hgpaper.htm), November.
38. Kalb, P.D., J.W. Adams, L.W. Milian, G. Penny, J. Brower, A. Lockwood, 1999, ***Mercury Bakeoff: Technology Comparison for the Treatment of Mixed Waste Mercury Contaminated Soils at BNL***, Proceedings of Waste Management 1999, Tucson, AZ, March.
39. Kalb, P.D., D. Melamed, M. Fuhrmann, J.W. Adams, M. Sapanara, C. DeTello, 1998, ***Sulfur Polymer Stabilization/Lolidification of Elemental Mercury Mixed Waste***, presented at the 19th U.S. Department of Energy Low-Level Radioactive Waste Management Conference, Salt Lake City, UT, November.
40. U.S. Environmental Protection Agency (EPA), 1991, ***Superfund Engineering Issue, Treatment of Lead-Contaminated Soils***, EPA 540/2-91/009, <http://www.epa.gov/tio/tsp/download/leadcont.pdf>, April.
41. U.S. Environmental Protection Agency (EPA), ***In Situ Thermal Treatment and Thermal Enhancement Technologies: Notes on an Engineering Forum Roundtable Discussion***, C. Janowski, P. Leonard, and R.L. Stamnes, EPA Technical Support Project Meeting Best Western Salt Lake Plaza Hotel, Salt Lake City, UT.
42. U.S. Army Environmental Center (USAEC), ***In Situ Electrokinetic Remediation for Metal Contaminated Soils***.
43. Lindgren, Dr. Eric R., 1996, ***Electrokinetic Extraction at the Unlined Chromic Acid Pit, Sandia National Laboratories, New Mexico***, May 15 to November 24.
44. FRTR, 2002, ***4.5 Electrokinetic Separation (In Situ Soil Remediation Technology)***, Remediation Technologies Screening Matrix and Reference Guide, Version 4.0, July 29.
45. FRTR, 2002, ***4.6 Electrokinetic Separation (In Situ Soil Remediation Technology)***, July 29.
46. WM'99 Conference, 1999, ***Mercury Bakeoff: Technology Comparison for the Treatment of Mixed Waste Mercury Contaminated Soils at BNL***, Brookhaven National Laboratory, February 28-March 4.
47. U.S. Environmental Protection Agency, 1997, ***Recent Developments for In Situ Treatment of Metal Contaminated Soils***, Office of Solid Waste and Emergency Response, Technology Innovation Office, Washington, D.C., 20460, March 5.
48. U.S. Environmental Protection Agency, 1991, ***EPA Superfund Engineering Issue, Treatment of Lead-Contaminated Soils***, Office of Solid Waste and Emergency Response, Washington, D.C., 20460, April.

49. U.S. Environmental Protection Agency, 2000, ***Cost and Performance Report, Electrokinetic Extraction at the Unlined Chromic Acid Pit, Sandia National Laboratories, New Mexico***, March.
50. Singh, Dileep, Principal Investigator, 1996, ***Immobilization of Fission Products in Phosphate Ceramic Waste Forms***, Argonne National Laboratory, Argonne, Illinois.
51. Fleming, David, Greg Beyke, 1999, ***Six Phase Heating at the Skokie, Illinois Site***, Current Environmental Solutions, Marietta, Georgia, June 4, 1998 to April 30, 1999.
52. U.S. Environmental Protection Agency (EPA), 2000, ***Abstracts of Remediation Case Studies, Volume 4***, Member Agencies of the Federal Remediation Technologies Roundtable, June.

APPENDIX 1 :ORIGINAL STATEMENT OF WORK FOR PHASE 1 AND OPTIONAL PHASE 2

STATEMENT OF WORK FOR PHASE 1 AND OPTIONAL PHASE 2

The project was organized into a series of tasks. These tasks were defined in an agreed upon Statement of Work (SOW) for Phase 1 and the draft SOW for Phase 2. These tasks are as follows.

1.0 PHASE 1 -- BENCH SCALE TESTING

1.1 TASK -- COORDINATION AND PLANNING

This task shall consist of necessary activities to ensure coordination and planning of the project with DOE/NETL, representatives from the site that provided the contaminated soil samples, and the DOE Environmental Management (EM) technology development program. These shall include but are not limited to the following: Project Kick-off Meeting; submission of required National Environmental Policy Act (NEPA) documentation; submission of copies of required permits/licenses; specified document preparation and submission such as Work Plans, Sampling and Analysis Plans, Environmental, Safety, and Health Plans; requirements/coordination for packaging, shipping and receiving soil samples provided by DOE or DOE-selected representatives and for returning residual soil; and others.

1.2 TASK -- BENCH SCALE TESTING WORK PLAN

The Contractor shall prepare and submit for review a Work Plan for the bench-scale testing of mercury contaminated mixed waste soil with specific RCRA metals and radionuclides. The purpose of the Work Plan is to provide written documentation that describes how the testing will be performed, and detail the technical and operational elements of testing. Guidance to standardize the testing procedures and reporting of test results, shall be provided by DOE or DOE-selected representatives, and this guidance shall be considered in the development of the Work Plan. This draft plan is expected to include, but not be limited to, technology description; experimental design and procedures; testing protocol; test success criteria; and equipment and material. Key aspects of the plan are expected to include, but not be limited to: sampling and analysis plan (SAP); Environmental, Safety, and Health (ES&H) plan; shipping plan; waste management and decontamination plan; and Quality Assurance/Quality Control (QA/QC) plan. The draft Work Plan shall be submitted to DOE and DOE-selected representatives from the site providing the soil samples for review and approval. Any revisions must be made, and the Work Plan must be resubmitted and accepted for approval.

1.3 TASK -- BENCH SCALE TESTING

The Contractor shall conduct a bench-scale treatability study at the Contractor-provided facilities. Testing shall be conducted in accordance with the approved Work Plan. The testing

shall include operation of the technology system, sampling, and analyses. DOE or DOE representatives will provide soil samples and the site characterization results on the materials to be treated in the study. It is anticipated that one to three soil samples will be provided to the Contractor. In general, the objective for *in-situ* treatment is to render soil below RCRA-characteristic levels for the specified contaminants. The specified contaminant is mercury, with other RCRA metals and radionuclides (e.g. uranium) as a secondary concern. Initial test results shall be evaluated and minor modifications or refinements to the treatment system may be completed to enhance operation of the technology system, and retesting may be completed. Technology performance and cost benefit analyses shall be completed using results from the bench scale testing.

- **Subtask – Initial Testing of Technology System, Sampling and Analyses, Evaluation of Results, and Modification/Optimization of Technology System**

Initial tests will be completed to identify reagent formulations for scale-up proving tests and other technology system parameters. Tests will be completed according to the approved Work Plan. Sampling and analysis, according to the approved Work Plan, will be completed to evaluate test results. Results will be evaluated for achieving TCLP criteria for mercury, metals, and radionuclides, as well as stabilization performance for radionuclides. Minimizing final waste volume increases will also be emphasized. Based on initial test results, improvements in the test system shall be completed to optimize the performance of the technology system

- **Subtask – Re-testing of the Technology System, Sampling and Analyses, Evaluation of Results, and Cost Analysis**

Proving tests will be conducted to verify process chemistry using designated reagent formulation(s) derived from Scoping tests and any adjustments to the technology system. Sampling and analysis from the proving tests shall then be completed according to the approved Work Plan. Evaluation of technology performance shall be completed. A cost analysis of the treatment technology shall also be completed and compared to baseline technologies.

1.4 TASK – REPORTING

The Contractor shall prepare and present a briefing of Phase 1 results, and planned project work for Phase 2, at DOE NETL or another site selected by DOE. This presentation shall take place not less than 45 days before the expiration of the Phase 1 contract. The Contractor shall prepare for review and comments, a draft Topical Report on the Phase 1 contract activities. This report

shall follow the guidelines set forth in the contract and shall include, but not be limited to, treatment system design; Work Plans; description of and results from Phase 1 testing; and cost benefits of technology compared to baselines and alternative technologies. After review and comment by the DOE and DOE selected representatives, the Contractor shall revise the report and submit to DOE. Attendance and presentation at the Annual Industry Programs Conference and the Subsurface Contaminant Focus Area Annual Midyear Review shall also be completed.

At this time, the DOE will evaluate the merits of the project to determine if, and to what extent, Phase 2 shall be conducted. The Contractor shall not proceed with these Phase 2 tasks unless the Contracting Officer (CO) issues a modification to the contract to exercise Phase 2. If the Government elects not to pursue the Phase 2 tasks, the Contractor shall complete all of the remaining work defined in the Phase 1 contract.

2.0 PHASE 2 -- FIELD TREATABILITY TESTS (OPTIONAL)

2.1 TASK – COORDINATION/PLANNING AND SITE SELECTION/ASSESSMENT IN PREPARATION FOR FIELD TREATABILITY TESTS

This task shall proceed only after the review of the Phase 1 contract results by DOE and DOE-selected representatives, and the approval to proceed from the DOE/NETL Contracting Officer. This task shall include activities necessary to ensure coordination and planning of the field treatability testing at the selected site. These shall include but are not limited to the following: submission of required NEPA documentation; specified document preparation and submission such as Work Plans, SAPs, ES&H Plans; planning/coordination to carry out field treatability site selection/assessment; and others. The Contractor shall provide a base map of appropriate scale of the site selected for the field treatability tests shall be provided to DOE.

2.2 TASK – FIELD TREATABILITY TEST WORK PLAN

The Contractor shall develop and submit to DOE and DOE-selected representatives for review, a detailed Work Plan for the field treatability testing of the soil contaminated with specified metals and radionuclides. The Work Plan shall detail the technical and operational elements of this testing. The Work Plan shall be written in the context of the selected test site and shall contain all components required by the host site. The Work Plan must be approved prior to site mobilization to the field test site. Typical components of a Work Plan include, but are not limited to, the following: technology description; field test objectives; success criteria; field test design and procedures; site preparation, equipment, and materials; SAP; data management; data analysis and interpretation; ES&H plan; waste management and decontamination plans; public

participation; reporting; schedules; regulatory compliance; project organization; and supplementary material (provides more detailed information for SAP, QA/QC Plan, ES&H plan, waste management plan, public participation plan, readiness review plan, regulatory compliance plan, and standard operating procedures [SOP]). The Work Plan shall also include a plan for the verification of the field test results using independent field sampling data, control tests, or other acceptable means. The Work Plan shall be submitted to DOE for review and approval. Any modifications must be made, and the Work Plan must be resubmitted and accepted for approval.

2.3 TASK – FIELD TREATABILITY TESTS

The Contractor shall conduct a field treatability test at the site selected, using the treatment technology utilized in the Phase 1 bench-scale testing. This task shall include activities necessary to complete verification of these field test results using an independent methodology. Task also includes support activities necessary to complete field testing that shall include, but are not limited to the following: site mobilization/demobilization; site preparation; facilities/utilities requirements; waste management and decontamination; and site restoration. The Contractor shall provide construction support, ES&H support, and project supervision support as required for the field treatability study. The Work Plan must be approved by DOE and DOE-selected representatives of the field test site prior to conducting these activities. The Contractor shall coordinate fully with the demonstration site to perform these activities as planned in the approved Work Plan.

Upon conducting the field treatability tests, and not later than 48 hours from the start of the testing, initial test results from the data acquisition shall be provided in a “Quick Look” report that shall be in a format that is specified in the approved Work Plan. Upon completion of testing, results shall be evaluated with respect to meeting specific contaminant reduction goals; in general, the treatment performance requirement for this study shall be to render in-place soil below RCRA-characteristic levels for the specified contaminants. An engineering cost analysis of the technology shall also be completed and the cost benefits, compared to current baseline treatment technologies, shall be provided.

- **Subtask – Site Mobilization, Characterization of Test Site, Site Preparation, and Facilities/Utilities Requirements**

The Work Plan must be approved prior to site mobilization and conducting any other activities at the field test site. Mobilization shall consist of mobilizing personnel and equipment to and from the field test site.

Characterization of the test site shall be completed according to the approved Work Plan. It is anticipated that characterization of the field test site to determine below-surface features (for example, pipes, large conduits, and/or drums) that may interfere with delivery of the treatment, shall be completed using methods such as geophysical surveying. If such features are identified, either the test site will be moved, or DOE or DOE-representatives will remove, empty, or complete some other process to assure no interference with the planned delivery of treatment.

Site preparation activities shall be completed according to the approved Work Plan. It is anticipated that these may include such activities such as surface brush clearing for the excavation and staging areas, setting up personnel and equipment decontamination areas, setting up a water and reagent storage tank, and establishing an area of contamination that will encompass the excavation and the soil staging areas. As required, sheet pile will be installed at the field test site to contain the contaminated area; adequate freeboard will be provided to contain the soil after it expands.

- **Subtask – Setup/Shakedown and Testing of Treatment Technology**

The Contractor shall setup up all the equipment and run shakedown tests to optimize the equipment performance prior to treating contaminated soil. Upon completion of shakedown tests, the Contractor shall complete the remainder of the field treatability test. A Quick-Look report shall be written to show initial results from the Phase 2 testing. Sampling and analysis shall be completed according to the approved Work Plan. It is anticipated that ten to fifteen composite samples shall be collected for analytical analyses. Waste management, decontamination, site restoration, and site demobilization during and after completion of testing shall be in accordance with the approved Work Plan.

Upon completion of testing, results shall be evaluated with respect to meeting specific contaminant reduction goals. The Contractor shall also complete an engineering assessment of all costs associated with the treatment technology in this field treatability study, and provide cost benefits relative to current baseline treatment technologies.

2.4 TASK -- REPORTING

The Contractor shall prepare and present a briefing of Phase 2 results at DOE/NETL or another site selected by DOE. This presentation shall take place not less than 45 days before the expiration of the Phase 2 contract. The Contractor shall prepare for review and comments, a draft Final Report on the Phase 1 and Phase 2 contract activities. This report shall follow the

guidelines set forth in the contract and shall include, but not be limited to: treatment system design; Work Plans; description of and results from Phase 1 and Phase 2 testing; and cost benefits of technology compared to baselines and alternative technologies. After review and comment by the DOE and DOE-selected representatives, the Contractor shall revise the report and submit to DOE. Attendance and presentation at the Annual Industry Programs Conference and the Subsurface Contaminant Focus Area Annual Midyear Review shall also be completed.

APPENDIX 2: NFS QA FROM EnviroTest LABORATORY

ENVIRO-TEST QC REPORT

Client: Nuclear Fuel Services
1205 Banner Hill Road 110B Lab
Erwin TN 37650-9301

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Report Date: May. 14, 2002

Workorder: L5116

Contact: Melinda Keller

Test	Batch	Method	Reference	Units	Results	Family	Analyzed
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CR-EXT-LOW-CA

Water

Batch R17853

WG13558-1 BLANK

Chromium (Cr)

<0.02

mg/L

10-MAY-02

WG13558-3 DUP

Chromium (Cr)

L5116-5

<0.02

0.02

RPD-NA

mg/L

RPD

72

20

10-MAY-02

WG13558-2 LCS

Chromium (Cr)

104

%

Amount

2

80-120

10-MAY-02

WG13558-4 MS

Chromium (Cr)

WG13558-3

101

%

Amount

2

75-125

10-MAY-02

Batch R17854

WG13559-1 BLANK

Chromium (Cr)

0.20

mg/L

10-MAY-02

WG13559-3 DUP

Chromium (Cr)

L5116-2

<0.02

0.02

RPD-NA

mg/L

RPD

16

20

10-MAY-02

WG13559-2 LCS

Chromium (Cr)

107

%

Amount

2

60-100

10-MAY-02

WG13559-4 MS

Chromium (Cr)

WG13559-3

114

%

Amount

2

75-125

10-MAY-02

HG-EXT-HYD-CA

Water

Batch R17939

WG13629-1 BLANK

Mercury, (Hg)-Extractable

<0.0002

mg/L

13-MAY-02

WG13629-6 BLANK

Mercury, (Hg)-Extractable

<0.0002

mg/L

13-MAY-02

WG13629-3 DUP

Mercury, (Hg)-Extractable

L5116-5

0.0378

0.0342

RPD

mg/L

RPD

76

20

13-MAY-02

WG13629-2 LCS

Mercury, (Hg)-Extractable

99

%

Amount

N/A

80-120

13-MAY-02

WG13629-7 LCS

Mercury, (Hg)-Extractable

101

%

Amount

N/A

80-120

13-MAY-02

WG13629-4 MS

Mercury, (Hg)-Extractable

L5116-5

70

%

Amount

N/A

75-125

13-MAY-02

WG13629-8 MS

Mercury, (Hg)-Extractable

L5116-2

103

%

Amount

N/A

75-125

13-MAY-02

WG13629-5 MSD

Mercury, (Hg)-Extractable

WG13629-4

68.0

mg/L

RPD

1.4

20

13-MAY-02

WG13629-9 MSD

Mercury, (Hg)-Extractable

WG13629-8

104

mg/L

RPD

0.97

20

13-MAY-02

HG-EXT-LOW-CA

Water



Enviro • Test
LABORATORIES LLC.

820 West 1st Street Casper, Wyoming 82601
Phone: (307) 235-6741 Fax: (307) 266-1878
Toll Free 1(800)888-0066

Results are only applicable to samples submitted for analysis.
Limit of Liability: Although care and due diligence is taken in the performance of our services, our liability in all cases is limited to re-analysis at our expense or refunding the analytical costs charged for the work performed.



ENVIRO-TEST QC REPORT

Client: Nuclear Fuel Services
1205 Banner Hill Road 110B Lab
Erwin TN 37650-9301

Contact: Melinda Keller

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Report Date: May. 14, 2002

Workorder: L5116

Batch	Matrix	Reference	Result	Qualifier	Unit	Limit	Analyzed
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HG-EXT-LOW-CA

Water

Batch R17853

WG13558-1	BLANK		<0.006		mg/L		10-MAY-02
Mercury (Hg)							
WG13558-3	DUP	L5116-5	0.068	RPD-NA	mg/L	RPD 103	20 10-MAY-02
Mercury (Hg)		N/A					
WG13558-2	LCS		94		%	Amount 0.2	80-120 10-MAY-02
Mercury (Hg)							
WG13558-4	MS	WG13558-3	110		%	Amount 0.2	75-125 10-MAY-02
Mercury (Hg)							

Batch R17854

WG13559-1	BLANK		<0.006		mg/L		10-MAY-02
Mercury (Hg)							
WG13559-3	DUP	L5116-2	0.053	RPD-NA	mg/L	RPD N/A	20 10-MAY-02
Mercury (Hg)		N/A					
WG13559-2	LCS		111		%	Amount 0.2	80-120 10-MAY-02
Mercury (Hg)							
WG13559-4	MS	WG13559-3	82		%	Amount 0.2	75-125 10-MAY-02
Mercury (Hg)							

PB-EXT-LOW-CA

Water

Batch R17853

WG13558-1	BLANK		0.011		mg/L		10-MAY-02
Lead (Pb)							
WG13558-3	DUP	L5116-5	0.021	RPD	mg/L	RPD 139	20 10-MAY-02
Lead (Pb)		0.115					
WG13558-2	LCS		104		%	Amount 2	80-120 10-MAY-02
Lead (Pb)							
WG13558-4	MS	WG13558-3	104		%	Amount 2	75-125 10-MAY-02
Lead (Pb)							

Batch R17854

WG13559-1	BLANK		<0.004		mg/L		10-MAY-02
Lead (Pb)							
WG13559-3	DUP	L5116-2	<0.004	RPD-NA	mg/L	RPD 1400	20 10-MAY-02
Lead (Pb)		<0.004					
WG13559-2	LCS		104		%	Amount 2	80-120 10-MAY-02
Lead (Pb)							
WG13559-4	MS	WG13559-3	104		%	Amount 2	75-125 10-MAY-02
Lead (Pb)							

SE-EXT-LOW-CA

Water



Enviro • Test
LABORATORIES LLC.
420 West 1st Street Casper, Wyoming 82601
Phone: (307) 255-5741 Fax: (307) 266-1676
Toll Free 1(800)666-0306

Results are only applicable to samples submitted for analysis.
Limit of Liability: Although care and due diligence is taken in the performance of our services, our liability in all cases is limited to re-analysis at our expense or refunding the analytical costs charged for the work performed.



APPENDIX 3: IT/NFS WORK PLAN

IT CORPORATION
With NUCLEAR FUEL SERVICES
BENCH TEST WORK PLAN

Alternative Field Methods to Treat Mercury in Soil

Prepared by:



And
Nuclear Fuel Services, Inc.



Prepared for:

**National Energy
Technology Laboratory
(NETL) and
Department of Energy
(DOE)**

October 25, 2001

Revision No: 0
Date: 12/14/01

IT CORPORATION
BENCH-SCALE TREATABILITY STUDY
BENCH TEST WORK PLAN

For:

**National Energy Technology Laboratory (NETL) and
Department of Energy (DOE)**

Contract No. DE-AC26-01NT41345
IT Project No. 832410

Approved by:

Steven K. Downey, IT Project Manager

Date:_____

Patrick L. Gray, IT Quality Assurance Officer

Date:_____

John E. Razor, CIH, IT Program Director

Date:_____

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LIST OF ACRONYMS

ACWP	Actual Cost of Work Performed
ALARA	As Low As Reasonably Achievable
APC	Air Pollution Control
APHIS	Agricultural Animal and Plant Health Inspection Services
ARASA	Alternative Remedial Action Subcontracting Approach
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
ATG	Allied Technology Group
ATL	Applied Technology Laboratories
BCD	Base Catalyzed Decomposition
BCWP	Budgeted Cost of Work Performed
BCWS	Budgeted Cost of Work Scheduled
BDAT	Best Demonstrated Available Technology
BNL	Brookhaven National Laboratory
CAMU	Corrective Action Management Unit
CFR	Code of Federal Regulation
CHP	Certified Health Physicist
CHMM	Certified Hazardous Materials Manager
CLA	Certified Lead Auditor
CLP	Contract Laboratory Procedure
CONUS	Continental United States
CPFF	Cost Plus Fixed Fee
CPI	Cost Performance Index
CPR	Cardiopulmonary Resuscitation
CQCSM	Certified Quality Construction Systems Manager
CVAA	Cold Vapor Atomic Adsorption
cy	Cubic yards
D&D	Decontamination and Decommissioning
DARA	Disposal Area Remedial Action

List of Acronyms (continued)

DCMA	Defense Contract Management Agency
DEAR	DOE Acquisition Regulation
DeHg [®]	(Pronounced DEE-merc)
DoD	Department of Defense
DOE	Department of Energy
DOE/NV	DOE/Nevada Operations Office
DOE/ORO	DOE/Oak Ridge Operations Office
DOT	Department of Transportation
DQO	Data Quality Objective
DSSI	Diversified Scientific Services, Inc.
EA	Environmental Assessment
EM	Environmental Management
EMR	Experience Modification Rate
EMWMF	Environmental Management Waste Management Facility
EPA	U.S. Environmental Protection Agency
ERWM	Environmental Restoration and Waste Management
ES&H	Environmental Safety and Health
ES&HP	Environmental Safety and Health Plan
ETDC	Environmental Technology Development Center
ETTP	East Tennessee Technology Park
FAT&LC	Fernald Atomic Trades and Labor Council
FEMP	Fernald Environmental Management Project (formerly the Feed Materials Production Center)
FERMCO	Fernald Environmental Restoration Management Company
FFA	Flame Atomic Absorption Spectrometer
FF	Fluor Fernald
FTIR	Fourier Transform Infrared Spectrophotometer
FTIS	Fourier transform infrared spectrophotometer
GA	General Atomics
gal/d	Gallons/day

List of Acronyms (continued)

GC	Gas Chromatographs
GC/MS	Gas Chromatography/Mass Spectroscopy
GFAA	AA with Graphite Furnace Attachment
H&S	Health and Safety
HASP	Health and Safety Plan
HEPA	High Efficiency Particulate Air
HgWG	Mercury Working Group
IC	Ion Chromatograph with Various Detectors
ICP	Inductively Coupled Plasma
INEEL	Idaho National Environmental and Engineering Laboratory
ISMS	Integrated Safety Management System
IT	IT Corporation
ITSR	Integrated Technology Summary Report
LANL	Los Alamos National Laboratory
LC	Liquid Chromatograph
LDR	Land Disposal Restrictions
LEFPC	Lower East Fork Poplar Creek
LMES	Lockheed Martin Energy Systems
LSA	Low Specific Activity
MARSSIM	Multi Agency Radiation Survey and Site Investigation Manual
MLLW	Mixed Low Level Waste
MMES	Martin Marietta Energy Systems
MTCLP	Modified Toxicity Characteristic Leaching Procedure
MTRU	Mixed Transuranic Waste
MVA	Mercury Vapor Analyzer
MWFA	Mixed Waste Focus Area
MWIR	Mixed Waste Inventory Report
NETL	National Energy Technology Laboratory
NFS	Nuclear Fuel Services, Inc.
NPDES	National Pollution Discharge Emission Standard

List of Acronyms (continued)

NQA	National Quality Assurance
NRC	Nuclear Regulatory Commission
NSWC	Naval Surface Warfare Center
NTS	Nevada Test Site
ORNL	Oak Ridge National Laboratory
ORR	Oak Ridge Reservation
OSHA	Occupational Safety and Health Administration
OSW	Office of Solid Waste
OU	Operable Unit
PACDIV	Pacific Division
PARCC	Precision, Accuracy, Representativeness, Completeness, and Comparability
PC	Portland Cement
PCB	Polychlorinated Biphenyl
PE	Professional Engineer
POTW	Publicly Owned Treatment Works
PPE	Personal Protective Equipment
Ppm	Parts per million
PRDA	Program Research and Development Announcement
QA/QC	Quality Assurance/Quality Control
QAM	Quality Assurance Manuals
QAO	Quality and Operations
QAPP	Quality Assurance Project Plan
R&D	Research and Development
RA	Remedial Action
RCRA	Resource Conservation and Recovery Act
RCS	Regulatory Compliance Specialist
RFP	Request for Proposal
RMERC	Reporting of Mercury
ROD	Record of Decision

List of Acronyms (continued)

RPD	Relative Percent Difference
RPP	Radiation Protection Program
RSO	Radiation Safety Officer
RTA	Rotary Thermal Apparatus
RTG	Radioisotopically Powered Thermoelectric Generator
RWP	Radiation Work Permit
SAD	South Atlantic Division
SCFA	Subsurface Contaminant Focus Area
SOG	Standard Operating Guideline
SOW	Statement of Work
SPCC	Spill Prevention Control and Countermeasure
SRS	Savannah River Site
SSO	Site Safety Officer
SSS	Storm Sewer Sediments
SW	Solid Waste
TAL	Technology Applications Laboratory
TAG	Technology Applications Group
TCLP	Toxicity Characteristic Leaching Procedure
TDL	Technology Development Laboratory
TDRH	Tennessee Department of Radiological Health
TERC	Total Environmental Restoration Contract
TNRCC	Texas Natural Resource Conservation Commission
TOC	Total Organic Carbon analyzer
TOX	Total Organic Halide
TSCA	Toxic Substances Control Act
TSDF	Treatment, Storage and Disposal Facility
UEFPC	Upper East Fork Poplar Creek
UHC	Underlying Hazardous Constituent
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency

List of Acronyms (continued)

UST	Underground Storage Tank
UT	University of Tennessee
UTS	Universal Treatment Standard
UTS/UHC	Universal Treatment Standards/Underlying Hazardous Constituents
UV	Ultraviolet
UV/VIS	Ultraviolet/Visible Spectrophotometer
VOC	Volatile Organic Compound
WAC	Waste Acceptance Criteria
WBS	Work Breakdown Structure
WEMA	West End Mercury Area
WMP	Waste Management Plan
WPRAP	Waste Pits Remedial Action Project
WWT	Water Waste Treatment

1.0. BACKGROUND

1.1 Introduction

IT Corporation (IT) appreciates this opportunity to submit this work plan for bench-scale treatability programs for the fixation of Department of Energy (DOE) mercury-contaminated soils. Mercury, as well as other Resource Conservation and Recovery Act (RCRA) inorganic materials (D004 - D0011), will be fixated to meet or exceed the criteria stipulated in the solicitation (DE-RA26-01NT41030).

IT and its team member, Nuclear Fuel Services (NFS), have joined to bring the best technology and resources to treat a variety of mercury-contaminated soils typical of those found at DOE facilities. The soils for this study are not radioactively contaminated; however, this process may be used “as is” or with minor modifications to treat radiologically contaminated or mixed waste media soils/sludges/sediments/debris.

NFS developed the DeHg® (DEE-merc) process that will be used as the main treatment for this Alternative Field Treatment for Mercury in Soil program. The IT/NFS Team approach will ensure that the best commercial treatment process is available to treat the DOE contaminated materials. We have used the evaluation factors of process safety, effectiveness, implementability and cost effectiveness to meet DOE’s need for processing contaminated media to meet various RCRA and Land Disposal Restriction (LDR) criteria.

IT/NFS will demonstrate a batch mix stabilization system for treatment of the DOE mercury-contaminated soils and sediments. IT/NFS believes that a combination of both amalgamation and fixation chemistry will be required to adequately treat the full range of DOE soils, sediments, and wastes. The stabilization chemistry will utilize amalgamation, sulfide-based reagents and several other proprietary compounds as the primary mechanism for reducing the leachability of the mercury, RCRA metals, and radionuclides. Portland cement or other drying agents may be used in the stabilization process to solidify liquid(s) and to reduce leachability of other metals. If desired by the National Energy Technology Laboratory

(NETL), the process could be modified to yield a high strength impermeable structure that meets different final disposal criteria.

NFS has previously demonstrated this process on numerous specimens of radioactively-contaminated elemental mercury from federal facilities including the following: the Idaho National Engineering and Environmental Laboratory (INEEL) (oil contaminated elemental mercury); East Tennessee Technology Park (ETTP), formerly K-25, (“dirty mercury”); Los Alamos National Laboratory (LANL) (elemental mercury); Fernald Environmental Management Project (FEMP) (elemental mercury); high Tc-99 bearing ion-exchange resin contaminated with mercury and other RCRA metal bearing waste from the Portsmouth Gaseous Diffusion Facility (MER02); elemental mercury-contaminated (> 260 ppm) radioactive soils from Brookhaven National Laboratory (BNL) (MER03); and surrogate soil with high levels of mercury (MER04). In each demonstration project, the process achieved the Universal Treatment Standard (UTS) for mercury (<0.025 -mg/L mercury in Toxicity Characteristic Leaching Procedure [TCLP] leachate). (References 1-9)

DeHg® consists of a two-step treatment process. Following any necessary material pretreatment (e.g. sorting, separating, size reduction, etc.) the material will first be treated using an amalgamation technique to stabilize elemental mercury contained in the waste or mercury-contaminated media. This step is followed by a chemical stabilization process that utilizes a proprietary reagent to break mercury complexes and allow for removal of the mercury from the material slurry as a stable precipitate. Treatment parameters such as reaction times, blending speed, and reagent dosage have been established from prior experience noted above.

The overall program has two phases; Phase I – bench-scale studies and Phase II – field demonstration. After completion of Phase I, one or more successful companies will participate in the field demonstration. This workplan describes the plans for Phase I. If selected for Phase II, IT/NFS will write a separate workplan for Phase II. In Phase I, IT/NFS will screen the stabilization reagents in bench-scale tests to identify optimum formulations. Bench scale testing will be completed at both IT’s and NFS’s facilities. DOE will provide

one sample of contaminated soil to the both IT/NFS facilities by DOE or DOE-selected representatives from DOE's specified contaminated site. For the untreated soil provided for testing, DOE will provide an analysis of the contaminants of interest as well as soil properties and site condition variables. The treatment technology will be applied in bench- scale testing, and treatment performance will be evaluated based on removal or immobilization of specified metals and radionuclides such as mercury, uranium, and others. In this study, IT/NFS will conduct the necessary chemical and physical characterization of the contaminated soils and treated materials for compliance with the performance criteria and engineering analyses of the process. All soil samples will be returned to DOE or its representative.

If the project continues to Phase II, the most suitable formulation from Phase I would be used in the field demonstration test. The field demonstration treatability study will be conducted at a contaminated site selected by DOE. Candidate test sites for the field study include the Oak Ridge Y-12 National Security Complex, Offsite David Witherspoon properties, and other similarly contaminated sites. The field test results will be validated using an appropriate independent methodology. Evaluation of the technology performance and cost benefits will also be completed. IT/NFS assumes that the Phase II test area to be approximately 40 by 60 feet and at a depth of 10 feet. If desired by DOE and NETL, the treatment process may be easily modified from an in-situ treatment to a pugmill treatment of excavated material to achieve the same performance criteria.

Key experts with practical experience have been designated to oversee and coordinate removal and treatment actions. The importance of coordinating a multidisciplinary team, where each team member will be responsible for their part of the project, is recognized. Therefore, we have selected experienced team members who can make decisions independently and can clearly communicate those decisions to other team members while ensuring that the objectives of the project are met.

1.2 Background and DOE's Need

After years of designing, manufacturing, and testing nuclear weapons, DOE is faced with the challenge of containing contaminated soils and materials and cleaning up the hazardous waste left behind. More than 5,700 known DOE groundwater plumes have contaminated more than 475 billion gallons of water. DOE landfills contain more than 3 million cubic meters (m^3) of buried waste contaminating the surrounding environment. At DOE sites throughout the country, soil, groundwater, and landfills containing or contaminated with hazardous and radioactive contaminants have special cleanup needs.

DOE currently has in storage 167,600 m^3 of Mixed Low Level Waste (MLLW) and Mixed Transuranic Waste (MTRU) that are not being treated (based on Mixed Waste Laboratory Report [MWIR] 1995 database). Of this amount, approximately 28 percent is labeled as mercury-contaminated. According to the Proposed Site Treatment Plans, current facilities (on- or off-site) can only treat a fraction (5 percent) of the mercury-contaminated waste; the remaining 95 percent lacks the option of being treated at existing facilities. Treatment of these wastes may be divided into three different categories:

- Amalgamation for treatment of elemental mercury,
- Stabilization for wastes with <260 parts per million (ppm) of mercury, and
- Separation and treatment of wastes with >260 ppm of mercury.

About 75 percent of this mercury-contaminated waste is stored at the Oak Ridge Reservation (ORR), INEEL, and Savannah River Site (SRS). The majority of waste is in the form of debris, soils, sludges, and wastewaters.

- The ORR has 11,400 m^3 of mercury-bearing waste consisting of debris, soil, sludges, and waste waters and has a small volume (590 m^3) of unique or unknown waste with mercury
- INEEL has approximately 9,600 m^3 of mercury-bearing wastes; 99 percent are debris and sludges
- SRS has 7,700 m^3 of mercury-contaminated debris.

A complete list of mercury-contaminated waste sites is available at DOE's web site:

<http://www.ct.ornl.gov/stcg.hg>.

1.3 Project Specific Needs

This current NETL project deals specifically with mercury-contaminated soils which have not been excavated and are still under the control of DOE, or where DOE may be a principle in the clean up of a site. As such, the soil is not currently regulated by RCRA. DOE is evaluating treatment alternatives to control mercury concentrations in the groundwater and surface water. The treatment must reduce the transportability (particulate transport and leachability) of mercury to the creek. During the solicitation, the regulatory driver for this remediation was the National Pollution Discharge Emission Standard (NPDES) permits. This has changed such that the clean-up action is now under a Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) regulation.

Under this action, contaminated site soils and sediments at the Y-12 Plant are to be remediated to protect current and future industrial workers from exposure. These soils may be contaminated with mercury, other heavy metals, and radionuclides (primary uranium). The preferred remedy for roughly 130,000 yd³ of contaminated soils and sediments is excavation and disposal. It is anticipated that up to 50,000 yd³ of the excavated material will not meet the waste acceptance criteria (WAC) of the proposed on-site disposal location (the Environmental Management Waste Management Facility [EMWMF]) as a result of failing RCRA, LDRs. Although the excavated material is not anticipated to be a RCRA-listed waste, it is likely that portions of the soils will not pass the TCLP screen and will be considered RCRA-characteristic wastes for mercury. A soil or sediment fails the TCLP for mercury when the mercury concentration is greater than or equal to 0.2 mg/L in the TCLP extract.

This technology study is focused on needs identified by the DOE Nuclear Weapons Complex sites for technologies to treat, remove, or immobilize mercury in soil and sediment, although the treatment of uranium and other radionuclides and heavy metals is of secondary interest. DOE hopes that significant cost savings for remediation could result using improved

technologies compared to the baseline technologies. The baseline technologies are as follows:

- Removal of contaminated soils and sediments (and possibly buildings) and treatment of the contaminated material using thermal desorption. The approximate cost for this operation is \$740/cy. In 1993, DOE indicated that the costs for commercial thermal desorption activity range from \$125 - \$600/ton of material, with no proven alternative technologies to treat contaminated soil and control mercury release into groundwater.
- Capture of waters, which contain low levels of mercury bound for Upper East Fork Poplar Creek (UEFPC) and treatment via sorption with granular activated carbon and/or enhanced air stripping.

2.0. PROJECT DESCRIPTION

2.1 Overview and Significant Elements

IT/NFS are conducting bench- and optionally, demonstration-scale programs for the fixation of DOE mercury-contaminated soil for NETL/DOE. This work plan describes only the bench-scale treatability program for non-radioactive soil from the basement of the Alpha-2 building. Mercury, as well as other RCRA inorganic-contaminants materials, D004 - D0011, are to be stabilized to meet RCRA requirements for leachability. The other RCRA metals and radionuclides are of secondary consideration in this study. The final product structure can be manipulated to meet the WAC. for any on-site or off-site (e.g. Envirocare) landfills. The stabilization processes are very flexible and reagent mix and loading can be adjusted to meet a wide range of leachability requirements, as well as landfill acceptance criteria and permits.

The IT/NFS team's process is not only applicable to in-situ treatment of soil and sediments listed in the NETL solicitation, but with minor modification, may also be applied to most mercury-contaminated waste within the DOE complex. Depending upon the waste stream, the mixing operation can be adapted from an in-situ operation to in-drum stabilization or mixing in a pugmill. Since a batch in-situ process is being used in this project, the results are readily scaleable to larger projects.

The full-scale IT/NFS process will use commercially available equipment slightly modified to accommodate site-specific conditions. Using commercially available equipment allows for easier scale-up from the bench-, through field-testing, and into full-scale treatment operations. While a soil matrix or a waste stream may be complex, our treatment approach is straightforward and geared specifically toward full-scale operations.

During the demonstration phase, or full-scale operations, treatment under buildings is limited by access, so a modification of the current process would be used. In this modified process, reagents would be injected under the buildings. Tighter, less permeable soils typically require more horizontal wells to provide contact between reagents and elemental mercury.

The same basic chemistry would be used in this process, with some proprietary changes being implemented to ensure that the delivery system works effectively. As with the proposed process, multiple types of reagents would be necessary.

In this study, IT/NFS will demonstrate a batch mix fixation system for treatment of the DOE mercury-contaminated soils. The stabilization chemistry will utilize either amalgamation, sulfide-based reagents, or one of several proprietary commercial compounds as the primary mechanism for reducing the leachability of the mercury from the soil. IT/NFS will screen the fixation reagents in bench-scale tests to identify optimum formulations for the soils selected for this study. This formulation would then be used in the optional demonstration test. In the bench-scale study, the formulations will initially be samples weighing less than 1-kilogram. After the optimum formulation is selected, it will be scaled-up in the verification test to an approximately 15 - 25 kg batch test. The significant elements of IT/NFS's study are as follows.

- Project plans will be submitted to and approved by NETL/DOE.
- DOE will ship soil samples to IT and NFS laboratories to be homogenized as necessary and submitted for characterization analyses.
- Bench-scale tests will be conducted to screen fixation reagents and develop an effective formulation for fixation of mercury and as a secondary criteria, other RCRA metals.
- IT/NFS has been informed that the soil is contaminated with mercury and is not classified as a radioactive material. All materials will be shipped back to DOE at the end of the project. IT/NFS will not take ownership of the soil, waste, treated soil, treated waste, or secondary waste.
- A draft and final Topical report will be submitted to NETL/DOE.

This project will be executed at IT's Technology Development Laboratory (TDL) in Knoxville, Tennessee and NFS's Applied Technology Laboratory (ATL) in Erwin, Tennessee. All in-house metals analyses will be performed at these locations.

2.2 *Contaminated Soil Description*

As indicated in the solicitation, the material to be treated are soils which are contaminated with mercury from almost non-detectable values to greater than 0.7 percent. The soil was collected by DOE or its representative and the characterization analyses supplied to the IT/NFS team. The soil sampling crew observed metallic mercury beads on the soil from the Alpha-2 Building. From the supplied analytical report, the soil contains mostly metallic mercury (II), and mercury sulfide.

Our facilities have all of the required Nuclear Regulatory Commission (NRC), U. S. Environmental Protection Agency (EPA) and State licenses, registrations and permits to handle, store and perform these treatability tests. Our facilities are in compliance with the pertinent regulatory requirements and the Request for Proposal (RFP) requirements.

2.3 *Test Objectives and Success Criteria*

This study deals with fixation of non-radioactive mercury-contaminated soil which is potentially contaminated with other RCRA metals. As indicated in the solicitation, the treatment of the other RCRA metals and radionuclides are of secondary concern. The mercury in the soil may vary from less than detection limit to greater than 0.7 percent. Based on this range of mercury contamination, if the soil was excavated, destined for disposal, and failed the TCLP, the soil would be grouped into both the <260 mg/kg and high mercury (>260 mg/kg) categories. Even though the soils contain high levels of mercury, it is our understanding that since these soils have not been excavated for disposal, they are currently not regulated by RCRA. The materials IT/NFS are receiving are considered non-RCRA regulated samples.

As described in the solicitation, treated soil from the selected alternative treatment process will be sent to the onsite EMWMF for final disposal. The treatment technology applied to the contaminated soil and sediment is required to reduce the contamination levels so that (1) in-place soil will be rendered below RCRA-characteristic levels for the specified contaminants, or (2) excavated soil will be treated to meet RCRA alternative LDRs for soil. For an in-situ treatment technology this means that one or more of the following must be met:

- Soil less than the acceptable level for mercury (0.2 mg/L in TCLP leachate)
- Ten times the Universal Treatment Standard (UTS) (0.25 mg/L) in TCLP leachate.

For a technology conducted ex-situ, one or more of the following must be met:

- Ninety percent removal of total mercury measured in the soil (for mercury removal technologies)
- Ninety percent removal of mercury in the leachate from a TCLP test (for immobilization technologies)
- Ten times the UTS (0.25 mg/L in TCLP leachate).

As a secondary objective, the non-mercury RCRA metals will be treated to meet the Toxicity Characteristic (TC) limits.

In addition to the above treatment performance criteria for mercury, both the in-situ treatment and the ex-situ treatment must document an estimated performance cost lower than low temperature thermal desorption baseline treatment when implemented on a volume of up to 50,000 cy, including all capital costs.

The IT/NFS goal for these studies is to complete bench- and field-demonstration-scale treatability tests which:

- Meets all of the program objectives defined in the solicitation,
- Maintains compliance with applicable health and safety criteria for worker and public exposure,
- Provides data needed to design a scaleable process to treat mercury-contaminated soil and sediments, and

- Demonstrate improved field technologies that can treat soil and sediment contaminated with metals and radionuclides at a cost below the baseline technology.

2.4 *Technical Approach*

This project will be executed at the team laboratories (TDL and ATL). The TDL personnel have worked very closely with the engineering and remediation groups that design IT's full-scale commercial treatment systems that remediate government and commercial sites. This close interaction and feedback ensures that bench- and technology demonstration-scale studies are designed to develop critical data needed to scale-up and execute projects in the most cost-effective manner. Our approach to stabilizing this mercury-contaminated soil provides the best value to the DOE-wide complex. The technology selected for the bench-scale treatability study is amalgamation/stabilization/ fixation. Stabilization, or chemical fixation, transforms contaminants into their least toxic and/or mobile form, thereby reducing their impact on the environment and human health. Fixation/stabilization reagents will be based mostly on specialty chemicals from NFS.

The IT/NFS team's amalgamation/fixation process is based on over ten years of research and demonstrations by NFS and modifications of IT's mercury metal treatment process. The commercial name for the NFS mercury treatment process is DeHg[®]. (References 10-13)

DeHg[®] is an ambient temperature process which converts mercury in contaminated soils or mixed waste (waste containing mercury, potentially other characteristically hazardous materials, and radioactive components) to a characteristically non-hazardous material, or a non-hazardous low-level radioactive final waste form, respectively. These treated materials, or final waste forms, are suitable for land disposal. The DeHg[®] process was initially developed in the early 1990s. It has enjoyed excellent success in demonstrating capability to stabilize a variety of DOE mercury mixed wastes, including radioactively contaminated elemental mercury (MER01), mercury (<260 ppm) and metal contaminated waste (MER02), various mercury species stabilization (MER02A), and mercury stabilization in DOE soils (MER03). The MER01 and MER02 projects have been previously reported in DOE Environmental Management (EM) Integrated Technology Summary Reports (ITSRs).

DeHg[®] was also selected to participate in MER04, a demonstration of capability to stabilize a variety of mercury species in surrogate soil with total mercury concentration >260 ppm. The USEPA is participating in MER03 and MER04 by performing advanced leachability studies on the final waste forms to verify performance for various pH ranges. (References 4-7)

In each of these important demonstration projects, DeHg[®] achieved all final waste form objectives, namely UTS for mercury and metals, and other applicable waste acceptance criteria. DeHg[®] has also addressed a variety of commercial mercury-contaminated mixed wastes that have achieved acceptance criteria at licensed radioactive disposal sites. NFS has invoked a program of continuous improvement to enhance the capability of the process to address increasingly tighter regulations with regard to mercury stabilization.

The first stage of the DeHg[®] process involves amalgamation of the elemental mercury component (if present). Prior to amalgamation, it may be necessary to condition waste by shredding, grinding, and/or slurring with water. This amalgamation step converts elemental mercury to a very insoluble compound.

The second stage of the process is stabilization/fixation of soluble or complexed mercury species using the proprietary DeHg[®] reagent. This reagent has the capability to free mercury from stable, soluble complexes and subsequently allow for its precipitation as a stable, non-leachable salt. The DeHg[®] reagent was originally developed to treat solidified mercuric thiocyanate wastes when traditional mercury stabilization techniques failed to treat the strongly complexed mercury in this waste.

The process utilizes standard processing equipment connected in typical fashion. Following treatment using the DeHg[®] reagent(s), the stabilized waste forms are typically packaged for burial.

A similar amalgamation-stabilization treatment process has been used by IT. For a drum of mercury-contaminated soil, the treatment would be similar to the following. The drum is partially filled with soil/sediment. An agitator is inserted into the container and turned on; zinc powder is slowly added to the proper dosage from a small hopper using an auger while blending the mixture. Sulfur containing reagents are then added in the same manner to the proper dosage. After thorough blending to complete the amalgamation, cement mortar (e.g., 2 kg per 10 kg of amalgam) is added to the mixture for solidification. The agitator is withdrawn and the container is sealed and removed from the scale to cure prior to final disposition. The entire operation is conducted in an enclosure with proper ventilation and emission controls.

In this latter process, sulfur and sulfides are added to fixate small amounts of remaining metallic mercury, as well as non-metallic mercury and other metals and radionuclides. The metals and radionuclides are converted to sparingly soluble metal sulfides or metal sulfide type complexes. Sparingly soluble salts have very small solubility products (K_{sp}). Solubilities of most heavy metal sulfides are less than the corresponding hydroxide or carbonate salts.

In the MER02 project, IT used several sulfur-based fixation reagents. Similar reagents are used in this project.

2.5 Laboratory and Engineering Analysis Approach

The general flow of the laboratory process is shown in Figure 2-1. The soil characterization is described in Section 4 and the soil treatment is described in Section 5. As seen in Figure 2-1, mercury-contaminated soil samples will be received at both IT's and NFS's treatability laboratories; five and fifteen gallons of soil will be received, respectively. At the IT facility debris will be removed by a sieving operation while at NFS, all of the material will be size reduced in a jaw crusher. Both companies will homogenize all their particle size reduced soil, submit samples for characterization analysis, then treatability studies will be conducted. IT will exclusively use its internal analytical laboratory; NFS will use in-house analytical to conduct process refinement then an Utah certified analytical laboratory to verify the results. The two companies will analyze the results, define the treatment parameters then scale-up the

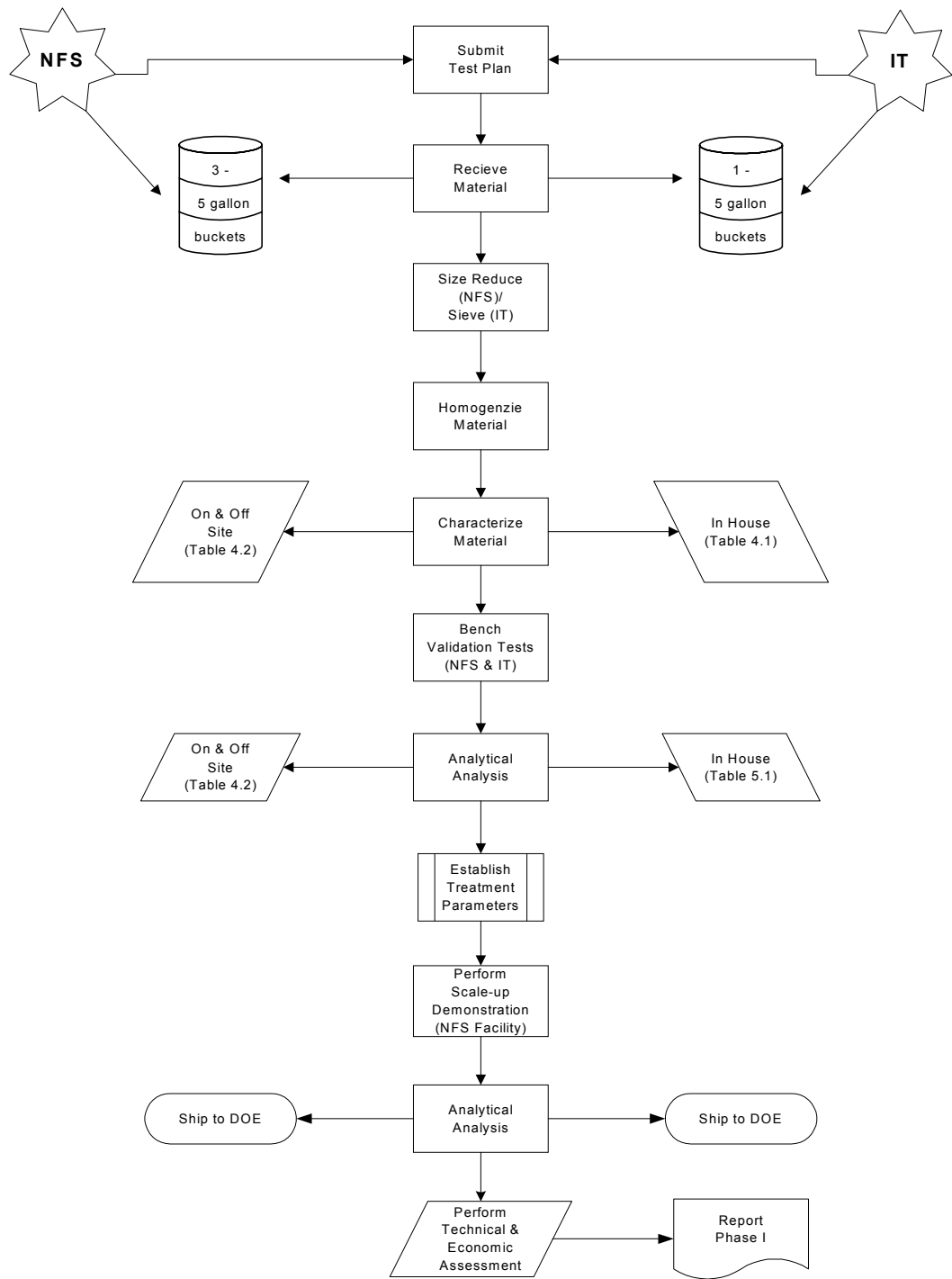
optimized process to approximately 15 to 20 kg size treatment. This larger size batch treatment will be conducted at the NFS facility. Two grab samples from this larger batch will be collected and analyzed at the Utah certified analytical laboratory. All results will be analyzed, the engineering and cost analysis performed, and the topical report written and submitted to DOE.

Since the technologies proposed for the commercial and bench-testing are both batch mix processes, scale-up is greatly simplified. The most important result from the bench-scale testing is identification of an effective treatment formulation. This includes types of reagents, reagent ratios and addition order, as well as mixing times and mixing intensity. These treatment parameters scale directly from the small test batches to the large-scale commercial systems.

The stabilization chemistry or formulation can be implemented, at commercial scale, by both in-situ and ex-situ techniques. In either case, reagent mass ratios (reagent to initial untreated soil) and reagent addition order will be the same as for the bench tests. Reagent mass ratio may, in some cases, be increased to account for less efficient mixing at the full-scale.

For the in-situ treatment approach, IT has proposed to use sheet pile to contain a segment of the soil to be treated inside of a mixing cell. Augers are used to incorporate the stabilization reagents into the soil. Bulk densities and volume ratios that are obtained directly from the bench-scale tests will be used to determine the size of the mixing cell. While rigorous scale-up of bench mixing procedures to the commercial auger system is not feasible, IT/NFS has extensive empirical experience in extrapolating bench tests to full-scale. In some cases, mixing time or reagent ratios will be adjusted to offset potential mixing inefficiencies at the

Figure 2-1
Technical Approach



full-scale. The power required for the mixing augers and the production rate of this system will be based on IT/NFS experience with other full-scale operations.

For an ex-situ approach, IT/NFS has proposed a pugmill based stabilization system. These batch systems scale almost directly from the small bench test batch procedures. The volume of the pugmill and batch cycle times will be based on the treatment formulation and past experience with similar soils.

In addition to treatment performance data, the bench tests will produce process control data on vapor pressure of mercury during soil treatment. This data will be used to design containment and mercury abatement equipment. This will include dust control measures and mercury removal from equipment containment vents.

The results of the bench-scale testing will allow IT/NFS to estimate the cost of soil treatment with a high degree of certainty.

2.6 *Relevant Chemistry*

Amalgamation of mercury has been well described by DOE and other sources.

Amalgamation often lowers the leachability of mercury by 100 to 1000 fold. The liquid mercury is converted to a solid metallic species in this process. An excellent DOE review of amalgamation is in the 1996 Mixed Waste Focus Area (MWFA) document at <http://wastenot.inel.gov/mwfa/acrobat/tsmainl.pdf>. This document also states that mercury is one of the most difficult hazardous materials to treat. This is why IT/NFS not only relies on amalgamation, but also on chemical fixation of the mercury to ensure that the leachability of mercury is controlled by the addition of sulfides or phosphates. A description of sulfide based chemistry follows. (References 10 and 11)

In the sulfur/sulfide process, mercury-contaminated soils or wastes are initially soaked in fixation solutions containing sulfur compounds to convert the mercury and other metals to sparingly soluble metal sulfides or metal sulfide type complexes. Sparingly soluble salts have very small solubility products (K_{sp}). Examples of the solubility product for metal

sulfides are shown in Table 2-1. Figure 2-2 (U.S. EPA, Federal Register. 52[155]: 29999 [Aug. 12, 1987]) shows the solubility of several RCRA metals hydroxides, carbonates, and sulfides as a function of pH. Analysis of Figure 2-2 indicates that the solubilities of most heavy metal sulfides are less than the corresponding hydroxide or carbonate salts.

Typical sulfur-based fixation reagents for this project may include solutions of BEST calcium polysulfide (CaS_4), Degussa TMT, and Nalmet 8154. Details for each reagent are presented in Table 2-2.

The combination of amalgamation, sulfide treatment and other proprietary reagents, if needed, allows for the consistent treatment of mercury well below the performance criteria in this statement of work.

Table 2-1

Solubility Product for common metal sulfides

Compound	Ksp	Compound	Ksp
Al ₂ S ₃	2.0×10^{-7}	MnS	1×10^{-11}
Bi ₂ S ₃	1×10^{-96}	HgS	3×10^{-52}
CdS	7.0×10^{-27}	Hg ₂ S	3×10^{-20}
CoS	5.0×10^{-22}	NiS	2.0×10^{-22}
CuS	8.0×10^{-36}	Ag ₂ S	8.0×10^{-50}
Fe ₂ S ₃	1.0×10^{-88}	Tl ₂ S	1.2×10^{-24}
FeS	3.0×10^{-17}	SnS	1.3×10^{-27}
PbS	1.3×10^{-28}	ZnS	1.6×10^{-23}

Figure 2-2. pH versus Concentration of Dissolved Metal (mg/l)

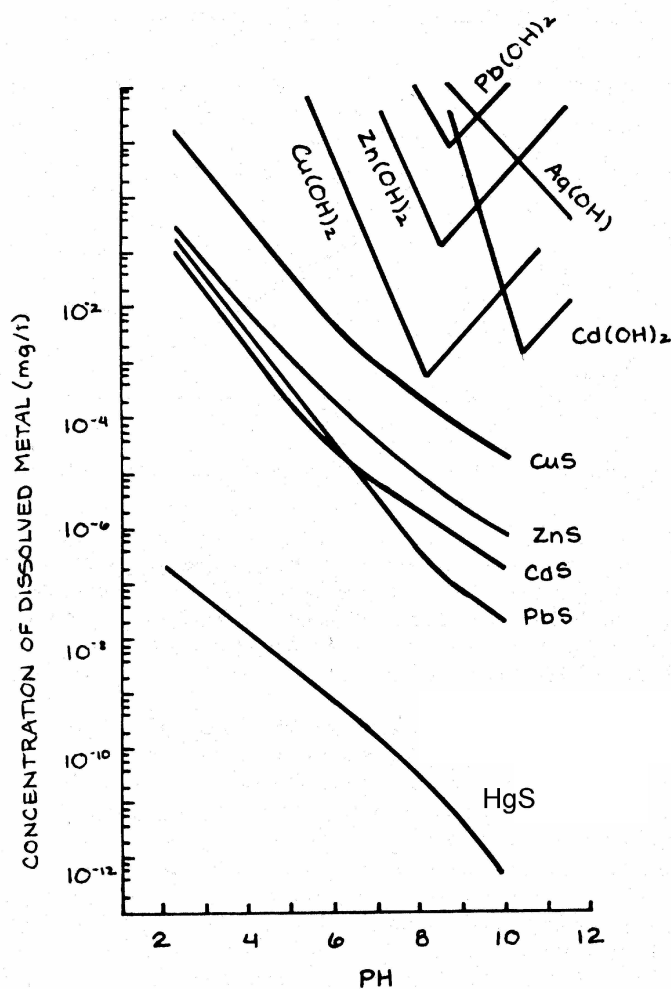


Table 2-2
Typical Sulfur/Sulfide-Based Mercury Fixation Reagents

<i>Calcium</i> Polysulfide	Soluble polysulfide releases reactive sulfides to fixate ionized mercury.
Degussa TMT	Commercial polymer that is very effective at precipitating many heavy metals, including mercury, and radionuclides; the chemical name is trimercaptotriazine.
Nalmet 8154	Commercial polymer that is very effective at precipitating many heavy metals, including mercury, and radionuclides; the chemical name is diethyldithiocarbamate.

3.0. CONTAMINATED SOIL RECEIPT AND PREPARATION

3.1 IT and General Requirements

3.1.1 IT Shipping

The handling and transportation of the soils from and back to DOE are described in detail Section 8.0. DOE will provide 5 gallons and 15 gallons of mercury-contaminated soil to IT and NFS facilities, respectively. The soil will be shipped in Department of Transportation (DOT) approved buckets.

Chain of custody forms will accompany the sample shipment. In addition, DOE will supply a completed TDL and NFS sample chemical and radioactivity characterization form or the information needed to fill out the forms. These forms contain the information necessary to identify the contents of the bucket(s), allow for tracking and identification of the bucket(s), completion of yearly treatability reports, and compliance with permit requirements. At the conclusion of the study, all materials will be returned to DOE to await disposition.

3.1.2 IT Sample Receipt

Upon receipt at the TDL or ATL, the soil samples will be given a unique identification number and logged into the TDL and NFS sample inventory systems, respectively. The unique sample number will be used to track materials such as samples, test-residues, test products and reagents throughout the project.

After receipt at the respective facilities, the outside of the bucket will be smeared to measure external radiological contamination and the dose rate at the surface of the bucket will be measured. Until the soil is characterized by IT, the initial data received from DOE will be used for assigning radiological categories to the bucket and any sub-samples taken from the bucket. In a fume hood, the buckets will be opened to visibly assess the condition of the contents. The headspace of the bucket will be analyzed for mercury using the Jerome, or

similar, instruments. A grab sample of sludge will be collected and analyzed for gross alpha/beta activity as a comparison to data supplied by DOE. The mercury-contaminated soils will be stored in a refrigerator (4°C) when not in use.

After the bucket is opened, any debris or plus 0.5 inch oversize material will be removed from the bulk soil by hand sorting and/or sieving and stored separately. The minus ½ inch soil particles will be mixed by hand using a spatula, in a Hobart mixer, or similar type equipment, until it is visually uniform. The homogenization operations will be conducted in a fume hood.

3.2 *NFS Specific Requirements*

3.2.1 NFS Shipping

Following a brief health physics, industrial safety, criticality and environmental safety review of the incoming contaminated soil profiles, NFS will provide permission to ship this material to the Erwin, Tennessee site. Once received at NFS, the contaminated material will be transferred to ATL for the demonstration.

3.2.2 NFS Sample Receipt and Return

NFS project staff will inspect the incoming soil and verify pre-treatment scenarios that are currently envisioned. Following this inspection, NFS will sample the incoming material to confirm both chemical and physical characteristics. All feed material will then be size-reduced to –6 mesh and homogenized.

Once NFS is satisfied with the treatability results, the results will be given to IT and any excess untreated and treated materials will be returned to DOE.

3.3 *Permits and Licenses*

Bench-scale treatability experiments will be conducted at the TDL and ATL facilities. Both facilities have all the required NRC, EPA and State licenses, registrations and permits to

handle, store and perform these treatability tests. Our facilities are in compliance with the pertinent regulatory requirements.

Both facilities meet the licensing and permit requirements specified in the RFP. Specifically, IT has the following.

- Intent to Perform Treatability Testing letter to Tennessee Division of Solid Waste Management dated 8-1-88 for TDL. The facility is authorized to perform treatability studies on RCRA materials under the treatability exemption found in 40CFR 261.4(e) and (f) and fully incorporated into Tennessee Rule Chapter 1200-1-11.
- EPA Identification (ID) numbers: TDL's number is TND000770479.
- U.S. Department of Transportation (DOT) Hazardous Materials Certificate of Registration number 071596 002 005E.
- Radioactive materials license at TDL for the types of radioactive materials stated in the RFP: TDL's license number is TRML R-47055-D05.
- State of Tennessee Licenses for Delivery: TDL's is number T-TN015-L01. A license for delivery authorizes shipment of radioactive waste to facilities in the State of Tennessee.
- Nuclear Liability Insurance: TDL's policy number is NF-0331.

Tennessee Air Pollution Control Regulation 1200-3-0.04(4)(I) provides exemption from the requirement to request a State point source air permit for laboratory testing. An exemption from air permitting requirements must be requested for pilot-scale testing.

NFS ATL has the following permits and licenses.

Solid Waste

EPA ID# TND 003095635

Revision No: 0

Date: 12/14/01

Notification of State Director of Intent to Conduct Treatability Studies

Annual Report to State Director

Liquid Waste

Laboratory liquid effluents will be discharged to the NFS NPDES permitted wastewater treatment facility (TN0002038).

Air Emissions

Air emissions from NFS laboratories are conducted in accordance with the State of Tennessee Air Pollution Control Regulations.

Radioactive Materials License/Treatability Programs

R-86008-J99 issued by the Tennessee Department of Environment and Conservation, Division of Radiological Health.

4.0. SOIL CHARACTERIZATION

4.1 IT (TDL) Soil Characterization

The general flow of the laboratory process is shown in Figure 2-1. The soil characterization is described in Section 4 and the soil treatment is described in Section 5.

- IT will characterize the soils and compare these results to the chemical, radiological, and physical characterization data supplied by DOE.

The soil will be inspected to visually identify the presence of elemental mercury.

The physical characteristics will be used to determine how the sample will be handled. The radiological data will be used for permitting, lab procedures, radiation work permits (RWP), and licensing requirements. The metal content will be used to define the amount of reagents used in the screening formulations and to determine which metals will be analyzed in the treated materials. Except for mercury, metals that are not present or whose total concentration is less than 20 times the TCLP limit will not be analyzed in the bench-scale treatment tests.

The initially sieved soil will be stored in a walk-in refrigerator when not in use. Rocks, twigs, wood, metal, or debris greater than 0.25 inches will be removed by hand and/or sieving before the characterization analyses. The soil will be mixed until visually homogeneous prior to characterization analyses and bench-scale stabilization treatability testing. The soil will be homogenized by first mixing with a spatula followed by blending for approximately 15 minutes in a Hobart planetary mixer. The soil will be homogenized by hand if the soil is too wet, tacky, or cohesive to mix in the Hobart mixer.

Two aliquots of the homogenized materials will be collected for in-house characterization analysis, as defined in Table 4-1. The table lists the tests, methods, purposes for the tests, and the analytical levels of the tests. As indicated in Table 4-1 a relative measure of mercury volatility will be determined by transferring an aliquot to a container with a tight fitting lid

and allowing it to equilibrate overnight at room temperature. The mercury vapors in the headspace will be measured with a Jerome mercury vapor analyzer (MVA). In addition, the soil will be analyzed for RCRA metals, physical consistency parameters, metals, and radiological analyses. The leachability and the pH of the final extract will also be measured by an in-house procedure, modified TCLP (MTCLP). This procedure is the same as the SW-846 method 1311 except:

- Smaller sample size is used, typically 20 to 45 grams, instead of 100 grams
- The samples are shaken, not tumbled
- Only metals are analyzed for in the leachate.

The MTCLP is used to minimize volume of secondary waste generation. The secondary waste generation is decreased by approximately 60-80 percent. The measured characterization data will be compared with the supplied chemical, radiological, and physical characterization data supplied by DOE.

Table 4-1
Characterization Analyses

Parameter	Method	Purpose	Analytical Level ¹
Percent Solids/Moisture	ASTM D2216	Calculate amount of water to add and process information	3
Liquid Limits (soils and sludges only)	ASTM D4318	Process information	3
Bulk Density	ASTM D5057	Process information	
Mercury in Head Space (~ 200 g in 8 oz specimen cup)	Jerome Model 431-X Mercury Vapor Analyzer	Determine volatility of Hg Process information	2
PH (1 part waste to 10 parts water)	Solid Waste (SW) 846 Method 9045B, modified	Process information, Control Sulfide emissions	2
Total Selected Metals Analysis (8 RCRA metals, Th, U)	SW 846 Methods 6010A & 7471A	Baseline metals	3
Modified TCLP for 8 RCRA Metals	SW 846 Methods 6010A & 7471A and Modified 1311	Baseline leachability	2
Gross Alpha/Beta	Standard Operating Guideline (SOG) TALRAD0012	Health & Safety, License compliance	1
Gamma Spectroscopy	Equipment manufacturer's method	Health & Safety, License compliance	1
¹ Analytical Level 1 is screening level, semi-quantitative analyses, performed in accordance with industry accepted procedures using little or no quality control. Analytical Level 2 is semi-definitive, quantitative/qualitative analyses, performed in accordance with approved or modified methods with minimum quality control. Analytical Level 3 is definitive, quantitative/qualitative analysis, performed in accordance with approved methods.			

4.2 *ATL Characterization and Sample Preparation*

The DeHg® soil stabilization reactor can accept up to 100 pounds of screened material whose size is less than 6 mesh (0.132 inches). To comply with this requirement, NFS intends to grind raw test soil feed, if necessary, to -6 mesh prior to testing. A 150-kg/hour-jaw crusher will perform this operation. Once reduced, feedstock will be inventoried for the subsequent tasks. NFS will composite all 15-gallons of received soil before conducting chemical analysis of the soil. All grinding and homogenization operations will be conducted in a specially designed containment area (described in Section 5). Airborne mercury will be monitored in the containment area during operations. Historically the mercury levels in the containment area are well below Occupational Safety and Health Administration (OSHA) standards.

Grab samples of incoming soil will be analyzed by NFS (on-site) and also sent to an approved laboratory (off site). NFS will perform TCLP mercury analyses on the feed; the off-site laboratory will perform TCLP assay for mercury and all RCRA metals as well as total mercury. The pH of the TCLP extract will be reported. These values will serve as a baseline for treatment performance and provide data needed to determine starting reagent dosages for chemical stabilization. The analytical methods and types of characterization are described Table 4-2. .

TABLE 4-2
NFS Sampling and Analysis Matrix

Description	Number of Samples, est. (on-site in par.)	Sampling Technique	Off-Site Analyses	NFS Analyses
Initial Characterization	2 (2)	Grab	Total Hg; TCLP Hg, other RCRA metals	TCLP Hg
Scoping Tests	0 (12)	Grab	None	TCLP Hg
Validation Tests (20 kg)	0 (4)	Grab	None	TCLP Hg
Proving Tests (40 kg)	2 (optional) (4)	Grab	TCLP (optional)	TCLP Hg
Final Waste Form	3 (3)	Grab	Total Hg; TCLP Hg, other RCRA metals	TCLP Hg
Secondary Waste from Testing	0 (4)	Grab	None	TCLP Hg
<p>Assumptions in designing test matrix:</p> <ul style="list-style-type: none"> ▪ All material will be ground to –6 mesh before analysis. ▪ There are up to (6) Scoping Tests (duplicate analyses). ▪ There are three (3) Validation Tests ▪ There are two (2) Proving Tests . ▪ The Project Lead/Chemist is responsible for evaluation of any Quality Control sample results, initiation of appropriate corrective actions, and for ensuring the overall quality of the mercury analyses. The actual number of samples taken for testing may be modified only by the Project Chemist depending upon early test results. 				

5.0. BENCH-SCALE TESTING PROCEDURES

Bench-scale studies will be conducted at IT's TDL in Knoxville, TN and NFS's ATL in Erwin, TN. All in-house metals analyses will be performed at these locations. Small-scale fixation tests will be conducted at both laboratories, while a series of larger size treatments (e.g., 15 - 25 kg) will be conducted at ATL. ATL is designed for conducting pilot-scale demonstrations and has segregated test areas equipped with special ventilation and spill control capabilities for such demonstrations.

5.1 IT Experimental Procedure

The following describes IT's procedure for IT bench-scale stabilization tests. Aliquots of the soil will be removed from the refrigerator and allowed to warm for at least two hours before further testing. Experiments will be conducted where up to three dosages of fixation reagents will be well mixed with each soil. After treatment, IT will visually inspect the product for elemental mercury.

Stock solutions of fixation reagents and water will be added to soil. Up to three different loadings of each fixation reagent, (e.g., calcium polysulfide, Degussa TMT, Nalmet 8154, and/or other proprietary reagent[s]), will be investigated. The percentage loading may be based on stoichiometric reactions or ratios based on the weight of the soil. The major metals defined for this project are mercury, along with the seven other RCRA metals. Enough stock solution and water will be added so that the soil exceeds the liquid limit moisture content and produces a fluid product. A cover will be placed over the container. This slightly fluid to fluid system will be shaken for ten minutes. As indicated in Table 5-1, the mercury vapor concentration in the headspace of the shaken samples will be measured using a Jerome MVA.

Table 5-1 lists all the analytical measurements for the bench-scale treatment process. The overall approach to our program is to conduct scoping experiment(s), then optimization experiments, and then scale-up to the pugmill-sized process. Initially, IT/NFS are applying their well-developed generic formulations to the soil; the project chemist may modify the amounts and number of reagents and number of experiments to achieve the performance criteria. Table 5-2 lists an estimate of the number of samples that will be prepared.

Table 5-1.
Treated Sample Analytical.

Analysis	Sample with Fixation Reagent @ 10 Minutes Stirring	Sample after 1 to 7 Days Cure	Method	Analytical Level
Mercury in Headspace	X	X	Jerome Model 431 MVA	2
MTCLP for Hg and RCRA Metals ¹	—	X	SW 846 Methods 6010A & 7471A	2
TCLP for Hg and RCRA Metals and Uranium ¹	—	X	SW 846 Methods 6010A & 7471A	3
¹ Measure RCRA metals that have total concentration values greater than 20 times the TCLP regulatory limits.				

Table 5-2
IT Testing Matrix

Description	No. Test s	Weight of Soil Tested, KG	Objective
Scoping Tests	15	0.1-0.3	Evaluate processing variables “reagent dosage” and “mixing time”.
Validation Tests	3	0.3	Replicate tests of preferred Scoping Test recipe.

Each mixture will be well stirred by hand with the selected additive, and then approximately half of the contents will be transferred to another container. Each container will be capped and shaken for an additional ten minutes. In one of the two capped containers for each formulation, the mercury vapor concentrations will be measured in the headspace of the containers using the Jerome MVA. The mercury headspace concentrations will be compared with each other and the untreated feed. It is expected that the mercury vapor concentration will be significantly less than the original untreated feed due to chemical reactions with the reagents and the wetting of the soil or waste. The other sample containers will be used for MTCLP analyses.

In the bench-scale screening study, the MTCLP extracts will be digested using a microwave technique, Modified SW-846 Method 3015. This microwave digestate will be split into approximate halves for mercury and RCRA metal analysis. To the first half of the digestate, the appropriate oxidizing agents, etc., will be added to the aliquot for mercury analysis prior to analyzing it by cold vapor atomic adsorption. The other half of the extract will be used for the other RCRA metals analyses. After digestion, it will be ready for analysis without further chemical treatment. The MTCLP digestates will be filtered through a 0.6 to 0.8 micron filter and diluted appropriately prior to analysis on the inductively coupled plasma (ICP) or cold vapor atomic adsorption (CVAA) instruments.

5.2 *NFS Scoping Experimental Procedure*

Samples of the minus 6 mesh size-reduced material will be submitted to a short series of bench validation tests to verify conditions of chemical dosage and reaction time. Once the scale-up parameters are established from these tests, bulk soil will be loaded into the DeHg® reactor in nominal quantities of 15 kg up to 25 kg and stabilized. Samples of material will then be taken for process control analyses. NFS will also provide specimens to a certified laboratory for secondary analysis and comparison. Unlike IT, the mercury headspace analyses will not be measured. In the previous MER03 study, NFS demonstrated that mercury emissions were very low during processing.

The NFS scoping test will use about 0.1 to 0.3 kg of contaminated soil. Small batch mixing systems will be used. It is anticipated that this task will be comprised of up to six (6) tests to evaluate the processing variables, reagent(s) dosage, and mix time for this particular soil or waste stream. NFS will perform TCLP leaches on the residuals from these tests to assess leachable mercury content. Residuals will also be visually inspected for elemental mercury. Based on the results of these tests, NFS will proceed with the scale-up demonstration, described in the subsequent section. Depending upon results of prior scoping tests, the Project Chemist may request additional tests. Table 5-3 lists an estimate of the number of samples that will be prepared.

Table 5-3**IT Testing Matrix**

Description	No. Tests	Weight of Soil Tested, KG	Objective
Scoping Tests	15	0.1-0.3	Evaluate processing variables “reagent dosage” and “mixing time”.
Validation Tests	3	0.3	Replicate tests of preferred Scoping Test recipe.

5.3 *NFS Soil Stabilization Scaleup Demonstration*

5.3.1 System Layout

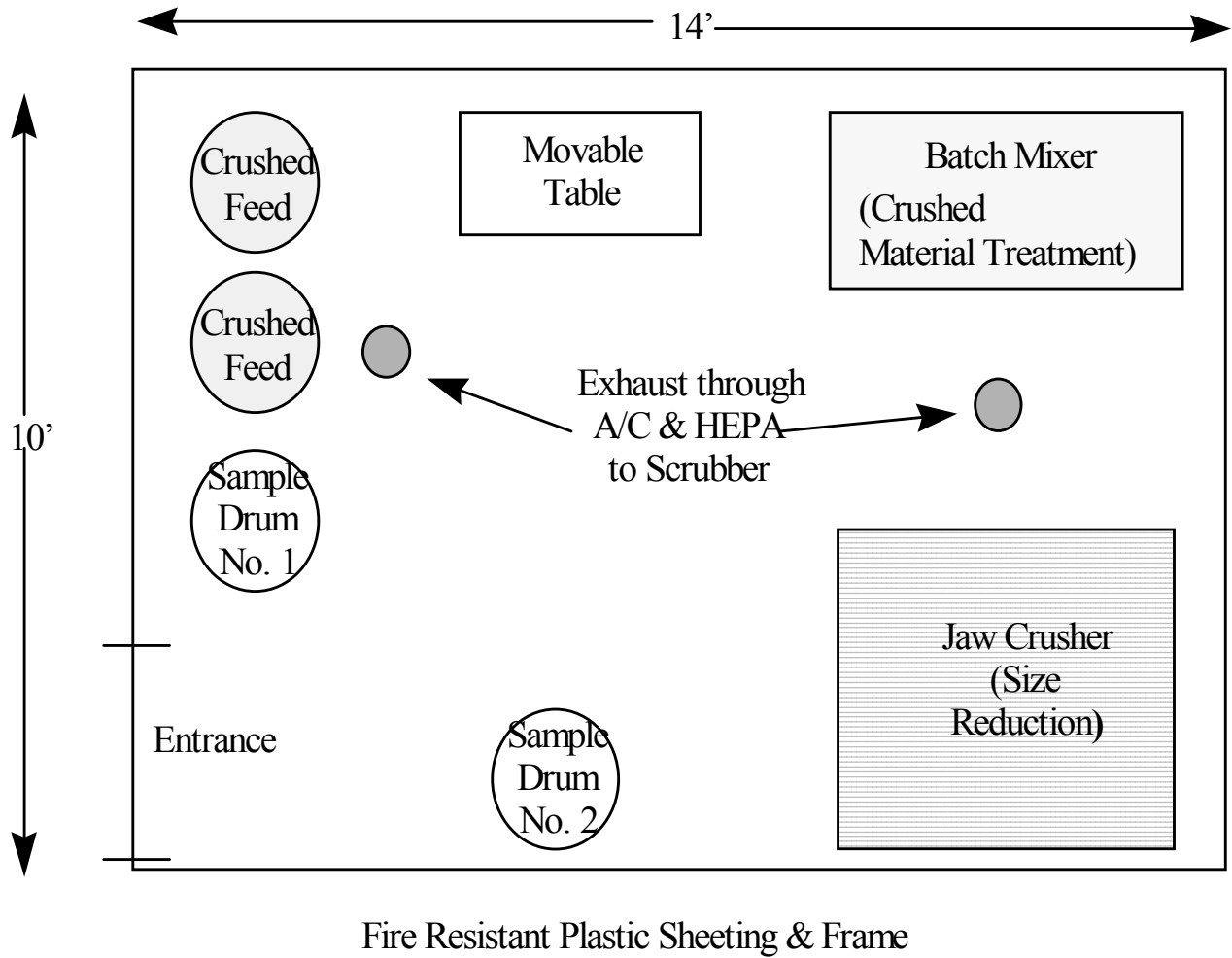
A general layout of the Demonstration Facility is depicted in Figure 5-2. This will be a ventilated containment area of approximately 140 ft² built specifically for this purpose. Within the containment area will be the following equipment: jaw crusher, DeHg® batch soil stabilization reactor, and soil inventory tanks. All soil stabilization tests will be performed at ambient conditions in an enclosed reactor vessel.

Grab samples of treated material will be taken and submitted to TCLP testing for process control purposes. At the option of the Project Chemist, grab samples from proving tests may also be submitted to an outside laboratory. Based on early process control analytical results, it may be necessary to adjust treatment parameters. Based on inspection and characterization of the soil, the Project will make this determination.

The general protocol for testing will be as follows:

1. Add designated quantity of soil to the reaction chamber.
2. Add the prescribed weight of reagent(s) to the soil in the reaction chamber.
3. Blend for the designated time.
4. Add additional reagent(s) as necessary.
5. Unload the stabilized material.
6. Sample for TCLP (SW-846, Method 1311).
7. Analyze for mercury in TCLP extract.

FIGURE 5-2
General Demonstration Facility Layout



5.4 *Decision Point*

The combination of leaching results, control of mercury concentration in the headspace, and cost of reagents will determine the selection of reagents for the demonstration phase. On completion of the bench-scale treatability study, reports will be submitted and DOE will review and decide if the IT/NFS Team will proceed to Phase II.

“At this time, the DOE will evaluate the merits of the project to determine if, and to what extent, Phase II shall be conducted. The contractor shall not proceed with these Phase II tasks unless the Contracting Officer issues a modification to the contract to exercise Phase II. If the Government elects not to pursue the Phase II tasks, the contractor shall complete all of the remaining work defined in the Phase I contract.”

6.0. *SAMPLING AND ANALYSIS*

The number and type of samples are detailed in their respective sections, i.e., Sections 4 and 5. Further details on chain of custodies are provided in Sections 9 and 10 (the QAPP sections.)

6.1 *Sampling and Analysis Procedures*

Sampling procedures, sample type and number of samples are specified in Sections 4 and 5. All IT testing analyses will be performed by the Technology Applications Group (TAG) at the TDL or IT's Environmental Technology Development Center (ETDC) in Oak Ridge, TN. NFS testing will be at ATL or at an off-site production analytical laboratory, Enviro-Test, LLC., for final certification.

Samples collected will have a unique sample identification which describes or relates to specific information about the sample, including at a minimum, description of sample, date of collection, name of sample collector and an indication of hazards associated with the sample.

Samples which are generated by the TAG or ATL personnel in the course of testing, that are analyzed either internally or submitted to an outside laboratory, will be noted in laboratory notebooks with a description of the sample and a sample number or designation.

6.2 *Sample Custody*

A strict chain of custody record shall be maintained for all samples received at both laboratories. The treatability facilities are controlled-access facilities, with multiple secured sample storage areas.

Sample transfers external to the treatability facility will be accompanied by an IT combined Analysis Request and Chain of Custody Record. Samples transferred to the treatability analytical department or Geotechnical Laboratory will also be accompanied by an IT

combined Analysis Request and Chain of Custody Record. Instructions for completing the form are on the back of the form.

Copies of all Analysis Request and Chain of Custody Records will be maintained in the project file.

Procedures for laboratory sample receipt, custody, coding, tracking, handling, and storage are in IT's Standard Operating Procedures (SOP) PDGADM0005.

Material received by NFS for treatability studies must conform to the requirements of NFS procedure *Receipt/Shipment of Sample Materials for Development/Treatability Studies, NFS-ACC-87, Rev. 1*. This procedure describes, in part, the requirements for material receipt, maintenance of material tracking and custody. The "how to" part is documented in the *Hazardous Waste Treatability Sample Documentation, Standard Operating Procedure 387*. Two key documents are used to track and maintain custody of the treatability material: (1) the Daily Hazardous/Mixed Waste Treatability Study Facility Logsheet, and (2) the Hazardous/Mixed Waste Treatability Study Log.

6.3 Contaminated Soil Characterization Sampling and Analysis

Material will be removed from the DOT-approved bucket as described in Sections 3 and 4. This material will be size reduced, homogenized, sampled and analyzed as described in Section 4.

6.4 Bench-Scale Sampling and Analysis

The tables in Section 5 show the samples to be collected and analyses to be conducted on samples of treated soils and/or material during the bench-scale treatability study.

7.0. TOPICAL REPORT

NFS will provide a report addressing test results, material amounts, and any possible recommendations for Phase II. IT will incorporate this report into an overall report for the Phase I project.

At the completion of the study, a report will be prepared detailing observations and procedures from the treatability study, and the results of the analytical tests. This report will include specific recommendations concerning full-scale treatment of the soils. The report will include, but is not limited to the following sections:

1. Introduction
 - Purpose and objectives
 - Summary of testing and results
2. Mercury stabilization technology description
3. Treatability study approach
 - Experimental design and procedures
 - Equipment and materials
 - Deviations from the work plan
4. Treatability test results
 - Notes and observations
 - Analytical results
5. Conclusions and recommendations
6. Quality Assurance and Quality Control
7. Appendices
 - Batch data sheets
 - COCs and Analytical reports
 - IT/NFS Work Bench Test Work Plan

8.0. WASTE MANAGEMENT

Approximately 46,800 m³ of mercury-contaminated low-level waste exists in the DOE complex, mostly as debris, soils, sludges, and wastewaters. After reviewing DOE-provided analytical data and performing appropriate chemical and physical characterizations on sample materials, IT/NFS will perform bench-scale demonstrations of a batch mix stabilization system for treatment of the DOE mercury-contaminated soils and sediments. IT/NFS believes that a combination of amalgamation and fixation chemicals is the technology that is most likely to be applicable to the full range of DOE soils, sediments, and other wastes.

8.1 Residuals Management

8.1.1 Management Plan Overview

This project involves treatability testing of a sediment sample from DOE's Oak Ridge complex that will generate four residual streams. When testing is completed, all residuals will be characterized for DOE compliance and returned to DOE.

IT's Residuals Management Plan was developed to ensure that throughout the demonstration, waste minimization technologies are utilized to the fullest and that residuals collected at the end of the demonstration are properly managed. In addition, the Plan will ensure that the demonstration is conducted in full compliance with the regulations of the Tennessee Division of Solid Waste Management and the Tennessee Division of Radiological Health.

The elements of this plan include:

- Responsibilities and training
- Sample management and storage
- Waste minimization
- Estimated residual quantities
- Residuals characterization and classification
- Residual storage and return
- Effluents
- Emergency response and spill management.

8.1.2 Responsibilities and Training

IT's Regulatory Compliance Specialist (RCS) will oversee the receipt, on-site management and return of sample and residual materials for the TDL. Employees of the TDL have received facility-specific training in RCRA hazardous waste compliance, radioactive materials handling, and DOT hazardous materials general awareness.

8.1.3 Sample Management and Storage

DOE, or their representative, will transport one 5-gallon DOT authorized bucket of mercury contaminated soils to IT TDL and three 50gallon buckets of soil to NFS ATL. At IT, the container containing the soil will be stored in the walk-in cooler while awaiting testing, and in Laboratory 5 where the demonstration-scale work will be performed. During the project, portions of untreated or treated sediment may be transported by IT vehicle, with chain-of-custody documentation and in compliance with applicable DOT requirements, to the ETDC for geotechnical testing. All materials will be returned from the ETDC to the TDL in a similar manner after this phase of the study is complete. No materials will be retained at the ETDC. See Section 8.2.9 for more details.

At NFS, material is shipped using procedure NFS-ACC-87. The samples will not be stored in the refrigerator. This requires the generator to provide a profile to NFS for the incoming material. The profile undergoes a H&S review, and then NFS issues formal permission to ship the material. Once the soil is off-loaded at the site, it is moved to the designated facility for further testing and evaluation. Shortly after receipt, sample aliquots will be taken for in-house characterization analysis per the Work Plan. Transfers will be coordinated, prepared, recorded, and supervised by the RCS. At both facilities, any generated liquids, such as leachate, will be stored in secondary containment capable of holding 100 percent of the volume to prohibit the spread of contamination in the event of a spill.

8.1.4 Waste Minimization

The TDL Waste Minimization Policy is ". . . to reduce all radioactive, hazardous, and nonhazardous wastes to the minimum levels technically and economically practical while maintaining full compliance with all federal and state waste regulations." This Policy is driven by management's awareness of IT's environmental responsibility, public image, long-term liability, and waste management costs. The policy is implemented by the concerted efforts of the IT technical staff. The TDL has demonstrated that performing initial bench-scale treatability

tests with reduced sample quantities, utilizing effective nonhazardous chemical substitutes whenever possible, and considering beneficial reuse or reclamation options are all effective means to minimize the quantity of hazardous and radioactive wastes generated. These and other waste minimization techniques have been incorporated into the Work Plan to eliminate hazardous wastes and reduce radioactive waste volume.

8.1.5 Estimated Residual Quantities

Previous project experience enables IT to estimate the types and volume of residuals expected from testing these sediments. Based on information provided by DOE and the IT Work Plan, four residual streams have been identified and quantified. Descriptions and estimated quantities of each are shown in Table 8-1.

Table 8-1. Summary of Estimated Residuals to be returned to DOE		
<i>DESCRIPTION</i>	ESTIMATED QUANTITY	
	IT	NSF
Treated Material	6 kg	150 kg
Untreated Material	38 kg	<20 kg
Incidental Materials (leachates, filters, syringes)	32 kg	35 kg
PPE, Contact Waste	50 kg	<20 kg

8.1.6 Residuals Characterization

Unless further testing is required by DOE, the RCS will classify the residuals based on data provided by DOE, process knowledge, and post-treatment analyses. The treated sediment will be subjected to the TCLP. A summary of final residual quantities and characteristics will be submitted to DOE.

8.1.7 Residuals Storage and Return

All residuals will be properly containerized in appropriate shipping containers, segregated, and accumulated in the TDL or ATL. The residuals will be returned to DOE at the end of the project. The shipments will be carried out in accordance with the IT Transportation Plan.

8.1.8 Effluents

Emissions of radioactive materials in effluents from the TDL are regulated by the TDRH. No radioactive liquids are discharged from the facility to the ground or sewer. As indicated in Table 2-1, leachates from the demonstration will be collected and returned to DOE by IT. Gaseous emissions are discharged following treatment through vents that are continuously sampled for radioactive materials to ensure compliance with the Tennessee State Regulations for Protection Against Radiation.

Based on Tennessee Air Pollution Control Regulations and on TDL's off-gas treatment system, no air discharge permit or associated monitoring is required.

8.1.9 Emergency Response and Spill Management

The TDL has a current Emergency Action Fire Prevention Plan (EAFPP) that is designed to adequately cover projects like the current DOE project. This plan is implemented in the case of any emergency or spill at the TDL that may adversely affect human health or the environment. Preparedness or prevention as well as emergency response provisions are addressed in these emergency plans in the event of a fire, explosion, spill, release, or natural disaster. State and local agencies requested to provide emergency assistance are listed with their phone numbers in the EAFPP, and these names and numbers are posted adjacent to facility telephones. TDL personnel are currently trained and familiar with the procedures of the EAFPP. IT's project-specific Health and Safety Plan (HSP) identifies potential hazards associated with the project, thereby increasing the worker's preparedness and prevention awareness. Trained Primary and Alternate Emergency Coordinators have been assigned for 24-hour response, and emergency response equipment is readily accessible in all work areas.

8.2 *Transportation Plan*

8.2.1 Transportation Plan Overview

The Transportation Plan describes the requirements and responsibilities for transportation of DOE's sediment samples and residuals. Specifically, this plan addresses:

- Responsibility for transportation activities

- Collection, packaging, and documentation for transfer to TDL
- Radioactive material "authorization numbers"
- Transportation of DOE sediment to the treatability testing facility
- Transfer of samples between IT facilities
- Return of residuals.

8.2.2 Responsibilities

All shipments of samples from or between IT facilities will be conducted under the supervision of the designated IT Transportation Manager, Linda Lawhorn. Mrs. Lawhorn is a Certified Hazardous Materials Manager (CHMM) and has completed and taught numerous specialized training courses in the shipment of hazardous and/or radioactive materials and wastes per DOT, EPA, and IATA.

8.2.3 Collection, Packaging, and Preparation for Transfer to TDL

DOE sediment will be collected, packaged, marked, labeled and documented by DOE and transported to IT and NFS laboratories. IT will arrange transportation from TDL to the ETDC for geotechnical testing, if needed. As the party offering the initial shipment, DOE will be responsible for determining the proper shipping name based on DOE's knowledge of the material. The IT Transportation Manager will provide assistance in determining the proper shipping name if DOE requests it.

8.2.4 Radioactive Material Authorization Numbers

Each shipment of licensable quantities of radioactive materials to the TDL must be preapproved and assigned an Authorization Number. Authorization Numbers are assigned by the facility Radiation Safety Officer (RSO) or his designee. Shipments containing radioactive materials that are sent to these facilities without an Authorization Number will be returned. Authorization numbers must be on the sample container(s), on the shipping papers, or otherwise forwarded to the receiving facility prior to the scheduled receipt of the material. Authorization numbers for shipments of samples to, from, and between IT facilities will be obtained by the project Transportation Manager. NFS has similar procedures.

8.2.5 Transportation of DOE Sediment to the TDL

Any local transfers to, from, and between IT facilities that are made in an IT vehicle will be driven by an IT employee who is current in the general awareness/function-specific/safety

training required under 49 CFR 172.702. Any transfer to, from, and between IT laboratories will be made in accordance with applicable DOT requirements. All sample transfers will be accompanied by chain-of-custody documentation, bills-of-lading if required, radiological screening data, and package radiation surveys. Authorization numbers will be assigned by the IT facility RSO or his designee to all licensable radioactive materials prior to transfer. These authorization numbers will accompany the samples. The transfer of the DOE sediment to TDL will be coordinated with the designated IT Transportation Manager. Three 5-gallon pails are expected, weighing approximately 26 kg each when filled with DOE sediment.

8.2.6 Transfer of Samples

Should it become necessary to transfer treated or untreated materials to ETDC for geotechnical testing, aliquots will be classified, packaged, marked, labeled, and documented in accordance with 49 CFR. Samples will be appropriately cushioned prior to transport. After testing is complete, samples and sample containers will be returned in a similar manner to the TDL.

8.2.7 Return of Residuals to DOE

Return of all residuals to DOE will be coordinated by the IT Transportation Manager and returned to a DOE-designated location. IT will request the receiving facility's radioactive materials license number or verification of its exemption from licensing regulations prior to offering for shipment. DOE will be notified at least one week prior to the return. The residuals will be returned in accordance with 49 CFR via an IT vehicle and will be accompanied by chain-of-custody documentation, bills-of-lading, radiological screening data, and package radiation surveys. Table 8-2 is similar to Table 8-1 but includes the expected DOT classification, packaging, marking, labeling, and documentation that will be used for the residual return.

Table 8-2.
Summary of Applicable DOT Requirements for
Residual Streams to be Returned to DOE

Requirements assume one 5-gallon pail of mercury-contaminated soil weighing 26 kg. Requirements for the non-soil residual streams can be calculated in similar per-container fashion.

Material	Non-regulated	Regulated
Classification	If materials measure less than 5000 ppm (0.5%) mercury, the material will not meet DOT's definition of Class 6 or a RQ. Existing analyses demonstrate that the material does not meet the definition of Class 7.	If the materials measure 5000-20,000 ppm (0.5 – 2%) mercury, the material will ship as a DOT Class 6, Packing Group III; >2% up to 16.67%, as Packing Group II; and ~20% and higher as a Packing Group I. If the level of mercury equals or exceeds 17000 ppm (1.7%), the material will also be a RQ. Shipper should use this basic description: "Mercury compounds, solid, n.o.s., 6.1, UN2025, III", or "I" or "II" as appropriate.
Packaging	Containers of adequate strength to protect the contents.	Limited Quantity exceptions are not available for a 26 kg pail of this material in Packing Groups I or II; this single packaging does not qualify for the Limited Quantity exception for Packing Group III material. This pail must be shipped as fully regulated and it must meet UN-specification 1A2 at a testing level appropriate for the packing group of the material being shipped. See 49 CFR 173.211, .212, or .213.
Marking	To:/From:	Mercury compound, solid, n.o.s., UN2025 To:/From:, UN-specification, RQ, if required
Labeling	None	Class 6 label
Documentation	Chains of custody	Hazmat Bill of Lading and Chains of Custody

9.0. *QUALITY ASSURANCE QUALITY PLAN - IT*

9.1 *Quality Assurance/Quality Control*

IT has an established QA Program that describes requirements, roles, responsibilities, and methodologies for ensuring compliance with DOE Order 414.1 and 10 CFR 830.120. The IT Quality Program will be implemented for Phase I bench-scale testing will be managed by Mr. Patrick Gray, who brings 11 years of relevant experience to the project. This Quality Program has been applied and audited for similar design/build projects by IT nationwide. IT also has an established Nuclear Quality Assurance (NQA-1) Quality Assurance Program Manual. This manual describes our methodology for implementing the requirements of American Society of Mechanical Engineers (ASME) NQA-1-1994. This manual can be used as a template for the development of project-specific QA plans.

The NFS QA Program is based on the requirements and guidance of the ASME NQA-1-1989, which is functionally equivalent in its intent to DOE Order 5700.6C and the requirements in the draft *Test Plan Guidance*. The NFS QA Program is described in more detail in their Demonstration Test Plan.

9.2 *Project Organization and Responsibility*

9.2.1. *Project Organization Overview*

The organization chart shown in **Figure 9-1** identifies the organization to perform Phase I treatability testing work functions. The key personnel include identified members of NFS and IT. Key personnel from IT will include the Project Manager, Principal Investigator, and ES&H, QA and Environmental Compliance Coordination (ECC) personnel. These people have the proven ability to initiate, manage and complete the PRDA, because of the level of education, training, professional registrations, general experience, relevant experience, and longevity with the company represented.

The organizational structure will be kept simple and straightforward with clear lines of authority and responsibility as described in this section. The project is directed by Mr. Steve Downey, PE, who will be responsible for the overall execution of this project. Supporting Mr. Downey will be key staff members from NFS and IT. IT's ES&H Manager and the QA Manager report at the highest level in the IT organization to emphasize their role in all project activities. To maintain independence, the H&S and QA functions have a solid line reporting relationship to their respective corporate Vice Presidents, Mr. Warren Houseman (ES&H) and Mr. Dave Rohm (QA), and a dashed reporting relationship to Mr. Downey.

9.2.2 Responsibilities and Lines of Authority

Project Manager. The Project Manager, Steve Downey, PE, has exceptional experience as a project manager for similar complex projects, including work for DOE and the Department of Defense (DoD). Mr. Downey was selected for this position based on his qualifications, educational background, relevant experience in the type of required work, training, availability, and accomplishments. He has 22 years of experience in environmental program management, project management, business development, and hands-on engineering for over \$200 million in programs with DOE, the U.S. Navy and other government programs.

He has single-point authority and responsibility for contract management and project execution, and he holds full authority to make decisions to set priorities and allocate resources so that work is performed efficiently and effectively. Mr. Downey utilizes a Knoxville-based program management staff with extensive experience in projects similar in size and complexity to that described in the solicitation.

Assisting the project manager will be Corporate Sponsor, Mr. John Razor, who participates in reviews and evaluates information on costs, schedules, and technical performance. He will assure that IT has the best personnel resources dedicated to the project and will exercise his direct authority and experience to effect prompt resolution of issues with DOE NETL.

Principal Investigator. Dr. Ernie Stine is the Principal Investigator. He was selected because of his technical experience and qualifications with 16 years of relevant experience.

He reports directly to proposed Project Manager, Mr. Steve Downey. He will ensure the successful completion of all technical aspects of the project and will monitor most day-to-day operations. Dr. Stine is assisted by experienced IT treatability testing staff who will report directly to him on matters related to the performance of the Phase I work. These staff members include R. Greg Bennett, Ellen Lay and C. Ed Morren.

NSF Testing Personnel. NFS provides staff for the laboratory demonstration test as outlined in this work plan. NFS program management and technical personnel will interact with corresponding personnel from IT and will report to the Project Manager, Steve Downey.

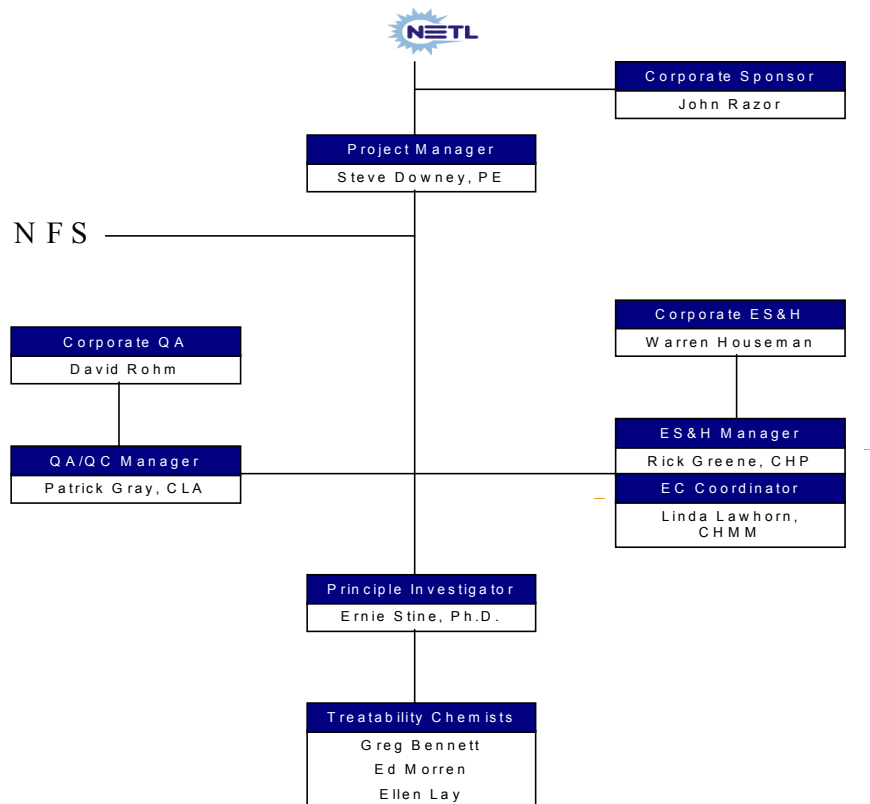
QA Manager. Mr. Patrick Gray is IT's QA Manager. He is a Certified Lead Auditor with 26 years of professional experience. He is responsible for QA matters related to IT's Phase I testing and the Phase II field demonstration testing. Mr. Gray reports directly to the Project Manager on project matters, however, he maintains a direct reporting responsibility to Corporate QA Vice President, David Rohm.

ES&H Manager. Mr. Rick Greene is a Certified Health Physicist with 23 years of experience in health physics, safety, and compliance activities with emphasis on program management, operational health physics and training. Mr. Rick Greene (ES&H Manager, Bench Scale Treatability) is available to provide the necessary ES&H oversight during bench-scale treatability studies. He will ensure that the study is conducted safely and within the requirements of the laboratory's Chemical Hygiene Plan and Radioactive Materials License; evaluate sample and treatability study hazards and prescribe control measures such as specific engineering controls, PPE, and administrative procedures; conduct training and/or briefings for unique or special hazards; and monitor function and use of prescribed hazard control methods. Mr. Greene reports directly to the Project Manager and will also maintain reporting responsibility to Corporate ES&H Vice President, Warren Houseman.

Environmental Compliance Coordinator (ECC). The Environmental Compliance Coordinator for this project is Mrs. Linda Lawhorn. Mrs. Lawhorn maintains compliance with local, state, and federal regulations, including the U.S. Department of Transportation

(DOT), International Air Transportation Association (IATA), U.S. Environmental Protection Agency's (EPA) Resource Conservation and Recovery Act (RCRA) and Toxic Substances Control Act (TSCA) requirements, and a variety of local air and water ordinances for IT Corporation's (IT) Technology Applications Laboratories (TAL). Ms Lawhorn is responsible for all aspects of TAL waste management and transportation. The ECC will report to the Health and Safety/Health Physics Officer.

Figure 9-1. IT Project Team



9.3 *Data Quality Objectives*

Data quality objectives (DQOs) are qualitative and quantitative statements used to design a study that will limit uncertainty to an acceptable level. DQOs provide a systematic process for verifying that analytical data are adequate for the intended use.

Five levels of analytical support (analytical levels) have been defined in "Data Quality Objectives For Remedial Response Activities," U.S. Environmental Protection Agency (USEPA), March, 1987. (Reference 13)

9.3.1 Analytical Levels.

- Level I - Analysis is performed on site. This level provides most rapid results, but data are of limited use. Quality control (QC) procedures are few to none. Level I is usually used to determine analyte presence, but may provide gross quantification.
- Level II - Analysis is performed on site. This level provides quick results, but data are of limited use. QC procedures are minimal, and data are qualitative, semi-quantitative, or quantitative. Data may be used to make decisions of limited scope. Analytical methodology is based on standard industry methods, but may be modified to provide quick results.
- Level III - Analyses are performed at an established laboratory in accordance with accepted methodology and internal laboratory QA program procedures. Analyses are designed to provide results within the accuracy of routine laboratory procedures. This level provides a good degree of confidence in the data, and may be used to make engineering design decisions. Reporting is done on a certificate of analysis and does not include QC sample data or analytical raw data. Data cannot be validated.
- Level IV - This level provides the highest level of confidence in analytical data. Analyses are performed at an established laboratory in accordance with EPA Contract Laboratory Program (CLP) requirements and industry accepted methods. For analytes not covered by

the CLP program, the laboratory internal QC program is used. CLP parameter reports are in the format specified by the (EPA) CLP program and include all raw data. Non-CLP parameters are reported on a certificate of analysis include all QC samples and raw data, and analyses are performed according to the laboratory QA program procedures. Data may be validated according to EPA requirements.

- Level V - Analyses are performed according to nonstandard methods. QC procedures are specific to the procedure used. Level V may also include physical property analyses.

At the present time, Analytical Levels IV and V are not anticipated for use with this project. However, due to the nature of process development work, situations may occur where these analytical levels may apply.

9.3.2 Data Precision, Accuracy, and Completeness

Precision, accuracy, representativeness, completeness, and comparability (PARCC) requirements are the standards against which chemical analysis data are judged. These parameters are discussed in more detail in a later section discussing calculation procedures. Section 14.0. PARCC requirements, except comparability, have been set for Level III analyses. There are no requirements for data comparability.

- **Precision.** The agreement among a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by means of duplicate/replicate analyses.
- **Accuracy.** The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and a common systematic error component.
- **Completeness.** The ratio of the number of measurements taken that meet QA objectives for precision and accuracy to the total number of measurements.

- **Representativeness.** The degree to which a sample or group of samples is indicative of the population being studied.

To assess the adequacy of these specifications, it is important to document their rationale. Specifications may be based upon regulatory requirements, experience, and project-specific technical requirements.

Precision and accuracy requirements do not apply to Analytical Level I, II or physical property analyses. Analytical Level III chemical analysis data precision, accuracy, and completeness requirements are presented in Table 9-1.

In addition to sampling, operational data will also be acquired during the pilot-scale verification testing. Accuracy of these measurements will be within the tolerances of the measurement equipment used. There are no requirements for measurement precision.

Table 9-1
DQO Requirements for Analytical Level III
Chemical Analysis

Analysis	Accuracy	Precision	Completeness
Metals (except mercury) Totals and TCLP	$\pm 25\%$	$\pm 30\%$	90%
Mercury Totals and TCLP	$\pm 25\%$	$\pm 30\%$	90%

9.3.3 Detection Limits

Detection limits for Analytical Levels II and III chemical analyses will be the best possible considering the methodology, instrumentation, and sample matrix.

9.4 Calibration Procedures and Frequency

Periodic equipment calibration records are maintained in the Quality and Operations files by the QAO.

Laboratory balance calibration procedures are described in Standard Operating Procedure (SOP) PDGADM0002, Appendix A. The user will perform a check using a known weight each day the balance is in active use. Because this calibration is performed only when the balance is used, calibration records will not necessarily exist for each workday. The calibration records will be kept in a balance logbook, one for each balance.

Calibration of bench-scale process monitoring equipment is not planned.

Thermometers are calibrated annually. The calibration is performed by comparing the readings of the thermometer with the readings of a certified thermometer at two temperatures. The difference between the two readings for each temperature should not exceed 2EC. This calibration should be performed before the first use and then annually thereafter. (Certified thermometers are excluded.)

Thermocouples may be used in place of thermometers. Thermocouple calibration is verified by the manufacturer prior to purchase. Operation of thermocouples used to measure the temperature of the waste during processing will be verified prior to pilot-scale operations. Thermocouple operation will be checked first by comparing room temperature readings to a calibrated thermometer. A range of plus or minus (\pm) 5.0°C is acceptable. The thermocouple will then be heated with a propane torch (or similar device) to check operation at an elevated temperature. The intent is to verify that the thermocouple will operate at elevated temperatures, not to verify calibration.

The pH meter will be calibrated at least once a day while it is actively in use. The pH meter will be calibrated using two pH standards in the range of the expected measures. The calibration will be verified using a third pH standard. These pH standard solutions are readily available commercially.

Analytical instrument calibration will be performed as described in the analytical methods or applicable laboratory SOPs as specified. Instrument background and efficiency will be determined daily on radiological screening and gross alpha/gross beta measurement instrumentation.

9.5 Treatability Testing and Analytical Procedures

General treatability testing procedures and sampling procedures are described in the work plan procedures. Specific analytical methods are also identified in the testing Work Plan.

9.5.1 Design

Design control, as it relates to this project, pertains to those processes intended to define, control, and verify technical investigations performed within this project (i.e., treatability studies tests).

9.5.2 Experimental Design

The experimental design governing the data to be generated during the treatability studies and demonstration tests will be generated by the principal investigators as part of the work plan procedures and will be reviewed and approved by senior technical staff on the project. The experimental design will investigate key variables, which affect the process performance and the ability to achieve the project objectives. The testing will be designed to obtain performance data so that the process can be optimized within the scope of the testing program and performance of a full-scale system can be predicted. The results of testing will be documented by the principal investigator in a research report. The resulting report will be reviewed and approved as described in this QAP.

9.6 Work Processes

The principal product of this project is laboratory-scale experimental data. The processes and equipment involved in the generation of these data must be managed and controlled in such a manner as to ensure the validity and quality of the product. Subcontractors working on this

project will be subject to control of processes and equipment as described in their respective Quality Assurance Manuals (QAMs).

9.6.1 Plans, Procedures, and Drawings

Plans, procedures, and drawings detailing the methodology for data acquisition will be developed, as appropriate, by the principal investigator or principal responsible person. Such documents will be approved by appropriate senior staff and will be incorporated into this document or considered an addendum to this document. These documents may include the Project Health and Safety Plan and the Project Waste Management Plan. These documents will be maintained in the project file.

9.6.2 Management of Technical Notebooks

Pertinent experimental data will be entered into technical notebooks in accordance with this QAPP. These notebooks will be identified by number and assigned to the principal investigator or project chemists under his supervision. The laboratory will maintain a master list of all notebook numbers and the individual responsible for maintaining the notebook.

9.6.3 Identification and Control of Laboratory Samples and Generated Data

Each individual assigned to this project is responsible for ensuring that all samples are identified properly and used as intended. Sample materials will be generated as part of this project for testing and evaluation. To ensure understanding of the obtained data, sample identification and distinguishing descriptions of samples will be recorded by personnel working on this project.

9.6.4 Identification and Control of Laboratory Equipment

Each individual assigned to this project is responsible for ensuring that all equipment is used as intended. Major equipment items used in sample preparation and testing will be identified in the procedures and approved upon review and approval of the final Work Plan.

9.6.5 Control of Waste Material

Quantities of “as-received” waste are not subject to the RCRA regulation requirements in 40

CFR 261-268. They are, however, subject to normal laboratory practices regarding segregation, spill protection, and other storage and handling issues as addressed in the Waste Management Plan. The plan also includes storage and handling requirements for the stabilized samples and residues subsequently generated from the received soil.

9.6.6 Handling, Storage, and Shipping

Procedures for the handling, storage and shipping of “as-received” waste, and samples generated from the waste as a result of treatability testing are specified in approved procedures, and the Waste Management Plan.

9.6.7 Control of Measuring and Test Equipment

Calibration procedures and frequency of calibrations for measuring and test equipment are specified in this QAPP, which will be reviewed and approved by the appropriate senior personnel.

9.7 DATA RECORDING, REDUCTION, REVIEW, AND REPORTING

9.7.1 Data Recording

Data collected during testing will be recorded in laboratory notebooks, data collection forms or in computer files. All laboratory notebooks are uniquely numbered and have sequentially numbered pages. The TDL SOP No. 1504, "Laboratory Notebook Recording Procedures," identifies the notebook criteria on data logging procedures (Appendix A).

Separate laboratory notebooks are designated for use to record the injection or introduction of samples into some analytical instrumentation as instrument run logs. These logbooks are also used to record maintenance or problems with the instruments.

9.7.2 Data Reduction

All data shall be recorded in a laboratory notebook or on a data collection form. If computer programs are used to perform calculations, the program shall be verified prior to use to assure all manipulations and calculations are performed as intended. Computer program verification

consists of comparing the results of a standard data set entered into the program with manually calculated results. Only standard formulas will be used to calculate data.

9.7.3 Data Review

Data review shall consist of checking transcriptions (100 percent) and manual calculations (100 percent) by a second party. All manual data entries into computer programs for data reduction shall be checked for data entry accuracy (100 percent). The principle investigator or his designee shall review all test results.

9.7.4 Data Reporting

A draft and final Toipical report shall be prepared. The report will contain results obtained from testing. All manual data entries into text, tables or figures for data presentation shall be checked for data entry accuracy (100 percent). The principle investigator or his designee shall review all reported results. The report shall include an analysis and interpretation of testing considering the effectiveness of the treatment process. Key parameters that may affect full-scale applications will be discussed. The draft report shall be reviewed by the project manager and senior technical or responsible staff that is knowledgeable of project requirements and appropriate areas of discipline. The report reviews will be documented.

A final report will be prepared after the draft report review by the client is completed. Comments received from the draft report will be reviewed and incorporated as appropriate. The project manager shall review the final report prior to submittal.

9.8 *Preventive Maintenance*

Equipment preventive maintenance procedures specified by the manufacturer are followed. There are no specific preventive maintenance requirements for the bench equipment other than those specified by manufacturers of subsystems and components. Calibration of equipment and instruments is discussed in other sections of this QAP.

9.9 *Procedures for Assessing Data Precision, Accuracy, Completeness, and Comparability*

9.9.1 Data Precision

Precision is an indicator of the repeatability of a measurement, and is expressed as the relative percent difference (RPD) of two analytical measurements. The formula below is used for calculating RPD, and is from "USEPA Contract Laboratory Program Statement of Work for Inorganics Analysis, SOW No. 788, Exhibit E, Quality Assurance/Quality Control Requirements. (Reference 14)

$$RPD = \frac{S - D}{\left(\frac{S + D}{2}\right)} * 100$$

where:

S = The larger of two measurements

D = The smaller of two measurements

9.9.2 Data Accuracy

Data accuracy is an indication of how close a measurement is to the actual value. Data accuracy is determined by spiking a sample with a known value and measuring the percent recovery (%R) of the spike. The formula listed below for calculating %R is also from the USEPA SOW No. 788.

$$\% R = \frac{SSR - SR}{SPIKE ADDED} * 100$$

where:

SSR = Spike sample result

SR = Original sample result

9.9.3 Data Completeness

Data completeness is defined as the percentage of useable data points from the set of total data points that are required to obtain a specified confidence level. Percent completeness (%C) is calculated according to the following formula:

$$\% C = \frac{V}{N} * 100$$

where:

V = Number of valid measurements

N = Number of measurements required to obtain a specified confidence level.

9.9.4 Data Comparability

Analytical data acquired by using different analytical methodology may not be directly comparable. Data obtained using the same procedures under different DQO Levels may not be directly comparable. There is no project-specific requirement for data comparability.

In order to be most comparable to IT analytical results, split samples given to other testing laboratories should be analyzed using the same or comparable methods specified in the Work Plan.

9.9.5 Percent Relative Standard Deviation

The %RSD will be used as a measure of homogeneity of a sample matrix and is calculated as follows:

$$\% RSD = \frac{O_{n-1}}{C_{ave}}$$

where:

O_{n-1} = Standard deviation of analytical results

C_{ave} = Average of analytical results.

9.10 Corrective Actions

Laboratory nonconformance/variance report forms are used to document problems,

deviations, or actions that are not performed in accordance with standard procedures, as well as the corrective actions taken. The laboratory nonconformance system is described in SOP TDL1705, Appendix A. An example of a nonconformance/variance form is included as Figure 9-2. The IT project manager will notify the NETL project manager or point-of-contact of any significant nonconformance issue that is expected to impact project schedule, cost, or the meeting of technical objectives. Copies of significant nonconformance reports will be transmitted to NETL.

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Figure 9-3
Nonconformance/Variance Report Form
NONCONFORMANCE/DEFICIENCY REPORT

NONCONFORMANCE Or DEFICIENCY (circle one)	Project Name/#:	Page 1 of
	NCR Number:	Date:
Nonconformance/Deficiency Description (include requirement violated):		
Identified by: _____ Date: _____		
Root Cause:		
Corrective action (include expected completion date): _____ Use as is _____ Rework _____ Reject _____ Repair		
To be performed by: _____ Expected Completion Date: _____		
Action taken to preclude recurrence: <input type="checkbox"/> Not Applicable		
To be performed by: _____ Expected Completion Date: _____		
Client notified (include client name, how notified, and response):		
Notified by: _____ Date: _____		
Corrective action completed by: _____ Date: _____		
Corrective action approved by:		
Project Manager: _____ Date: _____		
QA Comments:		
QAO Approval: _____ Date: _____		

9.11 Subcontracting and Control

Procurement of services and goods must be controlled to assure that the service or item purchased will fulfill its intended purpose. IT has established procedures for procurement of services and materials. The Defense Contract Management Agency (DCMA) has approved the IT Group corporate purchasing system effective until December 31, 2002. Procurement procedures are available online from the procurement subweb.

All purchases of services or materials must be reviewed and approved by the proper level of authority as specified in IT Project Guidance Manual and available online. The proper level of authority for this work will be the Project Manager.

Whether purchasing services or materials, enough information must be presented in the purchase order to sufficiently specify what is required. Quality assurance personnel review requirements for all special and non standard materials.

9.11.1 Receiving Inspection of Purchased Items

Items received from a vendor will be inspected to verify that the right item was received, any required calibrations were performed, and that the item functions as intended. This inspection will be documented by the receiver signing and dating the vendor's packing slip. Signed packing slips will be sent to accounts payable for processing.

9.11.2 Documentation of Purchasing Activities

Documentation of procurement activities is the responsibility of the procurement department.

9.11.3 Subcontracting

All IT subcontractors performing work on IT's client site must be prequalified according to procurement procedures. The procurement department will maintain a listing of prequalified subcontractors. Prequalification of subcontractors is the responsibility of the procurement department, and is initiated by either a specific request to procurement, or by procurement's receipt of a purchase requisition for services to be performed by a firm or individual that is not prequalified.

Prequalification of subcontractors may require several months, depending on the type of work to be performed. When time constraints make full prequalification impossible, the project manager can request partial prequalification to the extent necessary to reasonably assure that the subcontractor meets IT's requirements including proper insurance.

9.11.4 Contract Flow-Down Requirements

IT's contractual requirements will be passed along to subcontractors where required. IT Contracts Department identifies required flow-downs and it is the responsibility of procurement to verify that applicable contract requirements are passed along to subcontractors.

9.12 Document and Record Control

TDL maintains project-specific records and non project-specific, quality, health and safety, regulatory compliance, and radiological compliance records. These records must be controlled and retrievable. Procedures will be implemented for controlling these records and assuring retrievability. Records which are not project related, not required by regulation, not required to be maintained by other IT groups, or are not quality related are not required to be maintained.

All project records and documents, except laboratory notebooks, logbooks, proposal, and contract documents, shall be maintained in the project file. Laboratory notebooks and logbooks are controlled by the laboratory, and when completed are filed in the laboratory quality and operations files. Proposal and contract documents are maintained in the contract files.

Procedures for recording data in a laboratory logbook are in SOP TDL1503, Analytical Logbook Recording Procedures, Appendix A. Procedures for recording data in a laboratory notebook are in SOP TDL1504, Laboratory Notebook Recording Procedures, Appendix A.

This QAPP discusses data recording procedures using data collection forms. Data collection forms that become contaminated during pilot verification testing will be copied. The copies will be maintained in the project file, and original forms will be disposed of with other project waste.

The final project report may include copies of laboratory notebook pages, data collection forms, video/photographs and analytical data reports (not including raw data such as chromatograms, etc.). Submittal of the final report constitutes turnover of the project records to the client.

All project records will be categorized, filed, and retained for a minimum of one year from the date of final invoicing.

9.13 Laboratory Personnel Training and Qualification

All laboratory personnel receive continuous training on laboratory procedures, QA/QC, and laboratory safety. Training and qualification of laboratory personnel is documented in resumes and training files. Resumes include academic credentials, employment history, experience, and professional registrations. Individual training records are maintained that document on-the-job training in specific procedures or when formal training courses are taken. The laboratory maintains training records in the quality and ES&H operations files.

Personnel performing work or managing activities within the scope of this project shall be trained and qualified as appropriate to ensure job proficiency. The bench-scale task associated with this project is performing a treatability study in a chemical laboratory that contains radioactive materials. The training for such work is considered “routine,” not project specific.

Each project team member will have the appropriate required training (i.e., Chemical Hygiene Plan and Radioactive Materials Handling) for this type of work. Such training will be identified and recorded by, or be accessible through, the ES&H manager. Additional

training related to this specific project includes this document, all project related organization, procedural and safety meetings, as well as all standard operating procedures and reference methods for laboratory work, which are considered an addendum to this document.

9.14 *Continuous Improvement*

IT personnel will strive for continuous improvement of the quality and safety of work practices. A part of the continuous improvement program will be investigating nonconformances for the root cause, determining if action(s) can be taken to prevent recurrence, and implementing the action(s). Peer reviews, health and safety reviews, and project reviews are also a means for identifying and discussing methods for improving operations.

TDL Associates are encouraged to provide suggestions for improving operations. Suggestions should be made to the appropriate supervisor or responsible individual.

In addition to these activities, results of any systems audits of the TDL's laboratories are reported to the TDL Laboratory Director for follow-up response and corrective action.

9.15 *Facilities and Equipment*

IT operates two facilities that may be used on this project. TDL and ETDC.

The TDL, at 304 Directors Drive, Knoxville, TN 37923, is the main facility for performing bench-scale treatability studies and sample chemical analyses. This facility maintains a Radioactive Materials License from the State of Tennessee and operates under the treatability exemption in 40 CFR 261.4.

The ETDC at 1570 Bear Creek Road, Oak Ridge, TN 37831, houses the Geotechnical Laboratory. Personnel from the TDL may also use this facility. ETDC also maintains a Radioactive Materials License, and operates under the treatability exemption.

Both facilities have a U.S. Dept. of Agriculture Animal and Plant Health Inspection Services (APHIS) agreement for the importation of foreign and domestic soils.

Both TAG facilities are monitored by a security company. Access to each facility is strictly controlled.

Treatability studies and other activities are performed under the relevant permits, licenses, or exemptions. The TAL Director is responsible for verifying that TAL facilities maintain the required permits and licenses to meet specific requirements.

9.15.1 Technology Development Laboratory

The TDL was constructed in 1975 to evaluate waste treatment processes and develop new treatment technologies. The TDL was designed to allow bench-and pilot-scale testing of treatment processes with on-site analytical chemistry support. This facility is currently IT's primary laboratory for bench-scale testing of physical and chemical treatment processes on radioactive, hazardous and mixed waste samples. The TDL is capable of managing many treatability projects simultaneously with typically from 10 to 30 treatability projects active during any given period.

Significant features of this 16,000-square foot laboratory include the following:

- A 2,500-square-foot-limited access laboratory designed for the safe handling and analysis of chemicals and samples having high hazard or unknown properties. This laboratory has four isolated negative-pressure cubicles that contain fume hoods and laboratory benches and are constructed of materials selected for easy decontamination. Each cubicle's ventilation system is equipped with both high efficiency particulate air (HEPA) and activated carbon bed housings to allow proper

selection of effluent controls. The drains from the sinks in this laboratory are connected to a collection tank.

- Two general chemical engineering laboratories that are used primarily for feasibility testing of chemical recovery and treatment systems, and for bench-scale chemical and physical process development. Each laboratory has sufficient bench and hood space including walk-in, distillation, and bench-top hoods to allow a variety of operations to be set up and operated safely. Each laboratory has approximately 48 linear feet of bench space and 48 linear feet of fume hood space.
- A mini-plant area, mini-plant control room, and a chemical feed and storage area are connected to the limited-access laboratory. The mini-plant is used to perform larger-scale, controlled-condition research as well as to apply technology on chemical recovery and treatment systems. The mini-plant area contains the Rotary Thermal Apparatus (RTA). The RTA is a bench-scale rotating thermal test apparatus that allows the use of 1- to 2-kilogram (kg) samples, which are subjected to conditions that simulate processing in a full-scale rotary thermal treatment device. Treated residues from the tests provide adequate sample for thorough analytical characterization.
- There are also sample preparation and analysis areas at the TDL that are used for solvent extraction cleanup procedures, column chromatography and
- Instrumental analysis of samples of various matrices using a wide variety of methodologies.

9.15.2 Equipment

TAL maintains a large inventory of equipment for chemical analysis of liquids, solids, and gasses, geotechnical testing of soils, radiological analyses, and testing of chemical and biological treatment processes. TAL has a large variety of analytical instruments, including gas chromatographs (GC) with most detector types, GC mass spectrometer (GC/MS), flame

atomic absorption spectrometer (FAA), AA with graphite furnace (GFAA) attachment, inductively coupled argon plasma spectrometer (ICP, sequential), liquid chromatograph (LC) and ion chromatograph (IC) with various detectors, total organic carbon analyzer (TOC), ultraviolet/visible spectrophotometer (UV/VIS), fourier transform infrared spectrophotometer (FTIR), respirometer, microscopes, ion specific electrodes, gross alpha/beta counter, liquid scintillation counter, and gamma spectrometer. In addition to analytical equipment, TAL maintains a large supply of equipment to construct a wide variety of bench- and pilot-scale systems for testing waste treatment technologies.

10.0. QUALITY ASSURANCE QUALITY PLAN - NFS

10.1 Overview

The NFS Erwin facility has historically produced high-enriched uranium fuel for the Department of the Navy. To meet, in part, contractual requirements required by the Department of Navy a plant-wide Quality Assurance Program was implemented. The current program is based on the requirements and guidance of the American Society of Mechanical Engineers Nuclear Quality Assurance-1-1989 which is functionally equivalent in its intent to DOE Order 5700.6C and the requirements in the draft *Test Plan Guidance*.

The NFS QA Program is based on the requirements and guidance of the American Society of Mechanical Engineers Nuclear Quality Assurance-1-1989, which is functionally equivalent in its intent to DOE Order 5700.6C and the requirements in the draft *Test Plan Guidance*. The NFS QA Program is described in more detail in their Demonstration Test Plan.

The NFS Quality Assurance Program is described in *Quality Assurance Program, Procedure No: NFS-M-48, Revision 1*.

10.2 Organization

Figure 10-1 shows the organizational structure for the demonstration project. The Quality Assurance and Health and Safety report independently to the Vice President, Safety and Regulatory. Functional areas for the demonstration project and responsible individuals are shown in Figure 10-1. These are described as follows:

N. P. Jacob - Manager, ATL

Dr. Jacob acts as the NFS technical oversight and primary contact for performing the demonstration work. He oversees laboratory and analytical operations.

J. D. Davis - Project Chemist/Lead Mr. Davis directs the chemists performing the actual demonstration work. He also bears responsibility for QC of sample results, as discussed in Section 4.

Melinda M. Keller, Rachel Glass - Chemist(s)

Ms. Keller and Glass are the chemists responsible for performing all bench- and pilot-scale laboratory work to demonstrate the proposed mercury treatment process. Ms. Keller has been the laboratory lead in the successful demonstrations for MER03 (BNL) and MER04 (EPA).

J. W. Pugh - Transportation and Waste Management

Mr. Pugh is responsible for demonstration sample receipt at NFS, tracking of the material while it is at NFS, and final waste material shipment off site.

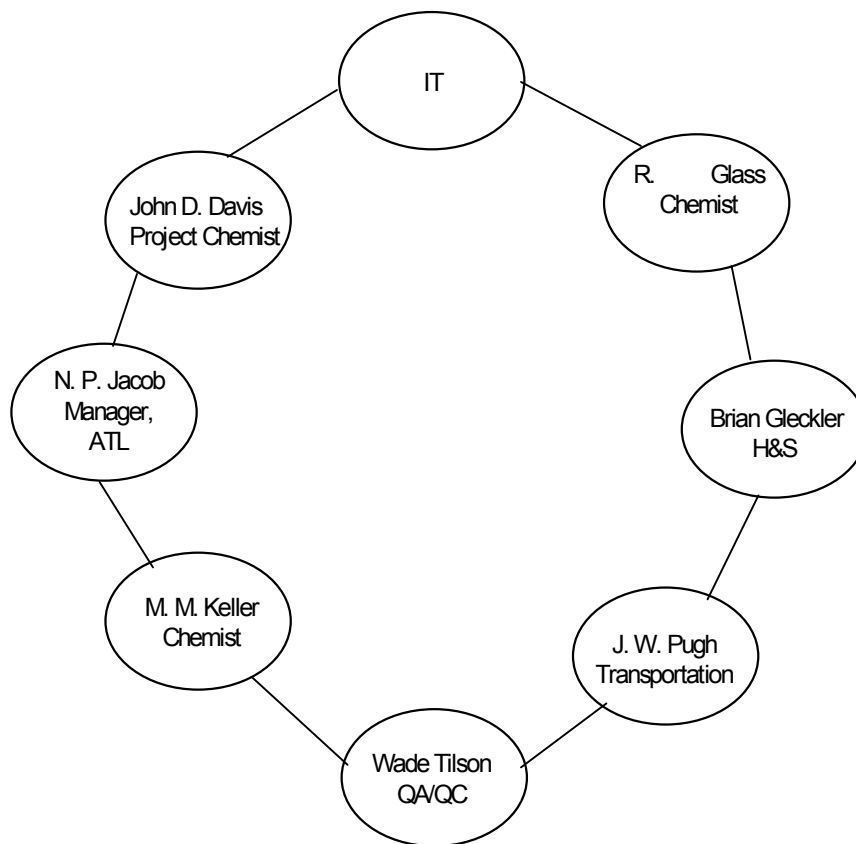
Wade Tilson - QA/QC

Mr. Pearson provides quality control oversight of the demonstration program, as appropriate.

Brian Gleckler - Health and Safety

Mr. Gleckler provides coordination with the Health and Safety function at NFS. He makes sure the necessary Health and Safety resources are available for the demonstration, and that the work is done in compliance with the applicable regulations.

FIGURE 8-1. DEMONSTRATION TEAM



10.3 Final Product or Waste Form Verification/Analysis

An off-site, Utah-approved laboratory (Enviro-Test, LLC, formerly Core Labs) performs final product analysis for final verification and regulatory compliance. In addition, as part of the NFS Quality Assurance Program, an informal independent assessment of this laboratory has been made. NFS has evaluated the following areas:

- Laboratory organization and personnel,
- Personnel training,
- Sample practices,
- Material procurement and control,
- Facilities and equipment,
- Analytical procedures,
- Instrument calibrations,
- Limits of detection,
- Analysis of samples and documentation,
- Corrective actions,
- Documents and quality assurance records control,
- Data evaluation,
- Holding times and preservatives for samples, and
- Internal audits.

Off-site analytical data packages will include an EPA Quality Assurance Level 3 package.

Material received for treatability studies must conform to the requirements of NFS procedure *Receipt/Shipment of Sample Materials for Development/Treatability Studies, NFS-ACC-87, Rev. 1*. This procedure describes, in part, the requirements for material receipt, maintenance of material tracking and custody. The “how to” part is documented in the *Hazardous Waste Treatability Sample Documentation, Standard Operating Procedure 387*. Two key documents are used to track and maintain custody of the treatability material: (1) the Daily Hazardous/Mixed Waste Treatability Study Facility Logsheet, and (2) the Hazardous/Mixed Waste Treatability Study Log.

10.4 NFS Facilities and Equipment

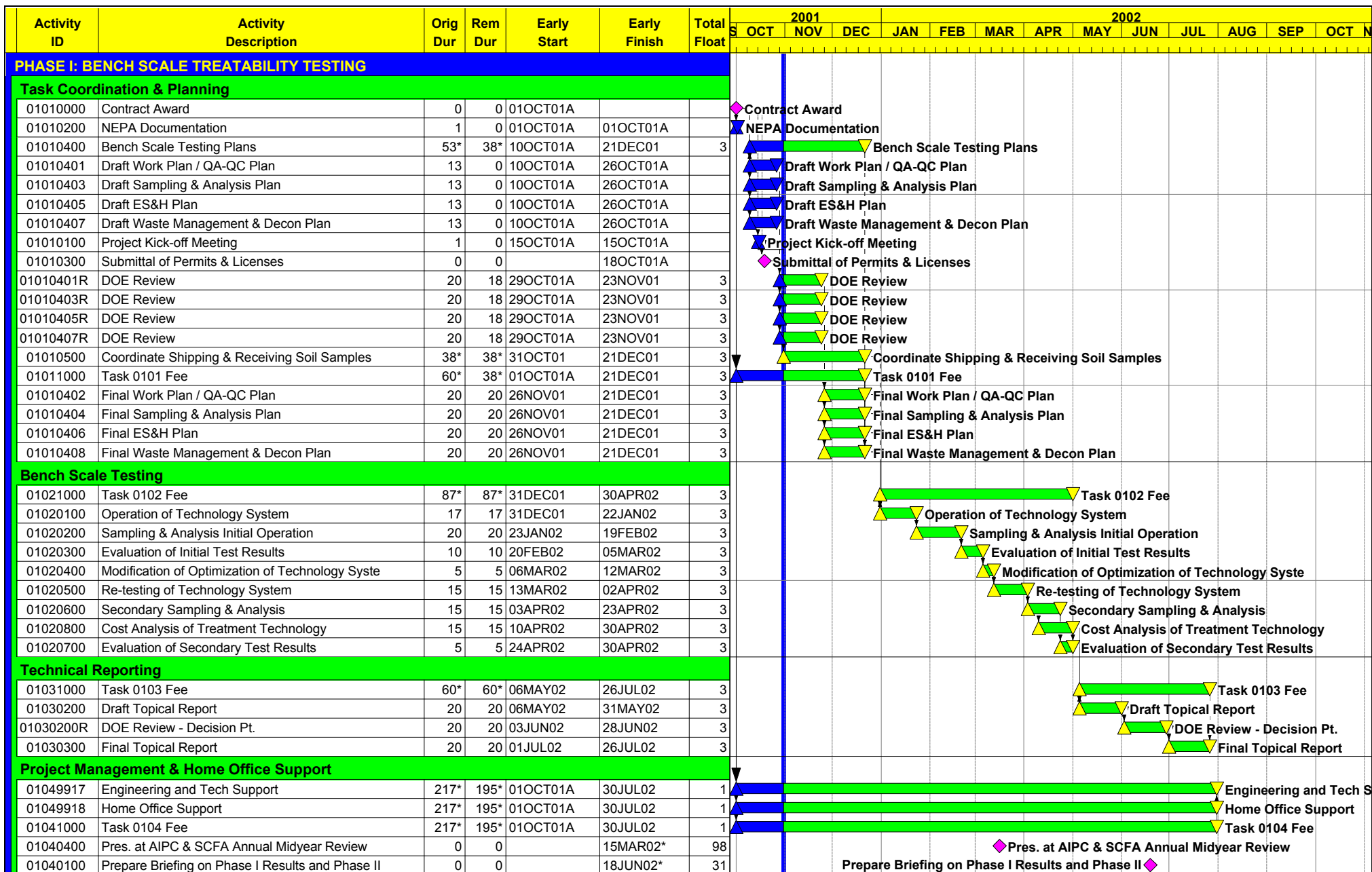
The development facilities described in this section are high quality test arenas for radioactive and hazardous materials. As such, it is appropriate to apply these facilities for the subject work. These facilities have been in operation over almost ten years, and have been applied to numerous DOE and commercial projects to demonstrate mercury and other metal stabilization technologies. The following facilities and equipment described in this section are “Contractor Owned”.

The NFS Applied Technology Laboratory (ATL) will be used to perform the scale-up treatability work for this project under the Task “Bench Scale Testing”. NFS handles research and development activities in ATL that involve radioactive materials under a State of Tennessee Radioactive Materials License # R-86008-J99. Treatability work is performed under the 40 CFR exemption provided for research and development activities. As such, the NFS facility has capability and experience in performing test work on mixed waste.




The treatability scale-up work will be performed in the high bay laboratory. This laboratory is comprised of approximately 3000 ft² of area for small scale and pilot testing and incorporates ventilated hoods and associated bench space. This lab also contains a separate, ventilated enclosure of approximately 300 ft² that was specifically developed for performing mercury scale-up testing. Potential mercury vapors generated within the enclosure during testing are conveyed from the enclosure area through ventilation ducting and trapped prior to environmental release. Within the enclosure is a pilot scale pug mill, capable of containing up to 45 kg of soil plus reagents, and a jaw crusher for reducing particle size of incoming soils, if necessary, down to 3 mm. NFS has proven this system successfully on the BNL soils offered for the MER03 Demonstration. During the course of scale-up testing, NFS also will deploy an existing Jerome Gold Vapor Mercury Analyzer to monitor mercury airborne activities during the course of the scale-up testing. NFS will accomplish total mercury and TCLP mercury analyses on test samples by a LECO AMA254 Advanced Mercury Analyzer with liquid sample auto-loader. This instrument uses a mercury absorption cell, but also uses a collection and purification technique that isolates the mercury in the sample from any organic compounds that could interfere with the analysis. This instrument is housed in the high bay facility.

11.0. SCHEDULE

The enclosed schedule identifies milestones and the interrelationships of the project tasks. Our schedule is included in the Gantt chart (Figure 11-1). IT anticipates completing performance within the requested eight-month period. However, for the Phase I overall performance period, IT is estimating and requesting a ten-month performance period to allow two months for NETL and DOE review and comment response on the work plans and final report.



Start Date 01OCT01
Finish Date 30JUL02
Data Date 31OCT01
Run Date 05DEC01 14:47

 Early Bar
 Progress Bar
 Critical Activity

NETL

Sheet 1 of 1

**DOE/NETL - Mercury
Phase I Schedule
Status Report 10/31/01
Contract# DE-AC26-01NT41345**

Date	Revision	Checked	Approved

12.0. ENVIRONMENTAL HEALTH AND SAFETY PLAN - IT

12.1 Introduction

Operations at the IT Technology Development Laboratory (TDL) are conducted under comprehensive environmental health and safety programs designed to ensure employee and environmental protection and conformance with state and federal regulations. The facility has the appropriate licenses and permits to perform the scope of work as detailed in the workplan. A staff specializing in health physics, laboratory safety, and environmental compliance administers these programs.

12.2 Licensing And Permitting

The facilities are licensed by the Tennessee Division of Radiological Health (TDRH) to perform analytical procedures and treatability testing on radioactively contaminated materials. These activities are performed in accordance with the state-approved Radiation Protection Program (RPP). The elements of the RPP include training, exposure limits, monitoring and survey requirements, exposure control, and radioactive material control.

The TDL (EPA ID No. TND 000770479) is authorized to perform treatability studies on RCRA hazardous wastes under the treatability exemptions of the TDEC, Division of Solid Waste Management (TN Rule Chapter 1200-1-11-.02[1][d]6). These regulations, equivalent to the exemptions in Title 40 Code of Federal Regulations (CFR) Part 261.4, grant exemptions for treatability studies provided that no more than 10,000 kg of "as received" hazardous waste are stored at the facility and that no more than 2500 kg of "as received" hazardous wastes are treated in all treatability studies per day. These regulations also require that the treatability samples be removed no more than 90 days after the completion of the study or no more than 1 year after the original sample shipment, whichever date occurs first. These exemptions apply to wastes that are classified as hazardous by RCRA or equivalent authorized state regulations. The contaminated soil to be used in this project is expected to meet the requirements of the treatability exemption regulations. A copy of the appropriate

section of the Tennessee regulations is available upon request.

The TDL has written operating procedures and tracking systems to document the receipt, storage, and return of treatability samples.

12.3 Health And Safety

Laboratory operations at the TDL are performed under the requirements of the facility CHP. Laboratory safety procedures, engineering controls, and personal protective clothing requirements for normal laboratory operations are found in the CHP.

This Environmental, Health, and Safety Plan is prepared to supplement the RPP and CHP for the NETL Mercury treatability project. It is intended to identify the hazards and prescribe hazard control methods for the bench scale treatability testing of stabilization processes for mercury-contaminated soils. This treatability study is designed to test the effectiveness of various formulations for stabilization of mercury-contaminated soils.

The processes used in this study will consist of amalgamation/stabilization formulations and oxidation/stabilization formulations. One 5-gallon samples of mercury contaminated soil will be transported to TDL. The concentrations of contaminants in the sample are not known at this time. Sample characterization data will be evaluated for health hazards when it becomes available.

The bench scale effectiveness testing consists of the following general tasks:

- Collect samples for in-house characterization
- Prepare samples (mixing, sieving, aliquoting)
- Add reagents/mix
- Collect samples for analysis
- Clean-up/Waste management

12.4 Hazard Analysis

12.4.1 Prepare samples

Soil samples from a DOE site will be received at the TDL in one 5-gallon bucket. Aliquots of the soil will be sieved and homogenized. Preparing the samples will require repetitive manual lifting of the buckets and mixing (stirring) in order to homogenize the aliquots. The samples will not be spiked with metallic mercury. The potential also exists for exposure to contaminants in the soil. This task involves the small potential for spills of the soil during transfer and mixing of the samples.

12.4.2 Collect samples for in-house characterization

Aliquots of the samples will be collected for in-house characterization using standard TDL analytical methodologies. In addition, quantitative mercury vapor measurements will be made in the headspace of some containers. The potential exists for exposure to contaminants in the soil.

12.4.3 Add reagents/mix

After the samples have been prepared a variety of amalgamation/stabilization and oxidation/stabilization reagents will be added to the samples and thoroughly mixed. These reagents may include sulfuric or other acids for pH adjustment, calcium polysulfide, Degusa TMT, Nalmet 8154, various other proprietary fixation reagents and potentially Portland Cement. The selected set of reagents may be oxidizers, reducers, acids, or bases. Each container will be capped and shaken. Mercury vapor concentrations will be measured in the headspace of the containers. Hazards associated with this task include potential exposures to reagents used in the process, contaminants in the samples, and mercury vapor.

12.4.4 Collect samples for analysis

Aliquots of the samples will be collected for in-house characterization using standard TDL analytical methodologies. In addition mercury vapor measurements will be made in the headspace of some containers. The potential exists for exposure to contaminants in the soil.

12.4.5 Clean-up/Residue and Waste Management

All materials from this project are to be returned to DOE and as such the treated materials, residues, etc., are not wastes. Materials from treatability testing include residual untreated soil sample, residual treated soil sample, and contact material. Clean-up and sample/residual management activities will include the handling of buckets and treated sample containers, compositing compatible streams, and preparing materials to return to DOE for disposal.

Hazards identified by task are presented in Table 12-1.

Table 12-1.
Hazards Assessment by Task

Hazard	Task				
	Prepare Samples	Collect samples	Add reagents/mix	Collect samples	Clean-up/Waste Management
General Laboratory Operations	X	X	X	X	X
Repetitive motion	X	X	X	X	
Manual lifting	X				X
Slips/trips/falls	X		X	X	X
Chemical exposure	X	X	X	X	X
Radiation exposure ¹	N/A	N/A	N/A	N/A	N/A

¹If the soil is radioactive

12.5 Hazard Control

The hazards associated with the bench scale treatability testing of the mercury stabilization processes are addressed through the use of normal laboratory safety rules as described in the TAG CHP.

Laboratory safety rules are reproduced below:

- The associates should understand, and will implement the requirements of the CHP.
- All accidents, incidents, injuries, work-related illnesses and chemical releases will be

reported to the supervisor and to HS immediately following the event, or as soon as it is discovered.

- Management will be notified immediately of an unsafe condition, or safety equipment that is broken, damaged, or missing.
- Good housekeeping is of paramount importance. Associates will keep floors, working surfaces clean, dry and free from clutter. Spills will be cleaned up immediately. Glassware and other equipment will be stored in designated areas when not in use.
- Eating, drinking, chewing gum or tobacco products, or applying cosmetics or lip balm in the laboratory is not permitted.
- Hands, arms and other areas of the body that may have been in contact with chemicals will be washed before eating, drinking, or smoking.
- All facility personnel and visitors will wear ANSI-approved eye protection with side shields while in laboratory areas.
- Avoid use of contact lenses in the laboratory unless necessary. If they are used, inform the supervisor and HS so precautions, if necessary, can be taken.
- Chemical goggles or face shields over safety glasses will be worn while large quantities of hazardous liquids are being handled, concentrated acids or bases are used, or where there is a potential for chemicals to be splashed during transfer.
- Laboratory coats will be worn by all associates and visitors when working directly with, or in close proximity to, significant quantities of chemicals and samples, or contaminated equipment. Lab coats may not be taken home for laundering. Lab coats must be removed before entering areas where food consumption is permitted.
- Gloves must be worn when chemicals and samples are handled. The gloves will be selected on the basis of the materials handled, procedure, the temperature conditions, and the dexterity required.
- Associates will inspect gloves before use and make sure that the gloves provide adequate protection from the hazardous chemicals being used.
- PPE listed in the standard operating procedure for the task or project Health and Safety Plan will be used.

- Enclosed shoes will be worn in a laboratory.
- Any safety equipment, such as a laboratory coat, that is suspected of being contaminated will be removed immediately. A new laboratory coat or replacement safety equipment will be obtained.
- Clear access will be maintained to all emergency equipment, such as fire extinguishers, eye washes, safety showers, etc., and to electrical panels and other control equipment.
- Associates will know what emergency equipment is necessary in the event of an accident or spill and will be familiar with its location and operation.
- The use of respiratory equipment is restricted to associates trained in its use.
- A neoprene rubber or polyethylene carrier will be used to transport single bottles of chemicals from stockroom/storeroom and within the laboratory. Bottles shall not be carried unprotected.
- A cart with a leakproof top to provide secondary containment of chemicals will be used to transport multiple containers of chemicals from stock/storeroom and within the laboratory.
- Mouth pipetting is prohibited. Mechanical pipettes will be used.
- Chemicals or samples will not be smelled or tasted.
- Chemicals must be stored in safe locations according to compatibility.

Hazard control methods for the specific hazards identified are presented in Table 12-2.

Table 12-2
Hazard Control

Potential Hazards	Control Measures
General laboratory operations	<ul style="list-style-type: none"> Follow safe lab rules as detailed in facility CHP
Pressurized containers	<ul style="list-style-type: none"> Relieve pressure prior to and after mixing by venting reactors (loosen lid) Vent reactors into an operating hood Wear safety glasses with side shields
Repetitive motion	<ul style="list-style-type: none"> Take frequent breaks when performing repetitive tasks Position equipment to avoid awkward or strenuous positions
Manual lifting	<ul style="list-style-type: none"> Size up the job. Think it through. Lift with your legs, not your back. Use mechanical equipment whenever possible. Get assistance when manually lifting awkwardly sized items or those items over 60 pounds.
Slips/trips/falls	<ul style="list-style-type: none"> Clean up spills promptly Avoid storing materials in aisles Keep work areas clean and orderly
Chemical exposure	<ul style="list-style-type: none"> Wear nitrile gloves and safety glasses when measuring and mixing reagents Follow general laboratory rules for handling chemicals Follow MSDS Handle mercury and mercury contaminated samples in a properly operating hood
	<ul style="list-style-type: none">

13.0. ENVIRONMENTAL HEALTH AND SAFETY PLAN – NFS_____

13.1 Corporate Program

NFS has a corporate safety program that requires each operating group to document and implement procedures geared to the special requirements of its fixed facilities and field operations. Each facility/field operation has a safety and industrial hygiene program with written procedures for the management of safety in day-to-day operations. The standard safety program is comprised of specific procedures and general practices, which detail exactly how safety related tasks, are to be performed.

The health and safety aspects of each treatability study are evaluated separately in the procedure *Receipt/Shipment of Sample Materials for Development/Treatability Studies, NFS-ACC-87, Rev. 1*. This procedure documents, among other things, the health and safety requirements/concerns for each treatability project. Environmental evaluations for each project include a determination as to whether special controls are required for: (1) labeling the material, (2) receiving the material, (3) environmental discharges (air, liquid, solid waste) and (4) packaging/shipping. Similarly, for industrial safety each study is evaluated to determine special requirements for: (1) personnel monitoring, (2) protective clothing, (3) protective equipment, (4) material processing, and (5) material packaging, storage and shipping. A nuclear safety evaluation is performed to determine: (1) license amendment requirements, (2) special controls for criticality safety requirements and (3) special packaging and/or shipping requirements. The above programs also ensure that applicable material, control and accountability records are maintained. The procedure also provides for compiling the information and designates the responsible group for the annual report required to meet the regulatory requirement for the RCRA treatability study exemption. Management of spills and emergency response is included in the NFS *Spill Prevention Control and Countermeasure (SPCC) Plan, Rev. 2*, and the *Emergency Plan, Rev. 2*. The table of contents and approval record of each is included as Attachment C.

The health physics portion of the procedure addresses special controls/requirements for: (1) external radiation, (2) airborne radioactivity, (3) surface contamination, (4) protective clothing, (5) individual monitoring, (6) protective equipment, (7) processing requirements, (8) material storage requirements and (9) packaging/shipping requirements.

NFS also maintains a medical monitoring plan designed to protect the long- and short-term health of NFS employees and include at a minimum pre-employment physicals, annual physicals, post-employment physicals, personnel exposure logs and, when applicable, physicals to qualify for the wearing of respirators, *in-vivo* counting and bioassays.

NFS is insured with worker's compensation coverage for injuries and illnesses arising from the course of employment in accordance with the requirements of the State of Tennessee. Individual accident and injury/illness files are maintained whether or not they are OSHA recordable and whether or not they generate a workman's compensation claim.

All employees receive annual safety training that includes hazards communication. Supervisors receive additional training in accident investigation, incident report filing, and supervision of operations and hourly workers. Periodic training and safety sessions are held to familiarize employees with new equipment, hazards, and technology and to reinforce prior training. Required training programs are conducted by or overseen by an in-house training group. Training records for all employees are maintained in a centralized data file.

13.2 Treatability Program

13.2.1 Opening of Contaminated Soil Containers

Once the mercury contaminated soil has been received at the facility, containers will be opened while observing health and safety protocol. The health and safety procedures are formal, controlled documents that describe how jobs are to be performed safely and are

written to meet 29 CFR 1910 requirements. As part of the Health and Safety Program, only those personnel that have been trained on applicable procedures are allowed to perform work on the treatment project.

Personnel that open soil containers will wear appropriate protective clothing. For most work at NFS, including treatability studies, re-usable and/or dedicated personal protective clothing is used. Coveralls and shoe covers are maintained through an on-site radiological laundry that minimizes the generation of secondary waste materials. Alternatively, protective shoes may be dedicated to a specific controlled area. Gloves may be washable or disposable. The need for respiratory protection is determined on a case-by-case basis depending on the monitoring results of field instruments.

13.2.2 Sorting Soil

Packing material used to transport the contaminated soil is segregated from the soil itself. This material is evaluated to determine whether it is contaminated with radioactivity and/or is characteristically hazardous. Packing material that is radioactive only (and meets the acceptance criteria of the receiving disposal facility) is containerized and stored separately from the soil to be treated. If the material exhibits the hazardous characteristic for mercury, it will be submitted to treatability. Similarly, if the packing material is neither radioactive nor is characteristically hazardous, the material will be containerized and stored separately from the soil until sufficient material is accumulated for economical handling of the material for transport and disposal at a permitted disposal facility.

13.2.3 Gas and Liquid Discharges

Air Discharges. All process facilities and laboratories performing treatability studies are maintained under negative pressure. Exit air is through laboratory hoods and/or dedicated exhaust system that are discharged through a single, permitted stack. Air emissions from the R&D facility are permitted by the State of Tennessee (Permit Number 042347P).

Liquid Discharges. All process wastewaters are treated at a central wastewater treatment facility on site. Treated process wastewater is discharged under State of Tennessee NPDES permit TN0002038.

13.2.4 Spill Management and Emergency Response

All operations at the Erwin facility fall under and must comply with the NFS *“Spill Prevention Control and Countermeasure (SPCC) Plan, Revision 2.”* This controlled document is periodically reviewed/updated and includes activities associated with treatability studies.

14.0 References

- 1 “Stabilization Demonstration for Mercury-Contaminated Mixed Waste”, Gregory A. Hulet, Thomas B. Conley, Michael I. Morris, paper presented at the AIChE Spring Meeting, March 15-18, Houston, Texas (1999).
- 2 Mercury Contamination – Amalgamate (contract with NFS and ADA) Demonstration of DeHg® Process (TMS 1675) ITSr report from MWFA – MER01 project). Project presents both INEEL and K-25 mercury amalgamations.
- 3 Demonstration of the NFS DeHg® Process for Stabilizing Mercury (<260 ppm) contaminated mixed (TMS2229) ITSr Report from MWFA – MER02). Study does show impact of DeHg® on other RCRA metals.
- 4 Demonstration of the Nuclear Fuel Services, Inc. (NFS) DeHg Stabilization Process for Treatment of Radioactively Contaminated Wastes Containing >260 PPM Mercury (MER03) – September 2000.
- 5 Demonstration of the Nuclear Fuel Services, Inc. (NFS) DeHg® Stabilization Process for Treatment of Various Mercury Species in Surrogate Waste Containing >250 ppm Mercury (MER04) – September 2001.
- 6 DOE/EM-0472 Mercury Contamination – Amalgamate (contract with NFS and ADA) Stabilize Elemental Mercury Wastes Mixed Waste Focus Area Prepared for . . . URL: http://apps.em.doe.gov/ost/pubs/itsrs/itsr1675_2.pdf
- 7 DOE/EM-0471 Mercury Contamination – Amalgamate (contract with NFS and ADA) Demonstration of DeHg SM Process Mixed Waste Focus Area Prepared for U.S. . . . URL: <http://apps.em.doe.gov/ost/pubs/itsrs/itsr1675.pdf>
- 8 TMFA Documents “. . . Contamination – Amalgamate (contract with NFS and ADA) Stabilize Elemental Mercury Wastes (TMS 1675). . . Permit Procedures for Mixed Waste Treatment (99). . . URL: tmfa.inel.gov/Documents/AllDocs.asp
- 9 DOE/EM-0471 Mercury Contamination – Amalgamate (contract with NFS and ADA) Demonstration of DeHg SM Process Mixed Waste Focus Area Prepared for U.S. . . . URL: <http://apps.em.doe.gov/ost/pubs/itsrs/itsr2229.pdf>
- 10 DOE demonstration Projects: Mercury Amalgamation (MER01; Mercury Stabilization <260 ppm (MER02; Mercury Stabilization >250 pm (BNL Soils). . . URL:

<http://web.aed.anl.gov/TechCon/Projects/mercury/resources/docs/nfs.ppt>

- 11** Stabilization of Wastes Contaminated with >260 ppm Hg. . . . LMES MER02 ORNL Poplar Creek Hg Amalgamation. Commercial projects with wastes containing mercury and other metals. . . .URL:
web.ead.anl.gov/TechCon/Projects/mercury/resources/docs/itcorporation.ppt
- 12** TechCon Organization Capabilities Questionnaire “ . . .a variety of matrices. NFS permitted DeHg® in the State of Tennessee and operated a treatment system to convert more than 50m3 of mercury-mixed waste to a . . .” URL:
web.ead.anl.gov/TechCon/Public2/ShowCap.cfm?GetWhat=NUCLFUELHG&Interest+ORHG
- 13** TechCon Organization Capabilities Questionnaire “ . . .a variety of matrices. NFS permitted DeHg® in the State of Tennessee and operated a treatment system to convert more than 50m3 of mercury-mixed waste to a . . .” URL:
web.ead.anl.gov/TechCon/Public2/ShowCap.cfm?GetWhat=estine
- 14** U.S. Environmental Protection Agency (USEPA), 1987, "Data Quality Objectives for Remedial Response Activities."
- 15** U.S. Environmental Protection Agency (USEPA), "USEPA Contract Laboratory Program Statement of Work for Inorganics Analysis, SOW No. 788, Exhibit E, Quality Assurance/Quality Control Requirements."