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Informal Report

# MATERIALS TESTING FOR IN SITU STABILIZATION TREATABILITY STUDY OF INEEL MIXED WASTE SOILS

John Heiser and Mark Fuhrmann

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ENVIRONMENTAL & WASTE TECHNOLOGY CENTER
DEPARTMENT OF ADVANCED TECHNOLOGY
BROOKHAVEN NATIONAL LABORATORY, ASSOCIATED UNIVERSITIES, INC.
P.O. BOX 5000, UPTON, NEW YORK 11973-5000

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# Materials Testing for In Situ Stabilization Treatability Study of INEEL Mixed Waste Soils

September 1997

Prepared by:

John Heiser
Mark Fuhrmann
Environmental & Waste Technology Center
Brookhaven National Laboratory
Upton, New York





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# DISCLAIMER

## 1.0 INTRODUCTION

This report describes the contaminant-specific materials testing phase of the In Situ Stabilization Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) Treatability Study (TS). The purpose of materials testing is to measure the effectiveness of grouting agents to stabilize Idaho National Engineering and Environmental Laboratory (INEEL) Acid Pit soils and select a grout material for use in the Cold Test Demonstration and Acid Pit Stabilization Treatability Study within the Subsurface Disposal Area (SDA) at the Radioactive Waste Management Complex (RWMC). Test results will assist with selecting a grout material for the follow-on demonstrations described in *Test Plan for the Cold Test Demonstration and Acid Pit Stabilization Phases of the In Situ Stabilization Treatability Study at the Radioactive Waste Management Complex* (Galloway et. al. 1997).

## 2.0 SCOPE

The investigation-derived soil samples generated by the characterization study were classified as Resource Conservation and Recovery Act (RCRA) waste and were stored in drums at the INEEL MWSF, a RCRA treatment storage and disposal facility operated by the Waste Reduction Operations Complex (WROC). These soil samples were to be used for the laboratory mixing studies discussed in the project scope of work. The soil material selected for testing originated from Coreholes 11 and 12 from the 1991 Phase I borehole sampling for the Acid Pit Track 2 Characterization. Phase 1 sample analysis results indicated coreholes 11 and 12 had relatively high concentrations of mercury [>1000 parts per million (ppm)]. Table 1 lists Phase I analytical results from the soil samples selected for sample retrieval.

**Table 1. Acid Pit Soil Sample Results**

| Hole No. | Depth | Hg (mg/kg) | Metals (mg/kg) | Radionuclides (pCi/g) | Organics (ug/kg) | Nonmetal Inorganics (mg/kg) |
|---|---|---|---|---|---|---|
| 11 | 14-16 | 1420 | None reported | *Gamma emitting*<br>Co-60 1.56<br>Cs-137 1.14<br>Eu-152 0.523<br><br>*Alpha emitting*<br>Pu-239 0.20<br>U-234 2.9<br>U-238 0.11<br>U-235 3.0 | *Volatile*<br>Carbon tetrachloride 24<br>Chloroform 18<br>Trichloroethane 18<br>Methylene chloride 170<br>Acetone 103<br><br>*Semivolatile*<br>Bis(2-ethylhexyl)phthalate 5200<br>Tributylphosphate 45,000 | Nitrate 5590<br>Sulfate 10,600<br>TOC 11,400 |
| 12 | 14-16.8 | 1030 | Beryllium 2.5<br>Aluminum 27,800<br>Sodium 658 | *Gamma emitting*<br>None detected<br><br>*Alpha emitting*<br>U-234 3.0<br>U-238 2.5 | *Volatile*<br>Methylene chloride 160<br><br>*Semivolatile*<br>Bis(2-ethylhexyl)phthalate 1040<br>Tributylphosphate 1100 | Nitrate 7890<br>TOC 14,800<br>Magnesium 9073<br>Sodium 658 |

The specific Acid Pit soil samples were retrieved and shipped to BNL for bench-scale materials testing. Because the Acid Pit soil samples were stored as RCRA waste, the BNL bench-scale grout studies were conducted as a RCRA TS. The primary objectives of the materials testing RCRA TS was to conduct mixing studies with actual Acid Pit soils and candidate grout materials to select a suitable grout for the Cold Test and Acid Pit Stabilization Demonstrations. Parameters to be evaluated included contaminant stabilization, compressive strength, and implementability.

## 3.0 DESCRIPTION OF LABORATORY STUDIES

The technical support provided by BNL for the materials testing RCRA TS was divided into the following tasks:

Task I: Characterization Testing
Task II: Test Specimen Preparation
Task III: Performance Testing.

A 45-day RCRA treatability study notification was filed with the State of New York for performing the soil/grout tests at BNL. Samples were shipped to BNL before the 45-day approval to initiate characterization testing. Specimen preparation and performance testing did not commence until the 45-day notification had been approved.

### 3.1 Task I - Characterization Testing

The objective of Task I was to characterize the Acid Pit soils prior to performance testing. Before characterization testing, the soil was sized (3/8" sieve) and screened to produce a homogenous sample. A representative sample was collected from the composite mixture for characterization testing, including analytical testing to determine the total mercury concentration and soluble mercury concentration.

Five binding additives that could potentially enhance stabilization of the soluble mercury contamination are sodium sulfide, diatomaceous earth, zeolite, hematite, and fly ash.

#### 3.1.1 Initial Characterization of INEEL Soil and Addition of Hg

On receipt of the soil from INEEL, 5g aliquots of soil were taken for: pre-TCLP testing of pH. The samples were obtained from cans labeled: CH11 10-12' and CH 11 14-16'. Results indicated that TCLP Leachant #1 should be used. Three sample were tested using the TCLP:

1. CH 11 10-12'
2. CH 11 14-16'
3. CH 12 14-16'

The TCLP leachate was filtered through type GF/F glass Whatman filters. Analysis of the leachate for Hg was done by ICP using EPA Method 200.7 Rev. 4.4 (1994) "Methods for the Determination of Metals in Environmental Samples, Supplement I. Determination of Metals and Trace Elements in Water and Wastes by ICP-AES". Analysis was conducted at 194.163 nm on a Varian, Liberty 100 ICP (Figure 1). Standards, an analytical blank, a reagent blank, and a reagent spike were prepared. The detection limit was estimated to be 0.1 ppm. No Hg was observed in any of the TCLP leachates. This was in contradiction with information from INEEL that the soil contained approximately 1000 ppm Hg, which should give leachate concentrations of 50 ppm.

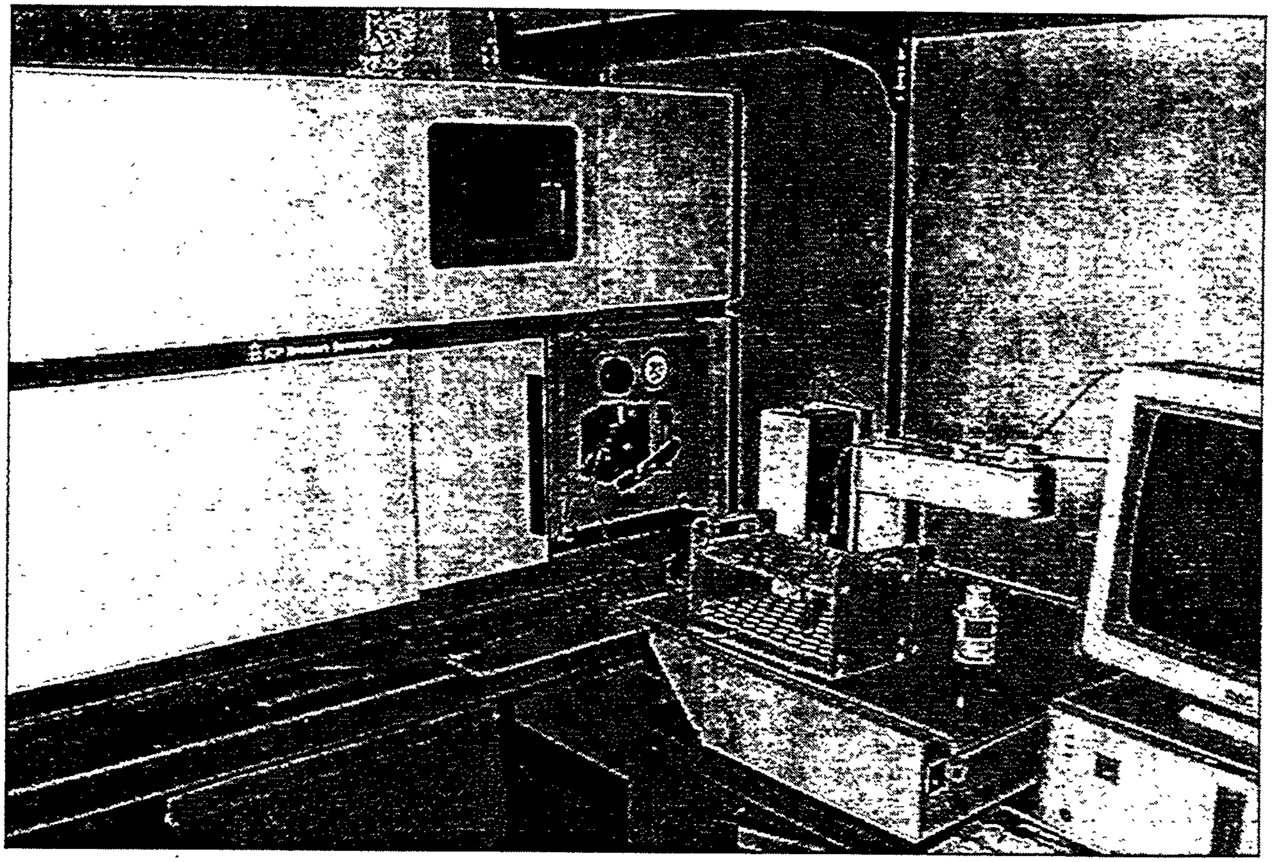

**Figure 1.** Varian, Liberty 100, Inductively Coupled Plasma Optical Emission Spectrometer.

To double check these results, digestions of samples of the INEEL soil were conducted according to SW 846. Aliquots of the soil, weighing about 2 g were digested in 15 mL of concentrated HCL and 5 mL of concentrated $HNO_3$. After reacting overnight the extract was filtered through Whatman # 41 filter paper and brought up to 50 mL with distilled/deionized water. Results are shown in Table 2.

**Table 2. Hg Content of INEEL Soil**

| Sample | Hg in Digest (ppm) | Dilution Factor | Hg in Soil (ppm) |
|---|---|---|---|
| CH11 10-12' | < 0.1 | 25.77 | <2.6 |
| CH12 14-16' | 0.41 | 25.51 | 10.5 |
| CH 11 14-16' | 0.47 | 21.01 | 9.9 |

Based on these results it was decided to spike the soil with Hg to obtain a concentration similar to that known to exist in other samples of the INEEL soil. To do this, all of the soil remaining in the can containing CH11 14-16' was weighed into a 5 gallon plastic pail. The soil weighed 3244 g. A solution, containing 4.38 g of $HgCl_2$ in one liter of distilled water, was mixed with the soil. This gave a very wet mixture which assured homogeneous mixing of the Hg. The material was left to air dry in a hood.

Later it was determined that more spiked soil would be required. An additional 653.8 g of soil from CH11 10-12' was added to the previously spiked soil, giving a total as received weight (of the spiked soil) of 3898 g. A solution consisting of 0.88 g of $HgCl_2$ in 200 mL of water was also added. After air drying for several days, with frequent mixing, the soil weight was 3448 g. The soil was restored to its original water content by adding distilled water.

To check if the Hg concentration of the soil was acceptable, three small samples of the spiked soil were digested as described above. Concentrations of Hg in the three samples of spiked soil were: 878, 1004 and 898 ppm, with an average of 927 ppm. This value, slightly lower than expected, may represent some loss of Hg to the vapor phase.

### 3.1.2 Additives

To minimize leaching of Hg from the solidified soil, it may be necessary to use an additive that is mixed in with the solidification agent. To determine the best additive to use, two series of tests were run in which the sorption of Hg by each of the additives was assessed. In one series, 1 g of each additive was weighed out and added to a bottle containing 40 g of a solution containing Hg. The pH of the solution had been adjusted to 7.8. In the second series, 0.5 g of additive was added to 9 g of solution. Results are shown in Table 3. Based on concentrations of Hg in the liquid after the tests, three materials outperformed the others: sodium sulfide, hematite and iron powder.

**Table 3. Sorption of Hg on Additives**

| Additive | Source | Wt (g) | Vol (mL) | Hg Start (µg/g) | Hg End (µg/g) | Mass of Hg Sorbed (µg/g) |
|---|---|---|---|---|---|---|
| Clinoptilolite | Teaque Minerals | 1 | 40 | 52 | 51.6 | 16 |
| Fly Ash, Type F | Detroit Edison | 1 | 40 | 52 | 33.4 | 744 |
| $Na_2S.9H_2O$ | reagent | 1 | 40 | 52 | 0.72 | 2050 |
| hematite | P. Shaw | 1 | 40 | 52 | 1.98 | 2000 |
| diatomaceous earth | Vortex Diatom Filter Powder | 1 | 40 | 52 | 41.1 | 440 |
| $Fe_2O_3$ (red) | Fisher | 0.5 | 9 | 34 | 13.4 | 370 |
| Iron powder $Fe^0$ | electrolytic reagent | 0.5 | 9 | 34 | 0.6 | 600 |
| limonite | Ward's | 0.5 | 9 | 34 | 3.2 | 554 |
| $Fe_3O_4$ (black) | Alfa Products | 0.5 | 9 | 34 | 4.4 | 533 |

### 3.1.3 TCLP of Spiked Soil and Soil with Additives

To minimize leachability of Hg, several grout formulations were prepared with additives that were determined to inhibit the release of Hg to the TCLP solution. Preliminary to this, a set of TCLP tests were conducted in which the behavior of the additives in the presence of spiked soil was assessed. Three samples were prepared. One was the spiked soil for use as a reference, the second was the spiked soil with 1% (by weight) of $Fe^0$, and the third was spiked soil with 1% of $Na_2S.9H_2O$. The tests were run according to the TCLP protocol except that the mass and volume of the sample was reduced to conserve the spiked soil. The ratio of solid to liquid was kept the same in all cases so results are directly comparable. The concentration of Hg in the leachate from the spiked soil was 23.9 ppm. This is far in excess of the TCLP limit of 0.2 ppm and amounts to about half of the total Hg in the soil. Results were similar for the soil with the iron powder, with Hg concentrations of 22.4 ppm in the leachate. The concentration of Hg in leachate from the soil with $Na_2S.9H_2O$ was below the detection limit of 0.1ppm.

## 3.2 Task II - Test Specimen Preparation

Five grouting materials were tested including two commercially available materials and three innovative materials. The two commercial grouts are Type I and Type H (similar to Type V) Portland cement. The three innovative grouting systems tested are Tect I, paraffin wax

(WaxFix), and a special magnesium phosphate cement. The innovative materials were formulated and provided by two different vendors. Tect I and WaxFix were provided by Carter Technologies (Contact-Ernie Carter). The third innovative grout tested, magnesium phosphate, was supplied by D. Singh from Argonne National Laboratory (ANL)-Chicago.

Test specimens were prepared/mixed using the standard grout/contaminated soil formulations listed in Table 4.

**Table 4. Grout/Soil Mixing Formulations**

| Grout Type | Grout | Soil |
|---|---|---|
| TECT I | 110 mL | 100 gm |
| Paraffin | 45 mL | 100 gm |
| Type H Portland | 90 mL | 100 gm |
| Type I Portland | 90 mL | 100 gm |
| Phosphate Cement | 50 mL | 100 gm |

Prior to sample preparation a series of measurements were made to determine the density of each grout. This information was required in order to prepare the proper volume of grout. The manufacturers mixing directions for the innovative grouts and the INEEL supplied cement grout formulations were given by weight of components (i.e. 1 mass part Portland cement to 1 mass part water). Grout compositions and densities as used are given in Table 5. The Tect I grout was mixed in a high shear mixer. The portland cements and Waxfix were mixed by hand. The Mag-Phosphate cement samples were prepared by Deleep Singh during a visit to BNL.

**Table 5. Grout Compositions/Densities**

| Grout Type | Wt of Solid | Wt of Liquid | Volume Produced | Density of Grout |
|---|---|---|---|---|
| Tect I/Tect-Hg | 200 g powder | 72.4g liquid | 120 ml | 2.27 g/cm³ |
| Portland Type I | 50 g powder | 50 g water | 67 ml | 1.49 g/cm³ |
| Portland Type H | 50 g powder | 50 g water | 67 ml | 1.49 g/cm³ |
| Waxfix | 100 g Waxfix 125 | 12 g Waxfix 12 | 57 ml | 0.88 g/cm³ |

After grout densities were measured, it was necessary to determine final sample densities to determine how large each lot should be. From the formulation in table one, 100 g of INEEL soil were mixed with the requisite amount of grout and poured into a graduated mold. Soil/grout densities are given in Table 6.

**Table 6. Soil-Grout Composite Densities**

| Grout Type | Soil/Grout Product Density |
|---|---|
| Tect I/Tect-Hg | 2.12 g/cm³ |
| Portland Type I | 1.75 g/cm³ |
| Portland Type H | 1.75 g/cm³ |
| Waxfix | 1.40 g/cm³ |

Each lot was formulated to produce 350 ml of product. Each grout lot was introduced into a cylindrical polyvinyl chloride (PVC) pipe mold measuring approximately 26–35 cm in length and 3.8 cm in diameter. The soil used was the mercury spiked soil prepared earlier. From the grout densities, component quantities were calculated to make 3 samples, each having a volume of 90 mL, in each mold. Additional material was used in each mold to allow for 10% loss for cutting samples. To assure that the samples were homogeneous, the samples were cut from the center of the mold, leaving about 4 cm on each end of the solidified material in the mold as waste after cutting.

Tect I and Tect-Hg samples were mixed in a high shear laboratory mixer (Figure 2). The product was very thick and had to be vibrated into the molds. After pouring and vibrating for one to two minutes the mold was capped and allowed to cure for thirty days. Waxfix samples were made by melting the wax at 75 °C and mixing in the Waxfix 12 using the high shear mixer. The still molten wax mix was added to the soil and mixed by hand. Due to cooling by the soil the mix hardened to a point that it had to be reheated. [Note: We did not preheat the soil for fear of driving off some of the mercury. Once wetted with the wax, the mercury would be less likely to escape.] After reheating, the wax-soil blend was re-mixed and poured with light vibration, into the mold. The mold was capped and left for thirty days. The portland cement samples [Type I and Type H) were mixed by hand. The grout was added to the soil and stirred until no clumping was observed. The mix was poured into the PVC mold, capped and allowed to cure for thirty days. The Mag-Phosphate cement samples were prepared by Deleep Singh during a visit to BNL. They were hand mixed and poured into similar PVC mold and allowed to cure.

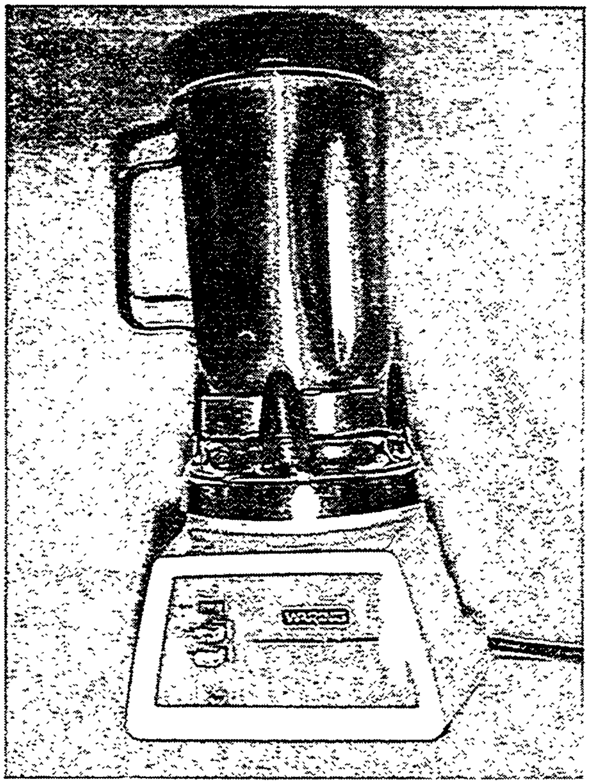

**Figure 2.** High-shear laboratory mixer used for the Tect-grout and Waxfix samples.

At the end of thirty days the caps were removed from all the molds and the molds and samples were cut into 7.6-cm lengths using a wet masonry-saw with a diamond-impregnated blade. The PVC casing was then removed from each individual sample. The samples were measured, weighed and numbered and then stored in 100% relative humidity until compressive strength measurements were made. Figures 3 depicts, from left to right, typical samples of Waxfix, Tect I, Mag-Phosphate cement, Tect-Hg and Portland Type I cement samples after curing, cutting and demolding.

**Figure 3.** Stabilized Soil Samples from the INEEL Acid Pit Treatability Study.

The same procedures were repeated for the grouts with the sodium sulfide additive. The mass of sulfide added was 2% of the soil mass. For the cements and TECT products the sulfide was dissolved into the liquid part. For the Waxfix, the sulfide was pulverized into a fine powder and thoroughly mixed into the molten wax grout. Table 7 lists the number of test specimen sets that were prepared for each grout material and the tests performed on each set. Appendix A gives the measurements weights and densities of the samples tested.

**Table 7. Test Specimen Sets and Associated Testing Requirements**

| Grout Type | Mixture Specification | Total Compressive Strength Samples | Total TCLP Samples* |
|---|---|---|---|
| Magnesium Phosphate Cement | No binding agent | 3 | 2 |
| TECT | No binding agent | 3 | 2 |
| | Sodium Sulfide additive | 3 | 2 |
| | Special mixture (Tect-Hg) | 3 | 2 |
| Paraffin | No binding agent | 3 | 2 |
| | Sodium Sulfide additive | 3 | 2 |
| Type H Portland | No binding agent | 3 | 2 |
| | Sodium Sulfide additive | 3 | 2 |
| Type I Portland | No binding agent | 3 | 2 |
| | Sodium Sulfide additive | 3 | 2 |

*Compressive strength samples were used for TCLP analyses of mercury.

## 3.3 Task III - Performance Testing

The objective of Task III was to evaluate the grout properties through a series of two standard tests; compressive strength and TCLP. The tests provided information for selecting a grout material for field demonstration testing.

Baseline compressive strength measurements were performed on test specimens of Acid Pit soils mixed with Tect I, Tect-Hg, Type I and Type H Portland cement, Magnesium-Phosphate cement, Tect I with additive, and Portland Type I and Type H with additive following American Society for Testing and Materials (ASTM) C-39, "Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens." The Waxfix and Waxfix with additive sample sets were compression tested using ASTM D-695, "Standard Test Method for Compressive Properties of Rigid Plastic." The grout/soil mixtures were compression tested in triplicate on a Versa Tester Universal Compression Testing Instrument (Figure 4). At the failure point the samples fractured and pieces flew off. Plastic bags, placed around the samples and taped shut were used to capture all the fragments of the samples during the compressive loading. This served two purposes; to collect all the mixed waste particulates for health and safety and to collect the fragments to use later on in the TCLP analysis.

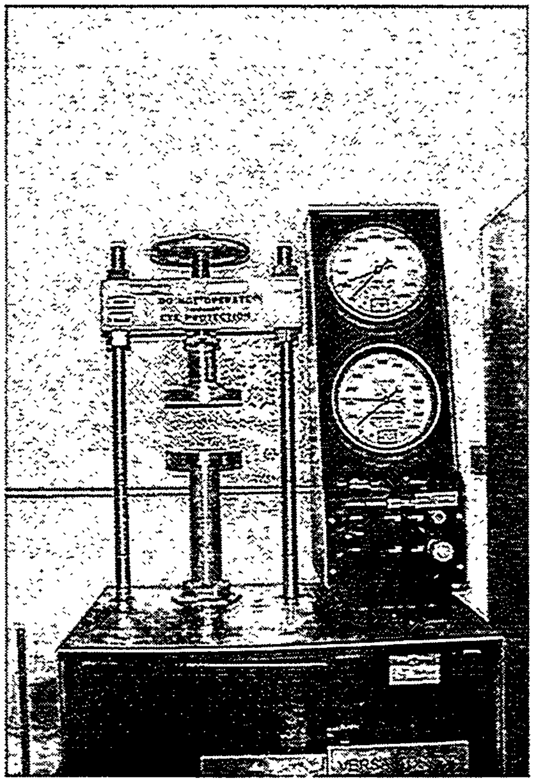

**Figure 4.** Versa Tester Universal Compression Testing Instrument

Results of the compressive strength testing are given in Table 8.

**Table 8. Compressive Strengths of Grouted Soils**

| Grout Type | ID | Load, Lbs | Compressive Strength, psi | Compressive Strength, MPa |
|---|---|---|---|---|
| Waxfix | 1-1 | 260 | 130 | 0.9 |
| | 1-2 | 290 | 150 | 1.0 |
| | 1-3 | 300 | 150 | 1.1 |
| | **Avg** | **280** | **140** | **1.0** |
| | Std. dev. | 17 | 9 | 0.1 |
| Tect-Hg | 2-1 | 2280 | 1120 | 7.7 |
| | 2-2 | 4360 | 2140 | 14.8 |
| | 2-3 | 4820 | 2370 | 16.3 |
| | **Avg** | **3820** | **1880** | **12.9** |
| | Std. dev. | 1100 | 543 | 3.7 |
| Portland Type I Cement | 3-1 | 3070 | 1510 | 10.4 |
| | 3-2 | 3340 | 1640 | 11.3 |
| | 3-3 | 3380 | 1660 | 11.4 |
| | **Avg** | **3260** | **1600** | **11.1** |
| | Std. dev. | 138 | 68 | 0.5 |
| Tect I | 4-1 | 4880 | 2400 | 16.5 |
| | 4-2 | 3740 | 1840 | 12.7 |
| | 4-3 | 4860 | 2390 | 16.5 |
| | **Avg** | **4490** | **2210** | **15.2** |
| | Std. dev. | 533 | 262 | 1.8 |
| Mag-Phosphate Cement | 5-1 | 280 | 140 | 0.9 |
| | 5-2 | 310 | 150 | 1.0 |
| | 5-3 | 300 | 150 | 1.0 |
| | **Avg** | **300** | **140** | **1.0** |
| | Std. dev. | 12 | 7 | 0.0 |

Table 8. Compressive Strengths of Grouted Soils (continued)

| Grout Type | ID | Load, Lbs | Compressive Strength, psi | Compressive Strength, MPa |
|---|---|---|---|---|
| Waxfix with Sodium Sulfide | 6-1 | 240 | 120 | 0.8 |
| | 6-2 | 320 | 160 | 1.1 |
| | 6-3 | 300 | 150 | 1.0 |
| | Avg | 290 | 140 | 1.0 |
| | **Std. dev.** | **34** | **17** | **0.1** |
| Tect I with Sodium Sulfide | 7-1 | 4960 | 2440 | 16.8 |
| | 7-2 | 3180 | 1560 | 10.8 |
| | 7-3 | 3520 | 1710 | 11.8 |
| | **Avg** | **3890** | **1900** | **13.1** |
| | Std. dev. | 772 | 382 | 2.6 |
| Portland Type I with Sodium Sulfide | 8-1 | 2320 | 1140 | 7.9 |
| | 8-2 | 1960 | 960 | 6.6 |
| | 8-3 | 2120 | 1040 | 7.2 |
| | **Avg** | **2130** | **1050** | 7.2 |
| | Std. dev. | 147 | 72 | 0.5 |
| Portland Type H with Sodium Sulfide | 9-1 | 1200 | 580 | 4.0 |
| | 9-2 | 1400 | 680 | 4.7 |
| | 9-3 | 1360 | 670 | 4.6 |
| | **Avg** | **1320** | **640** | **4.4** |
| | Std. dev. | 86 | 43 | 0.3 |
| Portland Type H | 10-1 | 1320 | 650 | 4.5 |
| | 10-2 | 1360 | 670 | 4.6 |
| | 10-3 | 1060 | 520 | 3.6 |
| | **Avg** | **1250** | **610** | 4.2 |
| | Std. dev. | 133 | 65 | 0.5 |

After compressive strength testing, duplicate samples of each grout/soil mixture were subjected to TCLP analyses for mercury. The procedures used for TCLP analyses were EPA method SW846 1311 for TCLP Extraction and EPA method SW846 method 7470, "Mercury"(Hg in Liquid Wastes-Manual Cold Vapor Method).

Size reduction was required to ensure the waste was smaller than 1 cm in its narrowest dimension [i.e., capable of passing through a 9.5-mm (0.375-in.) standard sieve]. Immediately following compressive strength testing, sample fragments were taken and further reduced in size. This was done by placing the fragments into multiple layers of plastic bags and striking the fragments with a hammer. The crushed pieces where then sieved using a series of sieves, starting with a 9.5 mm sieve, followed by an 8 mm, to a 6.7 mm, to a 4.75 mm, to a 1 mm and finally a catch pan. The 100 grams of material required for TCLP testing was obtained primarily from the material passing through the 9.5 mm sieve but retained on the 8 mm sieve. If 100 g could not be collected from the 9.5 mm sizing then material that passed the 8 mm sieve but was retained by the 6.7 mm was used. If this still did not collect 100 grams, then material that passed the 6.7 mm but was retained by the 4.75 mm sieve was used. In no case was it necessary to go below the 4.75 mm sizing. Five grams of material that passed through the 1 mm sieve were used to determine which TCLP leachate would be utilized (1 or 2, see TCLP characterization).

### 3.3.1 TCLP of Solidified Soils

A series of TCLP tests were conducted on soils that had been solidified with various reagents. A 5 g subsample of each was tested for pH as per the TCLP protocol. Four of the solidified product samples required type 1 leachant, while the portland cement material required type 2 leachant because of its high alkalinity.

While analysis of Hg for screening tests was done by ICP, analysis for other tests was performed by the Cold Vapor method required by the EPA. This method, # 7470, includes a digestion process which was also done. A Perkin Elmer model 4000 atomic absorption spectrophotometer, as shown in Figure 5, was used with an MHS-10 vapor generation system. Reagents were made fresh daily. Three standards and a reagent blank were processed with each batch of samples. A 50-fold dilution was made of each sample prior to digestion. Twenty mL of sample (including digestion reagents) were used in each cold vapor analysis. Results are given in Table 9. From these data, it is apparent that only the Tect-Hg grout unequivocally passed the TCLP limit for Hg of 200 ppb. The next best material was the Tect I grout.

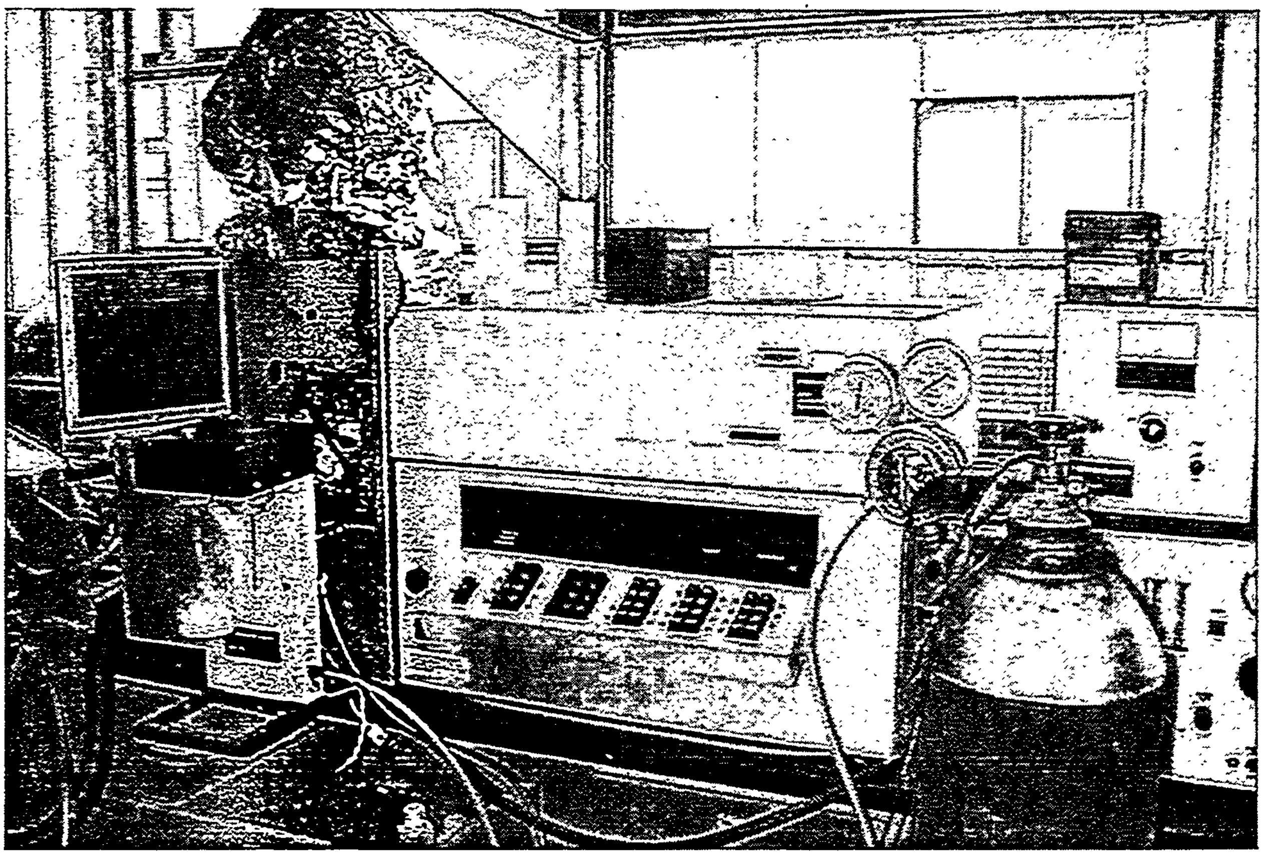

**Figure 5.** Perkin-Elmer Model 4000 Atomic Adsorption Spectrophotometer with the cold vapor generator for Hg analysis.

**Table 9. Hg Concentration in TCLP Leachate from Solidified Soil**

| Sample | Material | Hg (ppb) |
|---|---|---|
| 1-1 | WAXFIX | 630 |
| 1-2 | WAXFIX | 630 |
| 2-1 | Tect-Hg | 48 |
| 2-2 | Tect-Hg | 5.4 |
| 3-1 | portland type I cement | 570 |
| 3-2 | portland type I cement | 630 |
| 4-1 | Tect I (no additive) | 200 |
| 4-2 | Tect I (no additive) | 150 |
| 5-1 | Mag-phosphate | 530 |
| 5-2 | Mag-phosphate | 600 |
| Reagent Blank | TCLP reagent | 0.3 |

### 3.3.2 TCLP of Solidified Soil with Additives

In addition to the solidified soils, a set of 5 grout materials, 4 with additives, were prepared and tested with the TCLP protocol. Three materials were based on portland cement and required the type 2 leachant, while the other two required type I. In every case the additive was $Na_2S$. Results are shown in Table 10. Use of the additive profoundly reduced the concentration of Hg in the TCLP leachate, with all concentrations being significantly below the required 200 ppb. In fact, both types of portland cement containing the $Na_2S$ additive had releases of Hg that were no greater than the reagent blank for the analysis, indicating that Hg releases were below detection. The Tect I grout had only slightly higher Hg concentrations in the leachate; the difference between this material and the portland cements, as far as TCLP leaching is concerned, is negligible. The Type H portland cement (without additives), as with the other portland cements, did not pass the TCLP.

## 4.0 CONCLUSIONS

Of the as-received materials tested, only the Tect-Hg passed the TCLP. When $Na_2S$ was added, all materials passed TCLP.

**Table 10. Concentrations of Hg from TCLP of Solidified Soil Plus Additives**

| Sample | Material | Hg (ppb) |
|---|---|---|
| 6-1 | Waxfix + $Na_2S$ | 11.6 |
| 6-2 | Waxfix + $Na_2S$ | 14.6 |
| 7-1 | Tect I + $Na_2S$ | 0.6 |
| 7-2 | Tect I + $Na_2S$ | 0.7 |
| 8-1 | portland type I + $Na_2S$ | 0.3 |
| 8-2 | portland type I + $Na_2S$ | 0.3 |
| 9-1 | portland type H + $Na_2S$ | 0.5 |
| 9-2 | portland type H + $Na_2S$ | 0.3 |
| 10-1 | portland type H | 428 |
| 10-2 | portland type H | 272 |
| Reagent Blank | | 0.3 |

Appendix A. Test Specimen Data

| Sample ID | Diameter cm | Length cm | Weight g | Surface Area $cm^2$ | Volume $cm^3$ | Density $g/cm^3$ |
|---|---|---|---|---|---|---|
| Waxfix | | | | | | |
| 1-1 | 4.04 | 7.87 | 140.3 | 12.8 | 101 | 1.39 |
| 1-2 | 4.04 | 7.90 | 144.0 | 12.8 | 101 | 1.42 |
| 1-3 | 4.01 | 7.85 | 133.0 | 12.6 | 99 | 1.34 |
| **avg.** | **4.03** | **7.87** | **139.1** | **12.8** | **100** | **1.38** |
| std. dev. | 0.01 | 0.02 | 4.6 | 0.1 | 0.84 | 0.03 |
| | | | | | | |
| Tect-Hg | | | | | | |
| 2-1 | 4.09 | 7.92 | 200.0 | 13.1 | 104 | 1.92 |
| 2-2 | 4.09 | 7.92 | 200.4 | 13.1 | 104 | 1.93 |
| 2-3 | 4.09 | 7.90 | 201.6 | 13.1 | 104 | 1.94 |
| **avg.** | **4.09** | **7.92** | **200.7** | **13.1** | **104** | **1.93** |
| std. dev. | 0.00 | 0.01 | 0.7 | 0.0 | 0.16 | 0.01 |
| | | | | | | |
| Portland Cement Type I | | | | | | |
| 3-1 | 4.09 | 7.92 | 167.8 | 13.1 | 104 | 1.61 |
| 3-2 | 4.09 | 7.92 | 167.7 | 13.1 | 104 | 1.61 |
| 3-3 | 4.09 | 7.92 | 168.1 | 13.1 | 104 | 1.61 |
| **avg.** | **4.09** | **7.92** | **167.9** | **13.1** | **104** | **1.61** |
| std. dev. | 0.00 | 0.00 | 0.2 | 0.0 | 0.00 | 0.00 |
| | | | | | | |
| Tect I w/no additive | | | | | | |
| 4-1 | 4.09 | 7.90 | 206.0 | 13.1 | 104 | 1.99 |
| 4-2 | 4.09 | 7.92 | 204.0 | 13.1 | 104 | 1.96 |
| 4-3 | 4.09 | 7.92 | 203.7 | 13.1 | 104 | 1.96 |
| **avg.** | **4.09** | **7.92** | **204.6** | **13.1** | **104** | **1.97** |

| | | | | | | |
|---|---|---|---|---|---|---|
| std. dev. | 0.00 | 0.01 | 1.0 | 0.0 | 0.16 | 0.01 |
| | | | | | | |
| Mag-Phosphate Cement | | | | | | |
| 5-1 | 4.11 | 7.90 | 163.4 | 13.3 | 105 | 1.56 |
| 5-2 | 4.09 | 7.85 | 162.9 | 13.1 | 103 | 1.58 |
| 5-3 | 4.11 | 7.85 | 165.2 | 13.3 | 104 | 1.58 |
| **avg.** | **4.11** | **7.87** | **163.8** | **13.2** | **104** | **1.57** |
| std. dev. | 0.01 | 0.02 | 1.0 | 0.1 | 0.81 | 0.01 |
| | | | | | | |
| Waxfix w/Sodium Sulfide | | | | | | |
| 6-1 | 4.09 | 7.82 | 142.2 | 13.1 | 103 | 1.38 |
| 6-2 | 4.09 | 7.82 | 146.7 | 13.1 | 103 | 1.43 |
| 6-3 | 4.09 | 7.85 | 137.8 | 13.1 | 103 | 1.34 |
| **avg.** | **4.09** | **7.83** | **142.2** | **13.1** | **103** | **1.38** |
| std. dev. | 0.00 | 0.01 | 3.6 | 0.0 | 0.16 | 0.04 |
| | | | | | | |
| Tect I w/Sodium Sulfide | | | | | | |
| 7-1 | 4.09 | 7.82 | 202.4 | 13.1 | 103 | 1.97 |
| 7-2 | 4.09 | 7.82 | 203.9 | 13.1 | 103 | 1.98 |
| 7-3 | 4.11 | 7.80 | 206.3 | 13.3 | 104 | 1.99 |
| **avg.** | **4.10** | **7.81** | **204.2** | **13.2** | **103** | **1.98** |
| std. dev. | 0.01 | 0.01 | 1.6 | 0.1 | 0.44 | 0.01 |
| | | | | | | |
| Portland Cement Type I w/Sodium Sulfide | | | | | | |
| 8-1 | 4.09 | 7.82 | 166.4 | 13.1 | 103 | 1.62 |
| 8-2 | 4.09 | 7.85 | 165.7 | 13.1 | 103 | 1.61 |
| 8-3 | 4.09 | 7.82 | 168 | 13.1 | 103 | 1.63 |
| **avg.** | **4.09** | **7.83** | **166.7** | **13.1** | **103** | **1.62** |
| std. dev. | 0.00 | 0.01 | 1.0 | 0.0 | 0.16 | 0.01 |

| | | | | | | |
|---|---|---|---|---|---|---|
| Portland Cement Type H w/Sodium Sulfide | | | | | | |
| 9-1 | 4.11 | 7.82 | 174.8 | 13.3 | 104 | 1.68 |
| 9-2 | 4.11 | 7.82 | 173.2 | 13.3 | 104 | 1.66 |
| 9-3 | 4.09 | 7.82 | 171.7 | 13.1 | 103 | 1.67 |
| **avg.** | **4.11** | **7.82** | **173.2** | **13.2** | **104** | **1.67** |
| std. dev. | 0.01 | 0.00 | 1.3 | 0.1 | 0.60 | 0.01 |
| Portland Cement Type H | | | | | | |
| 10-1 | 4.09 | 7.82 | 175 | 13.1 | 103 | 1.70 |
| 10-2 | 4.09 | 7.85 | 176.9 | 13.1 | 103 | 1.72 |
| 10-3 | 4.09 | 7.85 | 176.3 | 13.1 | 103 | 1.71 |
| **avg.** | **4.09** | **7.84** | **176.1** | **13.1** | **103** | **1.71** |
| std. dev. | 0.00 | 0.01 | 0.8 | 0.0 | 0.16 | 0.01 |