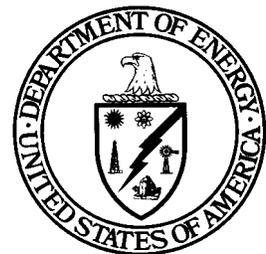




Summary Report DOE/EM-0487

# Demonstration of GTS Duratek Process for Stabilizing Mercury Contaminated ( $<260$ ppm) Mixed Wastes

Mixed Waste Focus Area



*Prepared for*  
**U.S. Department of Energy**  
Office of Environmental Management  
Office of Science and Technology

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# Demonstration of GTS Duratek Process for Stabilizing Mercury Contaminated ( $<260$ ppm) Mixed Wastes

OST Reference #2409

Mixed Waste Focus Area



*Demonstrated at*  
GTS Duratek, Inc.  
Kingston, Tennessee



## ***Purpose of this document***

Innovative Technology Summary Reports are designed to provide potential users with the information they need to quickly determine if a technology would apply to a particular environmental management problem. They are also designed for readers who may recommend that a technology be considered by prospective users.

Each report describes a technology, system, or process that has been developed and tested with funding from DOE's Office of Science and Technology (OST). A report presents the full range of problems that a technology, system, or process will address and its advantages to the DOE cleanup in terms of system performance, cost, and cleanup effectiveness. Most reports include comparisons to baseline technologies as well as other competing technologies. Information about commercial availability and technology readiness for implementation is also included. Innovative Technology Summary Reports are intended to provide summary information. References for more detailed information are provided in an appendix.

Efforts have been made to provide key data describing the performance, cost, and regulatory acceptance of the technology. If this information was not available at the time of publication, the omission is noted.

All published Innovative Technology Summary Reports are available on the OST Web site at <http://OST.em.doe.gov> under "Publications."

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## SECTION 1

# SUMMARY

### ***Technology Summary***

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Mercury-contaminated wastes in many forms are present at various U. S. Department of Energy (DOE) sites. Based on efforts led by the Mixed Waste Focus Area (MWFA) and its Mercury Working Group (HgWG), the inventory of wastes contaminated with < 260 ppm mercury and with radionuclides stored at various DOE sites is thought to be approximately 6,000 m<sup>3</sup> (Conley, Morris, Osborne-Lee, and Hulet 1998). At least 26 different DOE sites have this type of mixed low-level waste in their storage facilities. Extraction methods are required to remove mercury from waste containing >260 ppm levels, but below 260 ppm Hg contamination levels the U. S. Environmental Protection Agency (EPA) does not require removal of mercury from the waste. Steps must still be taken, however, to ensure that the final waste form does not leach mercury in excess of the limit for mercury prescribed in the Resource Conservation and Recovery Act (RCRA) when subjected to the Toxicity Characteristic Leaching Procedure (TCLP). At this time, the limit is 0.20 mg/L. However, in the year 2000, the more stringent Universal Treatment Standard (UTS) of 0.025 mg/L will be used as the target endpoint.

Mercury contamination in the wastes at DOE sites presents a challenge because it exists in various forms, such as soil, sludges, and debris, as well as in different chemical species of mercury. Stabilization is of interest for radioactively contaminated mercury waste (<260 ppm Hg) because of its success with particular wastes, such as soils, and its promise of applicability to a broad range of wastes. However, stabilization methods must be proven to be adequate to meet treatment standards. They must also be proven feasible in terms of economics, operability, and safety. To date, no standard method of stabilization has been developed and proven for such varying waste types as those within the DOE complex.

The MWFA is investigating possible stabilization methods for mercury-contaminated mixed waste streams and has funded demonstrations, several of which have been completed. The Technology Development Requirements Document (TDRD), developed by the MWFA, requires that the effectiveness of newly developed technologies be proven. New technology for mercury stabilization must adequately stabilize waste to the new UTS, and must provide measuring and monitoring methods to verify the process. In addition, the new process should:

- minimize worker exposure,
- minimize volume increase as waste is treated,
- minimize secondary waste generation,
- Maximize operational flexibility and radionuclide containment.

This report summarizes the findings from a stabilization technology demonstration conducted by GTS Duratek, Inc., under MWFA sponsorship.

### ***Demonstration Summary***

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When the MWFA contacted industry to determine the commercial capabilities in mercury-contaminated mixed waste stabilization, little actual waste had been treated. The MWFA's HgWG initiated actions to ensure that the commercial sector could safely treat DOE's mercury-contaminated mixed waste, to reduce the potential liability for future waste treatment efforts. Los Alamos issued a contract to GTS Duratek (GTSD) for a treatability study on actual waste. The MWFA added scope to the contract to gather additional data from the study. The GTSD demonstration of mercury waste stabilization is the primary focus of this report.

In addition, three vendors, Allied Technology Group (ATG), Inc., International Technologies, Inc., and Nuclear Fuel Services, Inc. (NFS), stabilized surrogate waste spiked with several mercury species in bench-scale studies. This work was performed as part of the work funded under the HgWG's MER02 Request for Proposal (RFP) for mercury-mixed-waste treatment. The results of these tests are reported



elsewhere. Two vendors, ATG and NFS, then demonstrated their pilot-scale processes on actual waste (MWFA 1999A, MWFA 1999B).

The GTSD demonstration consisted of a four-phase treatability study on sludge and laboratory residues generated at the Los Alamos National Laboratory (LANL). Phase I of the study involved receipt and repackaging of the material, followed by preparations for waste tracking. Phase II examined the bench-scale performance of grouting at two different loadings of waste to grouted mass. Phase III demonstrated in-drum mixing and solidification using repackaged drums of sludge. Phase IV initially intended to ship final residues to Envirocare for disposal. The sludge was generated from the LANL Phase Separators and Caustic Tank in the Phase Separator Pit and is a mixed waste due to the presence of radionuclides, heavy metals, and RCRA-listed organic compounds. This study was conducted at the GTSD Bear Creek Operations Facility located in Oak Ridge, Tennessee, from September 1997 through September 1998.

The goals of the demonstration were twofold: (1) evaluate the efficacy of Portland cement-based grout to stabilize this actual mixed waste material and (2) generate a disposable waste form, that meets the Envirocare Waste Acceptance Criteria (WAC) and RCRA Land Disposal Restrictions (LDR).

GTSD received four 55-gallon drums containing over 568 kg (i.e., 1,253 lb) of LANL sludge, which were repackaged to exclude plastics and liners internal to the 55-gallon drums. Bench-scale solidification tests at a low and a high waste loading were performed on composite samples of the raw sludge. GTSD evaluated compliance of bench scale solidification tests, at low waste loading, with Phase III RCRA LDR and Envirocare WAC. GTSD also evaluated compliance of bench-scale solidification tests at low waste loading with the Phase III and Phase IV RCRA LDR, for selected organic compounds and TCLP metals. One high-load bench-scale formulation failed to meet the Phase IV UTS for mercury.

GTSD was successful in demonstrating the full-scale drum solidification capabilities using the bench-scale low-waste-loading formula. Also, GTSD achieved compliance with the previous criteria for low and, except in one case, high waste loading. However, for certain organic compounds, LDR criteria (Phase III or Phase IV) were not met by tests at either loading of the solidified waste form and Envirocare WAC were not met. Unexpectedly high levels of organic compounds were encountered in the waste composed of laboratory residues. As is well-established, high levels of organic compounds can make stabilization difficult, if not impracticable (Connor 1990). In addition, GTSD found that radionuclide levels in the laboratory residues were much higher than expected, in some instances by an order of magnitude or more, based on characterizations of the waste provided to GTSD.

These two important factors pose serious challenges to stabilization efforts, and pretreatment steps will likely be necessary for such wastes. A thermal pretreatment step, for example, could be an effective means to remove or reduce organic compounds. However, the need for such a pretreatment was not anticipated based on initial characterization of the wastes. Also, although a successful formulation for higher radionuclide levels in the waste is feasible, the grout formulations developed in this demonstration were based on the initial characterization data, which were later found to contain inaccuracies.

Since the final grouted material was not in compliance with Envirocare requirements, it was returned to LANL for long-term storage. GTSD has begun to evaluate alternatives to address the complicating factors of high organic compounds and high radionuclide content. Further processing of the drums may be considered based on the availability and cost of alternatives.

## Key Results

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The key results of the demonstration are as follows:

- Solidification tests were performed at low and high waste loading, resulting in stabilization of mercury to meet the UTS of 0.025 mg/L at the low loading and for two of the three runs at the high loading. The third high-loading run had a TCLP of 0.0314 mg/L.
- Full-drum stabilization using the low loading formula was demonstrated.
- Organic compound levels were discovered to be higher than originally reported, including the presence of some pesticides. Levels of some radionuclides were much higher than initially reported to GTSD.



- Compliance with the Phase III RCRA LDR and the Envirocare WAC was evaluated. Compliance with Phase III and Phase IV RCRA LDR for selected organic compounds and TCLP metals was also evaluated. Because of high organic and radionuclide levels, the final waste form could not be disposed at Envirocare.
- Volume increases, secondary waste expectations, and life-cycle costs were not estimated, due to the need for: (1) further development to match treatment process with actual waste characteristics or (2) testing of the GTSD process with a waste matching the design profile.

## Contacts

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This report was prepared by the Mercury Working Group and Prairie View A&M University.

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## SECTION 2

# TECHNOLOGY DESCRIPTION

### Overview

The HgWG has identified approximately 8,000 m<sup>3</sup> of mercury-contaminated, mixed low-level and transuranic (TRU) wastes in the DOE complex. In addition to elemental mercury, these waste streams include sludges, soils, and debris waste with mercury concentrations ranging from less than 2 ppm to greater than 50,000 ppm. Approximately 6,000 m<sup>3</sup> of these wastes are contaminated with <260 ppm mercury. RCRA regulations require that mercury wastes with contamination levels at or above 260 ppm Hg be recovered by a thermal process, such as retorting, and stabilized using an amalgamation process. No specific treatment method is specified for wastes containing <260 ppm; however, RCRA regulations require that such wastes that exceed a mercury concentration<sup>1</sup> of 0.20 mg/L be treated by a suitable method to meet this standard.

The HgWG conducted a source selection for vendors to participate in demonstrations of different types of technologies capable of stabilizing wastes containing <260 ppm of mercury to meet the TCLP limit. Until recently, no studies beyond bench scale had been conducted on the amalgamation and stabilization of mixed mercury wastes. The primary technical issue associated with the treatment of such waste was related to scale-up of the process to a cost-effective operations level. However, the HgWG now reports the completion of three technology demonstrations on the stabilization of mixed wastes contaminated with mercury at levels <260 ppm.

GTSD, headquartered in Kingston, Tennessee, has recently applied its stabilization technology at bench and pilot scale to treat several drums of sludge and laboratory residues from LANL. ATG, located in Fremont, California, has applied its stabilization process, employing bench- and demonstration-scale processes to treat ion exchange resin from Piketon Gaseous Diffusion Facility (PORTS). NFS, located in Erwin, Tennessee, has demonstrated its proprietary stabilization process on a second sample of the PORTS ion exchange resin. The ATG and NFS technology demonstrations are each reported elsewhere in separate ITSRs (MWFA 1999A, MWFA 1999B). The GTSD technology demonstration is the focus of this report. Other recent ITSRs address the treatment technology for the amalgamation of radioactive elemental mercury (MWFA 1998A, MWFA 1998B). An additional Oak Ridge report summarizes the results of tests by three vendors, ATG, IT, and NFS, to determine the effects of speciation on the stabilization of mercury wastes (Osborne-Lee 1999).

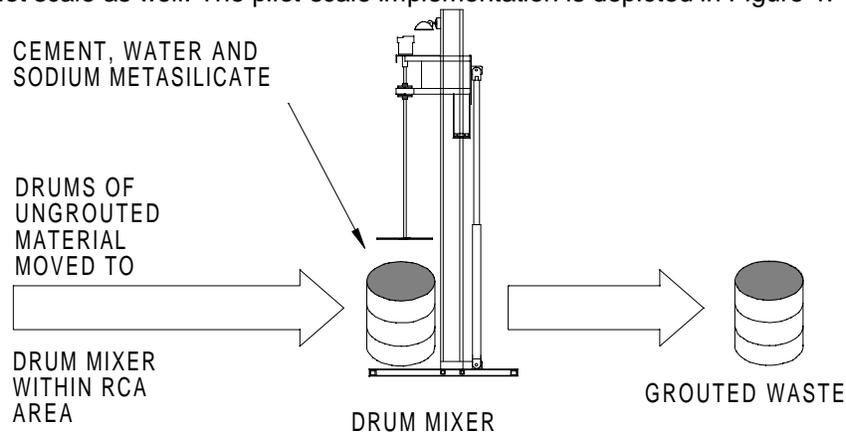
### GTSD Process Definition

The TDRD for Mercury Stabilization (DOE 1996) referred to many DOE sites that contain debris and sludges that are contaminated with mercury in many forms. The document also noted that then-existing commercial processes for stabilization of mercury in mixed waste were inadequate, or at least not yet demonstrated.

<sup>1</sup> As determined by Environmental Protection Agency (EPA) SW-846 Method 1311 Toxic Characteristic Leaching Procedure (TCLP).



GTSD developed a process for waste stabilization that involves the addition of water and then cement to samples of sludge. The grout mixtures are then blended with a stabilization agent, sodium metasilicate. A 2-day curing period follows. Once curing is done, the sample containers are sealed and kept for testing, including chemical analysis, isotopic determinations, and TCLP tests. GTSD developed this process at the bench scale. Formulations are also developed with bench scale tests. GTSD implemented their process at the pilot scale as well. The pilot-scale implementation is depicted in Figure 1.



**Figure 1. Pilot-scale representation of the GTS Duratek stabilization process.**

The initial objective of the GTSD demonstration was to evaluate stabilization or grouting methods necessary to immobilize the radioactive components and RCRA regulated components of the sludge as referenced in the LDR criteria applicable under Phase III of the LDRs in 40 CFR 268. Subsequent to the publication of the Phase IV limits in the 26 May 1998 Federal Register, the project scope was modified to include evaluation with respect to the same restricted list of parameters under the new Phase IV RCRA and LDR limits. Hence, a secondary objective was to evaluate compliance with the Phase IV limits using the maximum waste loading possible in the formation of a grouted waste. This is the “high” waste loading referred to in this report.

## GTSD System Operation

### Materials

The sludge (four drums) was generated at LANL upon the addition of a caustic solution to the condensate and particulates removed from laboratory fume hood exhausts by the phase separators in Building 2 of the TA35 facility. Three small containers of lab packs developed during LANL's analysis of the sludge before shipment of the material to GTSD were also included with the shipment and defined under the same chemical analysis as was the phase separator sludge. Condensates and solids had been generated by passage of laboratory exhausts through three phase separators operated in series.<sup>2</sup> Condensate and particulate material from each of the phase separators were routed to one of three tanks (TSL-4, TSL-5, and TSL-6). Liquid (condensate) from these tanks was pumped to a final tank where sodium hydroxide (caustic soda) was added until the isoelectric point of the waste was attained. Liquid from the caustic tank was then disposed at LANL. Solids from the four tanks and the waste laboratory materials involved in analysis of the material were placed into drums. These wastes comprise the TA35 sludge treated in the demonstration that is the subject of this report.

### Product Specifications

Table 1 lists the parameters indicated in LANL laboratory data as present in detectable quantity and the applicable disposal limit. Since data for RCRA “D” characteristic parameters were furnished by LANL in mg/kg rather than mg/L, TCLP and the manifest did not reference these parameters, it was assumed at GTSD that the material passed TCLP testing procedures for those parameters. Organic compounds shown as above detectable quantities, but below UTS limits, are also listed in the tabulation. Any parameter listed as below detection limits is excluded from the tabulation.<sup>2</sup>

The final grouted product must comply with all hazardous components regulated by EPA through RCRA as characteristic or listed wastes, including those not shown in Table 1, if present.

<sup>2</sup> This system is called the Phase Separator Pit (PSP) at LANL.



**Table 1. Disposal criteria applicable to Los Alamos National Laboratory wastes**

RCRA code	Parameter <sup>a</sup>	Quantity in Los Alamos National Laboratory data <sup>b</sup>	Envirocare WAC/RCRA limit	Complies with limit? (Yes/No) <sup>c</sup>
(D003)	(Sulfides, Reactive)	N/A	<60 mg/kg	Yes
(D004)	(Arsenic)	4.2 mg/kg	5 mg/L TCLP	Yes
(D005)	(Barium)	230 mg/kg	7.6 mg/L TCLP	?
D006	Cadmium	31 mg/kg	0.19 mg/L TCLP	?
D007	Chromium	690 mg/kg	0.86 mg/L TCLP	?
D008	Lead	230 mg/kg	0.37 mg/L TCLP	?
D009	Mercury	230 mg/kg	0.25 mg/L TCLP	?
(D010)	(Selenium)	<5.3 mg/kg	0.16 mg/L TCLP	?
(D011)	(Silver)	67 mg/kg	0.3 mg/L TCLP	?
	(Antimony)	Not Reported	2.1 mg/L TCLP	?
	(Beryllium)	<1.8 mg/kg	0.014 mg/L TCLP	?
	(Fluoride)	1.2 mg/kg	N/A	Yes
	(Nickel)	220 mg/kg	5 mg/L TCLP	?
	(Thallium)	10 mg/kg	0.78 mg/L TCLP	?
	(Vanadium)	<7.1 mg/kg	0.23 mg/L TCLP	?
	(Zinc)	270 mg/kg	5.3 mg/L TCLP	?
D028	1,2 Dichloroethane	<0.005 mg/kg	6 mg/kg & 0.5 mg/L TCLP	Yes
(D029)	(1,1 Dichloroethylene)	11,000 mg/kg	6 mg/kg & 0.7 mg/L TCLP	No
D043	Vinyl Chloride	220 mg/kg	6 mg/kg & 0.2 mg/L TCLP	No
F002	Tetrachloroethylene	0.037 mg/kg	6 mg/kg	Yes
	Methylene Chloride	12,000 mg/kg	30 mg/kg	No
	Trichloroethylene	<0.005 mg/kg	6 mg/kg	Yes
	1,1,1-trichloroethane	19,000 mg/kg	6 mg/kg	No
	Chlorobenzene	0.039 mg/kg	6 mg/kg	Yes
	1,1,2-trichloro-1,2,2-trifluoroethane	Not Reported	30 gm/kg	?
	Ortho-dichlorobenzene	<0.66mg/kg	6 mg/kg	Yes
	trichlorofluoromethane	<0.005 mg/kg	30 mg/kg	Yes
	1,1,2-trichloroethane	<0.005 mg/kg	6 mg/kg	Yes
F005	Toluene	0.052 mg/kg	10 mg/kg	Yes
	Methyl ethyl ketone	<0.0099 mg/kg	36 mg/kg	Yes
	Carbon disulfide	<0.005 mg/kg	4.8 mg/L TCLP	Yes
	Isobutanol	not reported	170 mg/kg	?
	Pyridine	not reported	16 mg/kg	?
	Benzene	<0.005 mg/kg	10 mg/kg	Yes
	2-ethoxyethanol	not reported	INCIN	?
	2-nitropropane	not reported	INCIN	?
UTS	4-Methyl-2-pentanone	0.62 mg/kg	33 mg/kg	Yes
(UTS)	(Chloroform)	1,500 mg/kg	6 mg/kg	No
(UTS)	(Chloromethane)	300 mg/kg	30 mg/kg	No
UTS	Ethyl Benzene	0.015 mg/kg	10 mg/kg	Yes
UTS	o, m, p Xylene	0.263 mg/kg	30 mg/kg	Yes
UTS	bis (2-Ethylhexyl) phthalate	2.9 mg/kg	28 mg/kg	Yes

<sup>a</sup> Manifest documentation does make reference to items in shown parentheses.

<sup>b</sup> Organic compounds present at less than detection limits are not shown.

<sup>c</sup> “?” indicates that no TCLP data were provided.



## Methods

The demonstration project was conducted in four phases, as shown in Table 2. Aspects of each project phase are discussed below.

**Table 2. Project phases for the GTS Duratek solidification demonstration**

Phase	Event(s)
I	Reception Repackaging Inventory Waste tracking Procedures
II	Bench-scale testing
III	Solidification using an in-drum mixing technique.
IV	Disposal at Envirocare or return to LANL

Additional methods and procedures, including the test plan, are detailed elsewhere (GTSD 1999, Appendix A-1). Note that sampling and analysis procedures are required to be in compliance with RCRA and Envirocare WAC.

### Waste Tracking System

A tracking system was developed to enable the tracking of waste quantities as small as 250 g. The system consists of a waste tracking diagram and a hazardous-wastes daily inventory. The tracking diagram contains detailed information, including the following:

- identification and mass applied to each container of raw waste,
- relabeling identification and mass at each relabeling event at GTSD,
- type of container,
- labeling identification of the various samples collected for bench-scale testing,
- amounts of cement, water, and sodium metasilicate added to each container,
- amount of plastic removed from each drum,
- summary tabulation of dates of treatment and quantities treated,
- mass and disposition of secondary wastes,
- mass and disposition of plastics,
- mass of metallic wastes routed to compaction (rusty LANL drums and sample containers),
- total mass of the eight drums of grouted material formed during the project,
- destination of grouted material.

The hazardous wastes daily inventory includes the date waste was treated or routed to laboratory analysis and the quantity of "as-received" waste involved.

### Bench-Scale Test

Grouting formulations were based upon the standard GTSD grouting formula. The standard GTSD formulation is usually applied to materials that are composed of ash or other dry solids. A water to cement ratio of 0.7 parts cement to 1.0 part of water is employed. As shown below, this results in a standard waste-to-grout loading of 0.4 parts of "dry solids" waste to 1 part of grouted weight plus addition of sodium metasilicate at approximately 9% of the total grouted weight. Thus, the actual waste loading is near 0.37. Mathematically, this is expressed as:

$$0.4 = M_W / (M_C + M_{H_2O} + M_W)$$



where  $M_W$  is the mass of waste (dry solids),  $M_C$  is the mass of cement, and  $M_{H_2O}$  is the mass of water. So, if  $M_{SM}$  is the mass of sodium metasilicate, the following relations express the formulation:

$$M_{H_2O} = 0.7 \times M_C = 0.61 \times M_{DS}$$

$$M_C = 0.88 \times M_W$$

Bench-scale tests were performed by adding water (0 to approximately 400 grams), followed by adding cement (0 to 1,033 grams) to 250-gram or 350-gram samples of sludge. The grout mixtures were then blended with sodium metasilicate. The samples were allowed to cure for 2 days. At the end of curing, the samples were closed and subjected to chemical analysis and isotopic determinations.

The repackaged LANL waste was 89.2% moisture. Thus, 1 kg of wet repackaged waste contained 0.108 kg of dry solids and 0.892 kg of water. Based upon use of  $0.7 \times M_C$  for the amount of water required, a 1 kg mass of wet repackaged sludge would require 0.892 kg of water which, in turn, would require 0.624 kg of cement for hydration and mixing. Thus, formation of grout using 1 kg of repackaged wet waste would result in 2.53 kg of grouted material. Hence, the waste loading, based upon initial repackaged total wet sludge weight would be 0.395. Finally, 0.23 kg of sodium metasilicate would be required and produce a final grouted mass of 2.75 kg or an apparent waste loading of  $1 \div 2.75 = 0.36$ . On a dry solids basis, the loading example discussed above is based upon 0.108 kg of dry sludge, resulting in a true dry solids loading of  $0.108 \div 2.75 = 0.039$ . This is the true waste loading of the high-waste-loading series of tests (i.e., those samples used to evaluate ORNL goals).

The LANL goals required formation of a grouted mass at substantially lower loading. To achieve this, 350 g samples of wet repackaged sludge were used. These samples contained approximately 325 g of water to which 400 g of additional water was added. To the resulting 725 g of water, approximately 1,025 g of cement was added to give a grouted weight of 1,775 g of grout. Addition of 31.5 g of sodium metasilicate resulted in a final grouted weight of 1,806 g. Based upon 25 g of dry solids in the original 350 g wet-sludge sample, the actual waste loading achieved was  $25 \div 1,806 = 0.014$  on a dry-solids basis. This was the low waste loading used to evaluate the LANL criteria.

### **Pilot-Scale Procedure**

Phase III testing was accomplished in drums using a vertical in-drum mixer (see Figure 1). The quantity of sludge in each drum was previously determined during reception and repackaging activities. The low-loading formula and addition sequence as described in GTSD Procedure LANL-PRD-0-01 (GTSD 1999, Appendix A-1) was used to form the drummed grout quantities in the pilot-scale phase of tests.

After blending, the grouted material was allowed to cure in drums for 48 hours and then the drums were closed. Grouted materials were then moved back to the hazardous waste storage area until the last drum was processed. As grout was being formed in the drums, grouted samples were formed in the laboratory with combined sample material using the low-loading formulation. The grouted samples were also allowed to cure for 2 days before being shipped to Barringer Laboratories for the full analytical determination required by Envirocare.



## SECTION 3

# PERFORMANCE

### Phase I—Waste Characterization

The initial characterization of waste shipped from LANL was extensive and GTSD (and its subcontract laboratories) performed additional characterization for the raw material. Together, the analysis included 43 isotopes, 87 volatile organic compounds, 14 other organic compounds, 32 inorganic compounds (including mercury), and 38 compounds classifiable as pesticides and herbicides. Tables 3 and 4 show a partial comparison of characterization data provided by LANL with that obtained by GTSD for isotopes and inorganic compounds. GTSD did not analyze for organic compounds in the initial phase of the project.

**Table 3. Comparison of raw sludge characterization data from Los Alamos National Laboratory (LANL) and GTS Duratek (GTSD) for select isotopes**

Isotopes	LANL data PicoCi/g	Sample A PicoCi/g <sup>a</sup>	Sample B PicoCi/g <sup>a</sup>	Composite sample PicoCi/g <sup>a</sup>
<sup>241</sup> Americium	128	1,617	422	820
<sup>137</sup> Cesium	9.4	138	31	67
<sup>51</sup> Chromium	-	<23	<5	<11
<sup>60</sup> Cobalt	55.6	915	62	347
<sup>238</sup> Plutonium	370	899	120	380
<sup>239/240</sup> Plutonium	19,000	57,200	8,790	24,927
<sup>89/90</sup> Strontium	3,940	318,000	48,400	138,267
<sup>3</sup> Tritium	1,210	644	693	677
<sup>233/234</sup> Uranium	39.4	754	263	427
<sup>235</sup> Uranium	1.79	32	6	14
<sup>238</sup> Uranium	55	1,200	387	658

<sup>a</sup> These data were obtained by GTSD through a Utah certified laboratory.

**Table 4. Comparison of raw sludge characterization data from Los Alamos National Laboratory (LANL) and GTS Duratek (GTSD) for select inorganic compounds**

Nonorganic	LANL Data mg/kg	Sample A mg/L TCLP <sup>a</sup>	Sample B mg/L TCLP <sup>a</sup>	Composite GTSD mg/L TCLP <sup>a</sup>
Mercury	230	0.1840	0.0399	0.1250
Arsenic	4.2	0.12	<0.07	<0.5
Barium	230	2.40	1.20	9.2
Cadmium	31	0.21	0.03	0.076
Chromium	690	0.30	0.10	<0.08
Cyanide, Reactive, Total	<0.2	<66.8 mg/kg	<67.5 mg/kg	-
Lead	230	0.65	0.29	<0.037
Nickel	220			0.7
Selenium	<5.3	<0.07	<0.07	<0.016
Silver	67	0.02	0.10	<0.03
Sulfur, Total	70	<66.8	<67.5	-

<sup>a</sup> These data were obtained by GTSD through a Utah certified laboratory.



## Phase II—Bench-Scale Tests

Bench-scale grouting tests used 250 or 350-g samples of wet sludge. High loading of grout samples was achieved using 250 g, while low loading was performed with 350-g samples to achieve the required detection limits during subsequent testing. These sample sizes minimized the amount of returned residue from the analytical laboratories. Given this selection of sample sizes, the amount of water and cement added to the samples determines the ultimate waste loading. The formulations developed in bench-scale testing are shown in Table 4.

**Table 4. Bench-scale formulation data developed by GTS Duratek**

Sample ID	A7013190	A7013190	A7013192	A7013192	A7013189	A7013189	A7013189
Waste loading	High load <sup>a</sup>	Low load <sup>b</sup>	High load <sup>a</sup>	Low load <sup>b</sup>	High load <sup>a</sup>	Low load <sup>b</sup>	Low load <sup>b</sup> with PAC
Test Weights	FORMULATION VALUES (all data in grams)						
Sample	250	350	250	350	350	350	350
Pre-wetted powdered activated carbon (PAC) added	0	0	0	0	0	0	3
Cement added	220	1,008	220	1,033	383	1,033	383
H <sub>2</sub> O added	0	399.7	0	398.0	0	411	0
H <sub>2</sub> O in sample	218.5	305.9	232.3	325.15	312.3	312.3	312.3
Sodium metabisulfite added	22.5	31.5	22.5	31.5	31.5	31.5	31.5
Weight of BS sample (after water, grout & Sodium metabisulfite addition)	492.5	1,789	492.5	1,812.5	764.5	1,825.5	1,175.5
Weight of BS sample (after evaporation allowance @ 30% of total H <sub>2</sub> O)	427.0	1,577.5	422.8	1,595.5	670.8	1,731.8	1,269.2
Mass increased by multiplier of	1.708	4.507	1.691	4.559	1.917	4.948	3.626

<sup>a</sup> The high loading case, of particular interest to end users at Oak Ridge National Laboratory (ORNL), involves no added water. Also, no water is removed before grouting.

<sup>b</sup> The low loading case, of particular interest to end users at Los Alamos National Laboratory (LANL), involves adding water to maximize capacity for radionuclide content in the final waste form.

Laboratory analysis results of samples grouted according to the formulation shown in Table 4 are shown in Tables 5 and 6. The PAC results were discarded after the TCLP procedures were determined to have been used for the volatile-organics determinations. Further details, including sample identifiers, data reports, and quality control documentation may be found elsewhere (GTSD 1999).

As shown in Table 6, bench-scale tests successfully stabilized mercury to the 0.025 mg/L UTS for the low-loading runs, although the grouted samples would fail to meet disposal WAC at Envirocare due to other constituents, such as <sup>90</sup>Sr. Two of the high-loading runs met the UTS, while the third one failed.



**Table 5. Isotopic analysis results for bench scale grouted samples<sup>a</sup>**

Isotope	Units	Grouted sample A		Grouted sample B		Grouted composite sample	
		Low load <sup>b</sup>	High load <sup>c</sup>	Low load <sup>b</sup>	High load <sup>c</sup>	Low load <sup>b</sup>	High load <sup>c</sup>
<sup>228</sup> Actinium	picoCi/g	-	-	-	-	15	32
<sup>241</sup> Americium	picoCi/g	378	1,243	72	281	329	644
<sup>125</sup> Antimony	picoCi/g	<4	7	<1	<2	-	-
<sup>212</sup> Bismuth	picoCi/g	-	-	-	-	14	29
<sup>141</sup> Cerium	picoCi/g	<2	<5	<1	<1	-	-
<sup>144</sup> Cerium	picoCi/g	<10	<23	<3	<7	-	-
<sup>134</sup> Cesium	picoCi/g	<1	<2	"0"	<1	-	-
<sup>137</sup> Cesium	picoCi/g	36	108	7	24	36	68
<sup>51</sup> Chromium	picoCi/g	<11	<14	<2	<7	-	-
<sup>57</sup> Cobalt	picoCi/g	<2	<4	<1	<1	-	-
<sup>58</sup> Cobalt	picoCi/g	<1	<2	"0"	<1	-	-
<sup>60</sup> Cobalt	picoCi/g	214	673	14	46	152	287
<sup>152</sup> Europium	picoCi/g	<3	<6	"0"	<2	-	-
<sup>154</sup> Europium	picoCi/g	-	-	-	-	2.0	<1.0
<sup>155</sup> Europium	picoCi/g	<8	<18	<3	<5	-	-
<sup>131</sup> Iodine	picoCi/g	<1	<3	"0"	<1	-	-
<sup>59</sup> Iron	picoCi/g	<3	<5	<1	<1	-	-
<sup>54</sup> Manganese	picoCi/g	<0.1	<3	<0.3	<1	-	-
<sup>94</sup> Niobium	picoCi/g	<1	<1.4	<0.2	<0.4	-	-
<sup>238</sup> Plutonium	picoCi/g	<72	<57.2	14	34	12	43
<sup>239/240</sup> Plutonium	picoCi/g	2,990	10,200	672	1,440	1,060	1,920
<sup>40</sup> Potassium	picoCi/g	8	<13	6	5	-	-
<sup>103</sup> Ruthenium	picoCi/g	<1	<2	0.3	0.5	-	-
<sup>106</sup> Ruthenium	picoCi/g	<8	<21	2	5	-	-
<sup>110m</sup> Silver	picoCi/g	<3	<5	1	2	-	-
<sup>89/90</sup> Strontium	picoCi/g	27,200	49,200	1,910	4,070	14,500	30,200
<sup>208</sup> Thallium	picoCi/g	-	-	-	-	4.7	8.3
<sup>232</sup> Thorium	picoCi/g	19	55	2	3	-	-
<sup>234</sup> Thorium	picoCi/g	-	-	-	-	80	149
<sup>113</sup> Tin	picoCi/g	<2	<3	1	1	-	-
<sup>3</sup> Tritium	picoCi/g	-	-	-	-	45	87
<sup>233/234</sup> Uranium	picoCi/g	57	206	27	59	47	86
<sup>235</sup> Uranium	picoCi/g	<2.9	14.1	2.6	4.9	2.0	5.0
<sup>238</sup> Uranium	picoCi/g	83	296	34	79	73	141
<sup>65</sup> Zinc	picoCi/g	<27	<7	1	1	-	-
<sup>95</sup> Zirconium	picoCi/g	<1	<2	0.4	1	-	-

<sup>a</sup> This is a partial listing of constituents analyzed. Those that were not detected in any sample or not analyzed for are not shown. A dash entry indicates that a particular analysis was not done.

<sup>b</sup> The low loading case, of particular interest to end users at Los Alamos National Laboratory (LANL).

<sup>c</sup> The high loading case, of particular interest to end users at Oak Ridge National Laboratory (ORNL).



**Table 6. Inorganic compound analysis results for bench-scale grouted samples<sup>a</sup>**

Parameter	TCLP Units	Grouted sample A		Grouted sample B		Grouted composite sample	
		Low load <sup>b</sup>	High load <sup>c</sup>	Low load <sup>b</sup>	High load <sup>c</sup>	Low load <sup>b</sup>	High load <sup>c</sup>
Mercury	mg/L	0.0169	0.0314	0.00127	0.00249	0.0114	0.0233
Sulfides, Reactive	mg/kg	-	-	-	-	80	80
Arsenic	mg/L	<0.07	<0.07	<0.07	<0.07	<0.5	<0.5
Barium	mg/L	2.2	1.4	1.3	0.85	2.8	3.4
Cadmium	mg/L	<0.01	<0.01	<0.01	<0.01	<0.019	<0.019
Chromium	mg/L	0.09	0.24	0.07	0.12	<0.08	0.09
Copper	mg/L	-	-	-	-	<0.05	<0.05
Cyanide, Reactive, Total	mg/kg	-	-	-	-	<0.3	<0.3
Lead	mg/L	<0.07	0.24	0.28	0.3	<0.037	<0.037
Nickel	mg/L	-	-	-	-	<0.5	<0.5
Selenium	mg/L	<0.07	<0.07	<0.07	<0.07	<0.016	<0.016
Silver	mg/L	0.08	<0.02	0.03	0.06	<0.03	<0.03
Sulfur, Total	mg/L	<6.76	<6.76	<6.76	<6.76	-	-
Thallium	mg/L	-	-	-	-	<0.078	<0.078
Vanadium	mg/L	-	-	-	-	<0.05	<0.05
Zinc	mg/L	-	-	-	-	<0.53	<0.53

<sup>a</sup> This is a partial listing of constituents analyzed. Those that were not detected in any sample or not analyzed for are not shown. A dash entry indicates that a particular analysis was not done.

<sup>b</sup> The low loading case of particular interest to end users at Los Alamos National Laboratory (LANL).

<sup>c</sup> The high loading case of particular interest to end users at Oak Ridge National Laboratory (ORNL).

### Phase III-Pilot-Scale Tests

The composition of grout formed during the pilot study is shown in Table 7. The in-drum mixer illustrated in Figure 1 was used to blend the quantities of materials to result in the values shown in the "Initial Total" column of Table 7. The drum weights after the drums were left open within the radiation controlled area and cured for 48 hours are shown in the "Cured Total" column. Condensation within the drums was absorbed by a layer of Sphagsorb applied to the top of the grouted monolith. Sphagsorb is a form of peat moss used commercially to absorb liquids. The drums were resealed and placed into 85-gallon overpacks for subsequent shipping.

**Table 7. Pilot-scale stabilization test data for GTS Duratek demonstration**

Drum number	Pounds						
	Waste	Cement	Water	Sodium metasilicate	Initial total	Cured total	Burial weight <sup>a</sup>
A8 004651	167	444	175	14	800	788	844
A8 004652	144	385	152	12	693	682	746
A8 004653	142	377	149	12	680	668	724
A8 004654	131	349	137	11	628	618	672
A8 004656	142	376	148	12	678	666	726
A8 004657	105	328	129	10	572	480	540
A8 004658	96	299	118	9	522	486	548
A8 004659	92	287	113	9	501	462	522
<b>Total</b>	<b>1,019</b>	<b>2,845</b>	<b>1,121</b>	<b>89</b>	<b>5,074</b>	<b>4,850</b>	<b>5,322</b>

<sup>a</sup> Includes weight of 55 gallon inner packs A (i.e., drums of waste) but not 72 lb per overpack.



## Phase IV-Disposal

After all drums of material were grouted using the low waste loading formula, samples of grouted product were submitted to Barringer Laboratories, Inc., a Utah certified laboratory, for complete analysis as required by Envirocare procedures. The applicable limits for each parameter are shown in Tables 8 and 9 for selected constituents.

The GTSD stabilization process successfully stabilized mercury and other inorganic contaminants shown below to disposal standards at Envirocare:

- Mercury
  - Reactive sulfides
  - Antimony
  - Arsenic
  - Barium
  - Beryllium
  - Boron
  - Bromide
  - Cadmium
  - Calcium
  - Chloride
- Chromium
  - Cobalt
  - Copper
  - Total reactive cyanide
  - Amenable reactive cyanide
  - Iron
  - Fluoride
  - Lead
  - Magnesium
  - Manganese
  - Nickel
- Potassium
  - Phosphorous
  - Selenium
  - Silver
  - Sodium
  - Strontium
  - Sulfur, Total
  - Thallium
  - Vanadium
  - Zinc

Although the mercury UTS was met, the disposal portion of the project was not completed because the grouted materials failed to meet other Envirocare WAC with respect to Phase III RCRA LDR requirements (see Tables 8 and 9).

**Table 8. Comparison of isotopic analysis of grouted samples from GTS Duratek demonstration with Envirocare disposal limits<sup>a</sup>**

Isotope	Low loading	High loading	Envirocare Disposal limit
	Units = picoCi/g		
<sup>228</sup> Actinium	15	32	
<sup>241</sup> Americium	329	644	820
<sup>212</sup> Bismuth	14	29	-
<sup>137</sup> Cesium	36	68	67
<sup>60</sup> Cobalt	152	287	347
<sup>154</sup> Europium	2.0	<1.0	-
<sup>212</sup> Lead	17	31	-
<sup>238</sup> Plutonium	12	43	380
<sup>239/240</sup> Plutonium	1,060	1,920	24,927
<sup>89/90</sup> Strontium	14,500	30,200	138,267
<sup>208</sup> Thallium	4.7	8.3	-
<sup>234</sup> Thorium	80	149	-
<sup>3</sup> Tritium	45	87	677
<sup>233/234</sup> Uranium	47	86	427
<sup>235</sup> Uranium	2.0	5.0	14
<sup>238</sup> Uranium	73	141	658

<sup>a</sup> This is a partial listing of constituents analyzed. Those that were not detected in any sample or not analyzed for are not shown. A dash entry indicates that a particular analysis was not done.



**Table 9. Comparison of analysis of grouted samples with Envirocare disposal limits for problematic organic compounds<sup>a</sup>**

Volatile or other organic compound	Low loading	High loading	Envirocare Disposal limit
	Units = mg/kg, except as noted		
<b>Volatile organic compounds</b>			
1,1,1-Trichloroethane; methylchloroform	45	1,500	6
4-Methyl-2-pentanone; methylisobutylketone	<50	<50	33
Benzo (a) pyrene	<0.66	<0.66	0.066
Dichloromethane; methylene chloride	4.3	59	30
Dichlorodifluoromethane	<10	<10	7.2
<b>Base/neutral organic compounds</b>			
Vinyl chloride; chloroethene	<10	<10	6 0.2 mg/L TCLP
<b>Pesticides and herbicides</b>			
2,4,-DB	<0.18	<0.18	0.13
Dalapon	<1.2	<1.2	0.066
MCPP	<39	<39	0.066
MCPA	<50	<50	0.066
Dichloroprop	<0.13	<0.13	0.066
Gamma-BHC; Lindane	0.63	1.2	0.066 0.4 TCLP
4,4'-DDE	0.34	0.58	0.066
Endrin Aldehyde	<3.3	<3.3	0.066
Heptachlor Epoxide	0.2	0.4	0.066 0.008 TCLP
PCB-1221	<0.067	<0.067	0.066
Methoxychlor	0.33	0.71	0.18 10.0 TCLP

<sup>a</sup> This is a partial listing of constituents analyzed. Of the constituents analyzed, only problematic ones are shown.

## Demonstration Results

### Compliance of Grouted Materials with LANL Requirements

GTSD used guidelines for evaluation of compliance with LANL contract requirements as follows:

- Use of low waste mass to final grouted mass loading.
- Application of the Envirocare WAC guideline with respect to radioactivity.
- Compliance with RCRA Phase IV LDR guidelines as per the 26 May 1998 Federal Register.
- While the proposal restricts evaluation of compliance of organics with the above regulations to those organics previously identified in the LANL raw waste analysis, acceptance by Envirocare is contingent upon compliance of all items referenced in the Envirocare WAC.

After treatment with the GTSD stabilization process, the following items were out of compliance with the Envirocare WAC and/or the RCRA Phase IV LDR guidelines:

- <sup>89/90</sup>Strontium
- <sup>241</sup>Americium
- 1,1,1-Trichloroethane
- 1,1-Dichloroethane
- Methylene Chloride
- Lindane, (gamma BHC)
- 4,4'-DDE



- Heptachlor Epoxide
- Methoxychlor

Demonstration test performance results are summarized in Tables 10 and 11, where they are evaluated against LANL and ORNL performance goals, respectively.

**Table 10. Summary results at low loading for evaluation of compliance GTSD stabilization demonstration products with Los Alamos National Laboratory goals<sup>a</sup>**

Parameter	Units	Compo- site sample	Sample A	Sample B	Phase IV LDR limit	Phase IV LDR compliance	Envirocare Acceptance criteria	Envirocare compliance?
<sup>228</sup> Actinium	pCi/gm	15	-	-	N/A	N/A	Not listed (not accepted)	<sup>239/240</sup> Pu daughter Yes
<sup>241</sup> Americium	pCi/gm	329	378	72	N/A	N/A	230	NO
<sup>212</sup> Bismuth	pCi/gm	14	-	-	N/A	N/A	Not listed (not accepted)	<sup>239/240</sup> Pu daughter Yes
<sup>137</sup> Cesium	pCi/gm	36	36	7	N/A	N/A	560	YES
<sup>60</sup> Cobalt	pCi/gm	152	214	14	N/A	N/A	360	YES
<sup>154</sup> Europium	pCi/gm	2	-	-	N/A	N/A	1,400	YES
<sup>212</sup> Lead	pCi/gm	17	-	-	N/A	N/A	Not listed (not accepted)	<sup>239/240</sup> Pu daughter Yes
<sup>238</sup> Plutonium	pCi/gm	12	<72	14	N/A	N/A	10,000	YES
<sup>239/240</sup> Plutonium	pCi/gm	1,060	2,990	672	N/A	N/A	9,950	YES
<sup>89/90</sup> Strontium	pCi/gm	14,500	27,200	1,910	N/A	N/A	11,000	NO
<sup>208</sup> Thallium	pCi/gm	4.7	-	-	N/A	N/A	Not listed (not accepted)	<sup>239/240</sup> Pu daughter Yes
<sup>234</sup> Thorium	pCi/gm	50	-	-	N/A	N/A	Not listed (not accepted)	<sup>238</sup> U daughter Yes
<sup>3</sup> Tritium	pCi/gm	45	-	-	N/A	N/A	Not listed (not accepted)	N/a
<sup>233/234</sup> Uranium	pCi/gm	47	57	27	N/A	N/A	37,000	Yes
<sup>235</sup> Uranium	pCi/gm	2	<2.9	2.6	N/A	N/A	770	Yes
<sup>238</sup> Uranium	pCi/gm	73	83	34	N/A	N/A	28,000	Yes
1,1,1- Trichloroethane	mg/kg	1,500	-	-	6	No	References EPA	No
1,1-Dichloroethane	mg/kg	25	-	-	6	No	References EPA	No
Methylene Chloride	mg/kg	59	-	-	30	No	References EPA	No
bis (2- Ethylhexyl) phthalate	mg/kg	2	-	-	28	Yes	References EPA	Yes
Mercury	mg/L	0.0114	0.0169	0.00127	0.25	Yes	References EPA	Yes
Barium	mg/L	2.8	2.2	1.3	7.6	Yes	References EPA	Yes
Chromium	mg/L	<0.08	0.09	0.07	0.86	Yes	References EPA	Yes
Lead	mg/L	<0.037	<0.07	0.28	0.37	Yes	References EPA	Yes
Silver	mg/L	<0.03	0.08	0.03	0.3	Yes	References EPA	Yes
Lindane, (gamma BHC)	mg/kg	0.63	-	-	0.066	No	Not accepted	No
4,4'-DDE	mg/kg	0.34	-	-	0.087	No	Not accepted	No
Heptachlor epoxide	mg/kg	0.2	-	-	0.066	No	Not accepted	No
Methoxychlor	mg/kg	0.33	-	-	0.18	No	Not accepted	No



## Compliance of Grouted Materials with ORNL Requirements

GTSD used the guidelines below for evaluation of compliance with ORNL contract requirements:

- use of high waste mass to final grouted mass loading,
- compliance with RCRA Phase IV LDR guidelines as per the 26 May 1998 Federal Register,
- compliance of organics with the above regulations based on organic compounds previously identified in the LANL waste analysis.

With the exception of one cell in Table 11, all mercury data-points shown in Table 11 comply with the 0.025 mg/L TCLP guideline. Other TCLP metals were in compliance with the new TCLP guidelines.

The following parameters were out of compliance with the Envirocare WAC and/or the RCRA Phase IV LDR limits:

- <sup>89/90</sup>Strontium
- <sup>241</sup>Americium
- 1,1,1-Trichloroethane
- Lindane, (gamma BHC)
- 4,4'-DDE
- Heptachlor Epoxide
- Methoxychlor.

## Final Disposition of Material

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Because of the noncompliance of specific parameters as shown in Tables 10 and 11 and the expiration of the 1-year limitation for retention of hazardous wastes in a treatability study, the material was returned to LANL under a Bill of Lading. A total of eight drums of material were returned having been prepared at low waste-to-final-grouted-mass loading.



**Table 11. Summary results at high loading for evaluation of compliance GTSD stabilization demonstration products with Oak Ridge National Laboratory goals<sup>a</sup>**

Parameter	Units	Compo-site sample	Sample A	Sample B	Phase IV LDR limit	Phase IV LDR compliance	Envirocare acceptance criteria	Envirocare Compliance?
<sup>228</sup> Actinium	pCi/gm	32	-	-	N/A	N/A	Not listed (not accepted)	Yes <sup>239/240</sup> Pu daughter
<sup>241</sup> Americium	pCi/gm	644	1,243	281	N/A	N/A	230	No
<sup>212</sup> Bismuth	pCi/gm	29	-	-	N/A	N/A	Not listed (not accepted)	Yes <sup>239/240</sup> Pu daughter
<sup>137</sup> Cesium	pCi/gm	68	108	24	N/A	N/A	560	Yes
<sup>60</sup> Cobalt	pCi/gm	287	673	46	N/A	N/A	360	Yes
<sup>154</sup> Europium	pCi/gm	<1	-	-	N/A	N/A	1,400	Yes
<sup>212</sup> Lead	pCi/gm	31	-	-	N/A	N/A	Not listed (not accepted)	Yes <sup>239/240</sup> Pu daughter
<sup>238</sup> Plutonium	pCi/gm	43	<57.2	34	N/A	N/A	10,000	Yes
<sup>239/240</sup> Plutonium	pCi/gm	1,920	10,200	1,440	N/A	N/A	9,950	Yes
<sup>89/90</sup> Strontium	pCi/gm	30,200	49,200	4,070	N/A	N/A	11,000	No
<sup>208</sup> Thallium	pCi/gm	8.3	-	-	N/A	N/A	Not listed (not accepted)	Yes <sup>239/240</sup> Pu daughter
<sup>234</sup> Thorium	pCi/gm	149	-	-	N/A	N/A	Not listed (not accepted)	Yes <sup>238</sup> U daughter
<sup>3</sup> Tritium	pCi/gm	87	-	-	N/A	N/A	Not listed (not accepted)	N/A
<sup>233/234</sup> Uranium	pCi/gm	86	206	69	N/A	N/A	37,000	Yes
<sup>235</sup> Uranium	pCi/gm	5	14.1	4.9	N/A	N/A	770	Yes
<sup>238</sup> Uranium	pCi/gm	141	296	79	N/A	N/A	28,000	Yes
1,1,1-Trichloroethane	mg/kg	45	-	-	6	No	References EPA	No
1,1-Dichloroethane	mg/kg	<5	-	-	6	Yes	References EPA	Yes
Methylene Chloride	mg/kg	4.3	-	-	30	Yes	References EPA	Yes
bis (2-Ethylhexyl) Phthalate	mg/kg	2.9	-	-	28	Yes	References EPA	Yes
Mercury	mg/L	0.0233	0.0314	0.00249	0.025	No	References EPA	Yes
Barium	mg/L	3.4	1.4	0.85	21	Yes	References EPA	Yes
Chromium	mg/L	0.09	0.24	0.12	0.60	Yes	References EPA	Yes
Lead	mg/L	<0.037	0.24	0.30	0.75	Yes	References EPA	Yes
Silver	mg/L	<0.03	<0.02	0.06	0.14	Yes	References EPA	Yes
Lindane, (gamma BHC)	mg/kg	1.2	-	-	0.066	No	References EPA	No
4,4'-DDE	mg/kg	0.58	-	-	0.087	No	References EPA	No
Heptachlor epoxide	mg/kg	0.4	-	-	0.066	No	References EPA	No
Methoxychlor	mg/kg	0.71	-	-	0.18	No	References EPA	No



## SECTION 4

# TECHNOLOGY APPLICABILITY AND ALTERNATIVES

## Competing Technologies

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### Baseline Technologies

While RMERC/IMERC has been used to address mercury within waste at concentrations exceeding 260 ppm, there is relatively little work on the effective stabilization of mercury in problematic mercury contaminated mixed wastes at <260 ppm. This need has driven the mission of the HgWG to identify and validate useful industry mercury stabilization technology for DOE mixed wastes. Although RMERC could be applied to mercury in the <260 ppm range, there are both public and regulatory concerns about mercury emissions. In addition, RMERC alone does not address the potential need to stabilize thermal residuals before disposal in a licensed and permitted landfill. Hence, no real baseline technology exists for mercury-contaminated mixed wastes at <260 ppm, although several promising new and developing technologies do exist.

Sulfur polymer cement offers some potential for mercury stabilization. However, this process is sensitive to water content of the subject material and requires elevated temperature for application. For the specific matrix tested in this work, it is unlikely that sulfur polymer cement would be as useful as other competing technologies, such as the GTSD stabilization process, given the high water content and the relatively low decomposition temperature of anion-exchange material.

The MER02 Statement of Work (SOW) specified technology demonstrations to address the deficiencies in technology for treating mercury-contaminated mixed waste. Under the MER02 SOW, two vendors were funded to demonstrate their newly-developed/developing processes on actual mixed wastes. In addition, LANL issued a contract to GTS Duratek to perform a treatability study on other waste. The MWFA added to that contract to have Duratek provide similar information to that being collected in the MER02 demonstrations. The processes demonstrated by ATG, Duratek, and NFS represent competing technologies. As newly demonstrated technologies, comparisons of these processes, among themselves and with respect to the baseline, are of particular interest. Important criteria for comparison include: (1) performance in treating mercury-contaminated mixed waste (<260 ppm), (2) applicability to the target and other waste categories, (3) cost, and (4) risk to workers and the public.

Currently, however, the GTSD demonstration findings are inconclusive, with respect to performance and cost, for example. Hence, a full comparison of competing technologies is not possible at this time. Nevertheless, important criteria for comparison are reported here for the GTSD process, as available, along with similar information for ATG and NFS.

### Technology Applicability

The stabilization system demonstrated by GTSD shows potential for treating many of the low-level streams listed in Table 1 of the MWFA TDRD (DOE 1996), including aqueous streams designated for the Toxic Substances Control Act (TSCA) incinerator and the transportable vitrification system, provided that levels of certain types of constituents are not too high. This study has clearly demonstrated the adverse impact that high levels of radionuclides and organic compounds can have on the performance of a stabilization technology. In the absence of such impediments, or in combination with an appropriate pretreatment technology such as thermal desorption, the GTSD stabilization technology could be appropriate.

The in-drum processing capability enhances the applicability of the technology. Of course, the hundreds of cubic meters of TRU wastes would require a larger facility than tested here and the heterogeneous debris wastes would also require equipment for size reduction.



## Other Competing Technologies

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Table 12 summarizes technology features and demonstration results for the three vendors participating in the MER02 mixed waste stabilization demonstrations.

### ATG Stabilization Technology

ATG has demonstrated a full-scale stabilization system that uses a dithiocarbamate (DTC) formulation to produce a stabilized waste, which satisfies the UTS treatment limits for mercury. The DTC formulations used by ATG reproducibly stabilized over 99% of the mercury initially present at levels of about 40 times the UTS in an ion exchange process stream from a Portsmouth, Ohio, facility. Volume increases were reported to be small, at 16% of the untreated waste volume. The process demonstrated some robustness to mercury at a bench scale stabilizing several different species of mercury, and at a pilot scale, stabilizing additional constituents, barium, cadmium, and chromium.

### NFS Stabilization Technology

NFS developed a process to address a large variety of chemical forms of mercury. The process has been successfully applied to and is useful for a wide selection of waste matrices including shreddable debris, nonshreddable debris, wastewaters, soils, sludges, and organic compounds.

The NFS process was used to successfully demonstrate treatment of elemental mercury waste in a MWFA-sponsored demonstration (MWFA 1998b) and was also applied to demonstrate the treatment of surrogate mixed waste containing mercury in the form of various chemical species (Osborne-Lee 1999). The mercury species tested include elemental mercury, mercuric chloride, mercuric iodide, phenylmercuric chloride, mercuric oxide, mercuric cyanide, and mercuric thiocyanate.

Mixed wastes with elemental mercury will require the addition of amalgamation, as well as stabilizing reagents to address the various chemical species of mercury. Soils and sludges containing elemental mercury represent a particular challenge. This is ascribed to potential matrix-mercury interaction effects, which render stabilization less effective. In addition, the elemental mercury contamination within these matrices is unevenly distributed. Therefore, mixing methods become important in dispersing amalgamating/stabilizing reagents to all sites within the matrix that contain the mercury contamination.

Potential commercial applications of the NFS process also exist. Certain select matrices, such as sludges, soils, and adsorbents, generated by commercial entities are similar to those within DOE, except without the radiological components.



**Table 12. Summary of findings from tests by GTS Duratek, Allied Technology Group, and Nuclear Fuel Services on mixed waste stabilization (<260 ppm Hg)**

Comparison factor	GTS Duratek, Inc.	Nuclear Fuel Services, Inc. (NFS)	Allied Technology Group, Inc. (ATG)
Waste type tested	Sludge and laboratory residues from Los Alamos National Laboratory	Ion-exchange resin from Portsmouth, Ohio facility	Ion-exchange resin from Portsmouth, Ohio facility
Process mechanism	In-drum mixer based operation	Standard laboratory glassware operation based in a ventilation hood	Pug mill, mortar mixer, hazardous material enclosure, with ventilated hood and air treatment system
Scale of bench test	0.25–0.35-kg bench-scale tests performed	1-kg scoping tests performed	0.6-kg bench-scale tests performed
Scale of demonstration	55-gallon drum (about 200–400 kg)	14 kg batches	33 kg batches (full-scale)
Final waste form	Passes TCLP for Hg and other inorganics; fails TCLP for organics and pesticides; fails WAC for radionuclides	Passes all TCLP and UTS tests (Hg, Cd, Cu, Zn, Ni, and Cr), but modified formulation required for Cr	Most effective for mercury and Cr, moderately effective for Ba and Cd
Stabilization process	Portland cement based grout	Uses proprietary formulation of additives and EPA-prescribed agents	Uses dithiocarbamate, or other nonproprietary agents, and a small amount of proprietary liquid
Effect of contaminants on the process	High levels of organic compounds or radionuclides make effective waste form difficult to achieve	Fines from resin created filtration challenge, solved by use of filter aids	Water <10% tolerated. Other contamination not addressed
Throughput	Not determined <sup>a</sup>	1,000 lb/hr, per SOW	1,200 lb/hr at full scale
Cost	Not determined <sup>a</sup>	~\$6/kg at 1,000 lb/hr to \$37/kg at 100 lb/hr.	<\$2 kg at 1,200 lb/hr
Waste Acceptance Criteria	Product did not meet WAC for radionuclides, pesticides, or volatile organic compounds	Both processes produce waste forms that meet current Envirocare WAC	
Moisture	Moisture (water in small amounts) is tolerated by all three processes		
Physical characteristics	Waste form characteristics are physically similar		
Regulatory and safety requirements	No additional hazards, safety, or regulatory issues found for either process		
Summary assessment	Potentially an effective stabilization process; but testing with an appropriate waste stream or pretreatment is needed	Effective stabilization process; less costly, better leach performance for more UHCs, but formulations are proprietary	Effective stabilization process; achieves higher waste loadings, less secondary waste, and fewer proprietary reagents

<sup>a</sup> Waste specimens tested by ATG and NFS are very comparable. The GTSD specimens were very different, thus the basis for comparison of three vendor technologies is difficult to achieve.

<sup>b</sup> Universal Treatment Standard (UTS). Underlying Hazardous Constituents (UHCs). Waste Acceptance Criteria (WAC).



## SECTION 5

### COST

#### **Cost Methodology**

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A treatment system capable of producing a compliant waste form for disposal was not achieved. Therefore, final system specifications could not be made. Thus, a basis for cost and production capacity estimates does not currently exist for the GTSD technology at this time.

#### **Cost Conclusions**

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No cost conclusions may be drawn without further demonstration of the technology.



## SECTION 6

# REGULATORY AND POLICY ISSUES

### Regulatory Considerations

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No federal permitting may be required for the GTSD transportable mercury stabilization system. Federal regulations allow onsite treatment of hazardous waste without a permit under certain conditions. Processing of lead-contaminated soils in a pug mill satisfied these conditions (EPA 1994) as should processing of mercury-contaminated mixed waste in an in-drum facility.

The regulatory/permitting issues related to the use of stabilization technology for treatment of mercury contaminated wastes, are governed by the following safety and health regulations:

- Occupational Safety and Health Administration (OSHA), 29 CFR 1926
  - 1926.28 Personal Protective Equipment
  - 1926.102 Eye and Face Protection
  - 1926.103 Respiratory Protection
- OSHA 29 CFR 1910
  - 1910.132 General Requirements (Personnel Protective Equipment)
  - 1910.133 Eye and Face Protection
  - 1910.134 Respiratory Protection

Disposal requirements/criteria include the following DOT and DOE requirements:

- 49 CFR, Subchapter C, Hazardous Materials Regulation
  - 171 General Information, Regulations, and Definitions
  - 172 Hazardous Materials Table, Special Provisions, Hazardous Materials Communications, Emergency Response Information, and Training Requirements
  - 173 Shippers—General Requirements for Shipments and Packagings
  - 174 Carriage by Rail
  - 177 Carriage by Public Highway
  - 178 Specifications for Packaging
- 10 CFR 71 Packaging and Transportation of Radioactive Material

If the waste is determined to be hazardous solid waste, the following EPA requirement should be considered:

- 40 CFR, Subchapter 1 Solid Waste

### CERCLA Criteria

The Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) has established nine criteria against which alternative treatment approaches are to be judged during the Remedial Investigation/Feasibility Study (RI/ FS) portion of the remediation action. A short explanation of each of the criteria (EPA 1988) and the assessment of GTSD's process against it follows.

#### Overall protection of human health and the environment

This criterion is an evaluation of the overall protectiveness of an alternative and should focus on whether a specific alternative achieves adequate protection and should describe how site risks posed through each pathway being addressed by the FS are eliminated, reduced, or controlled.

In a CERCLA environment, the resulting waste forms from the GTSD process, once proven successful, will provide improved protection of human health and the environment by reducing the mobility of the



elemental mercury. The final waste forms may need to be placed inside another container to further enhance protection.

#### Compliance with ARARs

This evaluation criteria is used to determine whether each alternative will meet all of its federal and state Applicable or Relevant and Appropriate Requirements (ARARs) that have been identified in previous stages of the RI/ FS process.

The LDRs are the most likely ARAR to be applied to a CERCLA site dealing with mercury wastes. Regulations under RCRA specify no standard treatment for elemental mercury. The GTSD stabilization process, or a similar competing process, could provide a potential future best practice for this waste type.

#### Long-term effectiveness and permanence

Alternatives under this criterion are to be evaluated in terms of risk remaining at the site after response objectives have been met. The primary focus of this evaluation is the extent and effectiveness of the controls that may be required to manage the risk posed by treatment residuals and/ or untreated wastes.

The long-term effectiveness of any remediation process has to be judged not only by the efficacy of the actual treatment process, but also by how well the process can be applied to the extent of the contamination. Assuming that the mercury waste can be efficiently brought to GTSD's equipment, the process should be able to provide environmental protectiveness. Tests to be performed at ORNL will provide a more definitive answer.

#### Reduction of toxicity, mobility, or volume through treatment

The statutory preference for selecting remedial actions that employ treatment technologies that permanently and significantly reduce toxicity, mobility, or volume of the hazardous substances are to be evaluated under this criterion.

Stabilization with the GTSD process, or a similar process, should significantly reduce mercury's mobility in a waste management scenario. In a CERCLA action, further study would be required to assess how the action of bacteria affects the waste form. Secondary containment may be prudent in any case.

#### Short-term effectiveness

This criterion addresses the effects of the alternative during the construction and implementation phase until remedial response objectives are met.

Once an effective process is proven, designed, and ready to be operated, the GTSD process should be protective of the community and the workers while not imposing meaningful environmental consequences during its operation.

#### Implementability

The implementability criterion focuses on the technical and administrative feasibility of implementing an alternative and the availability of various services and materials required during implementation.

The process should prove to be viable from the standpoints of both the technical (ability to construct, reliability, and monitoring) and administrative (coordination with other agencies) feasibility, as well as the availability of services and materials.

#### Cost

The costing procedures found in the *Remedial Action Costing Procedures Manual* are to be the bases for comparing alternatives with regard to costs.

The cost figures to be provided in the future were not based on the rigor detailed in the referenced document above.

#### State acceptance

This assessment evaluates the technical and administrative issues and concerns the state (or support agency in the case of state-lead sites) may have regarding each of the alternatives.

See "Safety, Risks, Benefits, and Community Reaction" subsection below.



### Community Acceptance

Under this criterion, an assessment is made on the issues and concerns the public may have regarding each of the alternatives.

See "Safety, Risks, Benefits, and Community Reaction" subsection below.

## **Safety, Risks, Benefits, and Community Reaction**

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The HgWG, considering eight criteria for the level of risk as associated with mercury stabilization, evaluated other aspects of risk, as follows:

- correctness (technical correctness),
- cost (effectiveness to use),
- permitability (ease of permitting),
- safety,
- sponsorship (commitment by sponsors),
- completeness (ready for use),
- acceptability (to stakeholders),
- timeliness (to meet schedules).

The risk values, established for the MWFA developed technology processes, have been derived from top-level requirements defined in the MWFA Systems Requirements Document. Evaluations of the technology and assignment of risk values were made by a team comprised of HgWG members, in consideration of the risk category definitions and performance observations from the demonstration experience. The assessments made are summarized below.

### **Correctness**

This risk category is moderate. The targeted volume of waste to be treated is not large compared with most other waste types. Successes with inorganic compounds, including mercury are promising. Prior successes with low-level radioactive wastes, bench-scale tests with mixed wastes, and now largely promising demonstration results indicate that stabilization is a reasonable option for many mercury contaminated mixed waste streams. Limitations to stabilization as a stand-alone technology for mercury waste treatment may be overcome by using it in combination with supporting treatment steps. The as yet unproven status of the GTSD process increases this risk parameter somewhat.

### **Cost**

This risk category is rated as moderate. The targeted volume to be treated is not large, but the waste possesses diverse characteristics. Oxidation, complexation, and speciation of mercury across various matrices add an element of uncertainty as to the difficulty of successfully stabilizing the bulk of inventory (and future generation) without process modifications. Although no cost estimates are available for this specific technology, cost estimates for similar competing technologies (such as those demonstrated by ATG and NFS) show that stream characteristics greatly influence unit cost.

### **Permitability**

This risk category is rated as very low. The treatment process is simple and based on a well-proven Best Available Demonstrated Technology for nonradioactive mercury waste. The volumes of waste involved are not large enough to pose much likelihood of regulatory problems.

### **Safety**

This risk category is rated as low. While mercury is a hazardous material of some concern and radioactive contamination has the potential to raise additional concern, mercury vapors and leaching appear to be well-controlled by the process and radioactive contamination is low. The stability of the final waste form is key in immobilizing both mercury and radionuclides, thereby minimizing concerns over worker safety, public safety, and environmental protection (Connor 1990).



## **Sponsorship**

This risk category is rated as moderately low. Interest by the sites has been good, and programmatic support for technology development has demonstrated good commitment. There is a small risk that some potential users may find a local or onsite solution for treatment of their mercury wastes.

## **Completeness**

This risk category is rated as moderately low due to the simple, proven nature of stabilization, in consideration of the potential complexity of mercury chemistry and diversity of waste matrices, especially in light of recent, largely successful demonstrations.

## **Acceptability**

This risk category is rated as very low. Stabilization is a process easily identifiable to the public because of the widespread use of cement and concrete. The waste form stability, simplicity, and familiarity to the public that characterize the technology are expected to make for easy public acceptance.

## **Timeliness**

This risk category is rated low. Based on preliminary information received to date from 10 DOE sites, the timeframe for treatment is late FY-99 and FY-00.

## **Public Participation**

The siting of a mixed waste treatment facility of any kind near communities will involve public input. Stakeholders are generally concerned about the type, toxicity, and amount of emissions to be discharged to the atmosphere and the disposal site for the final waste form.

The MWFA Tribal and Public Involvement Resource Team and HgWG initiated activities to involve and gather stakeholder issues, needs, and concerns about mercury treatment technologies. These activities included reviews, articles, and presentations. During November and December of 1997, the chair of the HgWG addressed both the Oak Ridge Local Oversight Committee and the Site Specific Advisory Board (SSAB). The purpose of the November 1997 meetings was to identify issues, needs, and concerns of various Oak Ridge stakeholders regarding technologies that may be applicable to Oak Ridge. The areas emphasized included continuous emission monitors, characterization, input to Technology Performance Reports, and the HgWG. These meetings were interactive, where participants explored the issues and problem-solved collectively. No formal presentations were made, but information was provided and progress on various MWFA projects was discussed. Participants included members of the local oversight committee, the Site Technology Coordination Group (STCG), and the general public.

The SSAB Environmental Technology Group meeting on December 10, 1997 involved providing stakeholder input into various technologies development projects at Oak Ridge. Those they have expressed interest in addressing are:

1. Transportable Vitrification System
2. TSCA Test Bed for Continuous Emissions Monitors
3. Mercury Working Group/Mercury Treatment Demonstrations
4. Removal of Mercury from Liquid Wastes.

A short presentation on the status of each activity was given and the proposed future scopes were discussed.

The MWFA assembled a Technical Requirements Working Group (TRWG); a stakeholder group capable of representing varied Tribal and public perspectives. The TRWG assisted MWFA technical staff in transforming or integrating site-specific issues, needs, and concerns into the TDRDs, and providing Tribal and public perspectives to technical staff for identifying and resolving technical issues. The TRWG reviewed and provided recommendations to the MWFA on changes to the Mercury TDRDs.

Lastly, the MWFA Resource Team facilitated Tribal and public involvement by issuing an article in the quarterly, July 1997, newsletter highlighting mercury treatment and disposal.



## SECTION 7

# LESSONS LEARNED

### Implementation Considerations

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Key lessons learned during this project were as follows:

- The GTSD process is effective in stabilizing inorganic constituents in the sludge and laboratory residue wastes studied.
- Volatile organic compounds, pesticides, herbicides, and radionuclides, if present at high levels, make stabilization of mixed wastes a difficult challenge. For the organic compounds, pretreatment such as might be achieved with thermal desorption will likely be needed. For radionuclides, reformulation of the grout recipe, possibly requiring the use of additional stabilizing agents, will likely be needed.
- This demonstration has served to reemphasize the importance of accurate characterization data for waste streams and serves as a reminder that existing data on currently-existing and, possible, future waste is subject to significant uncertainty.



## APPENDIX A

### REFERENCES

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## APPENDIX B

### ACRONYMS AND ABBREVIATIONS

ADA	ADA Technologies (Englewood, Colorado)
ARARs	Applicable or Relevant and Appropriate Requirements
ATG	Allied Technologies Group, Inc. (Fremont, California)
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
DeHg	A proprietary process by NFS for processing mercury mixed waste (pronounced de <sup>-</sup> merk)
DOE	Department of Energy
DOT	Department of Transportation
DTC	Dithiocarbamate, a reagent in a formula for stabilization
EPA	Environmental Protection Agency
GTSD	GTS Duratek, Inc., (Oak Ridge, Tennessee)
HgWG	Mercury Working Group, MWFA
INEEL	Idaho National Engineering and Environmental Laboratory
IT	International Technologies, Inc.
ITSR	Innovative Technology Summary Report
LANL	Los Alamos National Laboratory
LDR	Land Disposal Restrictions
MER01	A solicitation to industry (November 1996) entitled, "Demonstration of the Amalgamation Process for Treatment of Radioactively Contaminated Elemental Mercury Wastes"
MER02	A solicitation to industry (September 1997???) entitled, "Demonstration of the Stabilization Process for Treatment of Radioactively Contaminated Mercury (<260 ppm) Wastes"
MWFA	Mixed Waste Focus Area
NFS	Nuclear Fuel Services, Incorporated (Erwin, Tennessee)
ORNL	Oak Ridge National Laboratory
OSHA	Occupational Safety and Health Administration
PAC	powder activated carbon
PORTS	Piketon Gaseous Diffusion Facility
ppm	parts per million
PSP	Phase Separator Pit
RCRA	Resource Conservation and Recovery Act
RFP	Request for Proposal
RI/FS	Remedial Investigation/ Feasibility Study
SOW	Statement of Work
SSAB	Site Specific Advisory Board
STCG	Site Technology Coordination Group
TCLP	Toxicity Characteristic Leaching Procedure
TDRD	Technology Development Requirements Document
TLV	Threshold Limit Value
TRU	Transuranic
TRWG	Technical Requirements Working Group
TSCA	Toxic Substances Control Act
UHC	Underlying Hazardous Constituent
UTS	Universal Treatment Standard
WAC	Waste Acceptance Criteria



