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## IN-SITU STABILIZATION OF MIXED WASTE CONTAMINATED SOIL

**REFERENCE:** Siegrist, R.L., Cline, S.R., Gilliam, T.M., and Conner, J.R., "In-Situ Stabilization of Mixed Waste Contaminated Soil," Stabilization and Solidification of Hazardous, Radioactive and Mixed Wastes, ASTM STP 1240, T. Michael Gilliam and Carlton C. Wiles, Eds., American Society for Testing and Materials, Philadelphia, 1995.

**ABSTRACT:** A full-scale field demonstration was conducted to evaluate in situ solidification for stabilizing an inactive RCRA land treatment site at a DOE facility in Ohio. Subsurface silt and clay deposits were contaminated principally with up to 500 mg/kg of trichloroethylene and other halocarbons, but also trace to low levels of Pb, Cr, <sup>235</sup>U, and <sup>99</sup>Tc. In situ solidification was studied in three, 3.1 m diameter by 4.6 m deep columns. During mixing, a cement-based grout was injected and any emissions from the mixed region were captured in a shroud and treated by filtration and carbon adsorption. During in situ processing, operation and performance parameters were measured, and soil cores were obtained from a solidified column 15 months later. Despite previous site-specific treatability experience, there were difficulties in selecting a grout with the requisite treatment agents amenable to subsurface injection and at a volume adequate for distribution throughout the mixed region while minimizing volume expansion. Observations during the demonstration revealed that in situ solidification was rapidly accomplished (e.g., >90 m<sup>3</sup>/d) with limited emissions of volatile organics (i.e., < 1 wt% of initial Soil VOCs). Grout distribution within the mixed region was not uniform, with marked differences in compressive strength and elemental content with depth. Volume expansion was significant (30% v/v) and the hydraulic conductivity of the solidified soil was greater than that of the undisturbed natural deposit (10<sup>-6</sup> cm/s vs. 10<sup>-8</sup> cm/s). Leaching tests performed on the treated samples revealed non-detectable to acceptably low concentrations of all target contaminants.

**KEYWORDS:** closure, remediation, environmental restoration, solidification, cement, grout, RCRA

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## INTRODUCTION

An emerging approach to rapid in situ treatment of source areas (e.g., land treatment units) involves the use of soil mixing coupled with physicochemical treatment processes (Fig. 1). Application is attractive since it offers perhaps the only way to rapidly and extensively disperse treatment agents and concomitantly remove, degrade, and/or immobilize contaminants in place. Coupling the injection of solidification/ stabilization (S/S) agents with in situ mixing has been employed to treat inorganics and heavier molecular weight organics [1-5], but not mixed contaminants or media with low permeability (e.g., silts and clays). The goal of solidification is to produce a strong, structurally stable waste form with a reduced leach potential while the stabilization effort is to reduce a waste's solubility and/or chemical reactivity via use of a site-tailored reagent mix [1-3].

Stabilization/solidification techniques involving cement have commonly been used as a remediation technique for inorganic species. Many have questioned whether organic contaminants can be effectively stabilized and solidified in a cement-based grout. Volatile organics can be released during in situ mixing. High concentrations of organics can hinder the solidification process by adsorbing onto the cement particles and inhibiting the hydration process. Recent laboratory research has indicated that cement-based grouts can to some extent capture and immobilize organic compounds [1,5].

To evaluate the operation and performance of S/S, a full-scale field test was conducted as part of the X-231B Technology Demonstration project initiated at Oak Ridge National Laboratory (ORNL) by the U.S. Department of Energy (DOE) and Martin Marietta Energy Systems, Inc. (MMES) in November 1990 [6-12]. The overall goal of the project was to demonstrate a feasible and cost-effective process for closure and environmental restoration of the X-231B Solid Waste Management Unit at the DOE Portsmouth Gaseous Diffusion Plant located in southern Ohio. The X-231B Unit was used from 1976 to 1983 as a land disposal site for waste oils and solvents. Dense silt and clay deposits ( $K_{sat} < 10^{-6}$  cm/s) beneath the unit were contaminated with TCE, 1,1,1-trichloroethane (TCA), and other VOCs at concentrations up to 500 mg/kg and low levels of uranium and technetium (TABLE 1). The shallow ground water (water table at  $\approx 3.6$  to 4.2 m depth) was also contaminated, with TCE well above drinking water standards. As part of the closure of the Unit, reduction of >70% of the mass of VOCs present in the 0 to 6.6 m depth zone was required, while controlling the fate of heavy metals and radionuclides.

Due to the mixed contamination present and the great cost of ex situ treatment options, it was decided to conduct a demonstration to determine if an in situ treatment alternative could be employed. Over an 18 month period beginning in July 1991, the project included process and spatial modeling studies, bench- and pilot-scale laboratory experimentation, and full-scale field demonstration and testing.

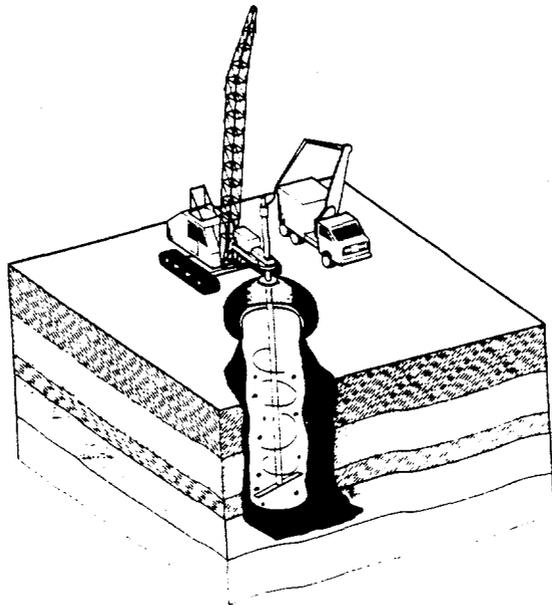


FIG. 1--Illustration of an in situ S/S concept where treatment agents are delivered through the mixing blade while emissions are captured in the shroud covering the mixed region.

TABLE 1--Representative characteristics of the site [5-8].<sup>a</sup>

| Characteristic                  | Nominal subsurface depth (m) |            |           |
|---------------------------------|------------------------------|------------|-----------|
|                                 | 0 - 0.6                      | 1.8 - 2.4  | 4.8 - 5.4 |
| Particle size distribution:     |                              |            |           |
| Clay: <0.002 mm, wt%            | ...                          | 22 - 25    | 12 - 15   |
| Silt: 0.002 - 0.05 mm, wt%      | ...                          | 65 - 67    | 39 - 64   |
| Sand: 0.05 - 2.0 mm, wt%        | ...                          | 8 - 12     | 22 - 46   |
| Water content, dry wt%          | 16 - 23                      | 17 - 19    | 22 - 24   |
| Liquid limit, wt%               | ...                          | ...        | 25 - 26   |
| Plastic limit, wt%              | ...                          | ...        | 20 - 23   |
| pH                              | ...                          | 5.3 - 6.0  | 6.2 - 7.4 |
| Total organic carbon, mg/kg     | ...                          | 600 - 1200 | 200 - 500 |
| Total VOCs <sup>b</sup> , ug/kg | 9900                         | 5300       | 1500      |
| Chromium mg/kg                  | ...                          | 14-31      | 9-20      |
| Lead mg/kg                      | ...                          | 20-28      | 16-23     |
| Nickel mg/kg                    | ...                          | 8-18       | 7-20      |
| Total alpha, nCi/kg (10)        | nd-150                       | nd         | nd        |
| Total beta, nCi/kg (10)         | nd-200                       | nd-31      | nd-33     |
| Total Uranium, mg/kg            | 2-150                        | 1-3        | 2-3       |
| Technetium, nCi/kg (2)          | nd-380                       | ...        | ...       |

<sup>a</sup> Representative results based on multiple sampling and analysis events conducted prior to the field demonstration. "nd" indicates constituent not detected at limit shown in ( ).

<sup>b</sup> Total VOCs = summation of trichloroethylene (TCE), methylene chloride (MC), 1,1,1-trichloroethane, 1,2-dichloroethylene, and 1,1-dichloroethane. Of these, the principal VOCs were TCE and MC.

Three mixed region treatment processes were evaluated: vapor stripping, chemical oxidation, and solidification. This paper presents a synopsis of the field demonstration of in situ S/S technology at the X-231B site.

While the overall X-231B Technology Demonstration was conceived and directed by ORNL and MMES, the responsibility for field demonstration of the in situ S/S process, including design of the operating conditions and implementation of the process, was awarded to Chemical Waste Management, Inc. (CWM) (Columbia, SC) and Millgard Environmental Corporation (MEC) (Livonia, MI). Further details regarding research of the in situ S/S process as well as other facets of the demonstration may be found elsewhere [6-12].

## **MATERIALS AND METHODS**

### Process Description

Replicated tests of in situ S/S were made using the MecTool<sup>®1</sup> mixing system. This system was comprised of a track-mounted crane with a hollow, Kelly bar attached to a drilling tool with two, 1.5 m long blades, yielding an effective mixing diameter of  $\approx 3$  m. In the field test, the MecTool was used to penetrate the subsurface while simultaneously injecting cement-based grout through 1.6 cm diameter orifices into soil regions, 3.1 m in diameter by 4.6 m deep. The ground surface above the mixed region was covered by a shroud maintained under low vacuum (e.g., 1 kPa) to capture air emissions for treatment by activated carbon adsorption and high efficiency particulate filtration (HEPA).

Laboratory treatability studies conducted in early 1992 by two private vendors demonstrated that clay soil at the Portsmouth site could be solidified, and a grout formulation was recommended [10]. The grout actually used however for the S/S demonstration was changed based on insight gained during pre-demonstration shakedown tests (Table 2). The formulation was adjusted by changing from powdered to granular activated carbon and by adding fly ash (see Table 2). This was done to increase the consistency and fluidity of the grout. A retarder was also added to provide a working time of 2 h. At the demonstration site, additional water was added to the grout to further increase workability. Approximately  $10.5 \text{ m}^3$  ( $14 \text{ yd}^3$ ) of grout were injected into each of three columns (approximately 30% v/v, 20% wt/wt grout). Two of the three columns were surrounded by undisturbed soil (Z1, Z2), while the third central column (Z3) was placed to overlap the other two columns by approximately 1 m (15% of column area) (Fig. 2).

The auger was first positioned above the column to be treated. The grout was prepared at a local batch plant and delivered to the site in  $5.3 \text{ m}^3$  ( $7 \text{ yd}^3$ ) cement trucks. The grout was deposited into a grout hopper, having 0.6-cm screen openings, and sieved before being pumped

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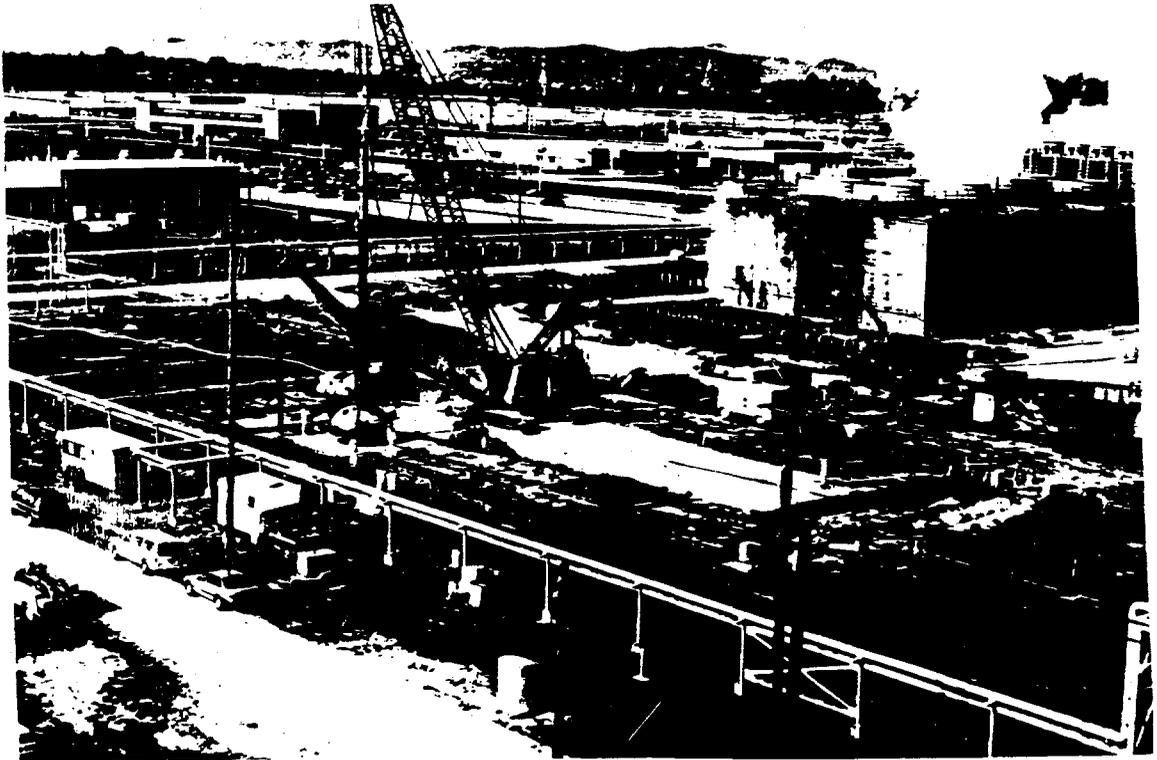


FIG. 3--Overview of the demonstration site with the MecTool® system and supporting test equipment and onsite laboratories.



FIG. 4--The grout delivery system used during the field demonstration in May 1992.

## Demonstration Monitoring

During the demonstration, monitoring activities focused on operational parameters such as auger rotation speed and depth, grout injection characteristics, off-gas composition, mechanical problems, etc. A computerized data acquisition system (DAS) linked to various sensors enabled near-continuous monitoring and real-time data display (i.e., recording intervals of 0.2 to 2 minutes). In addition, samples of soil were collected before and immediately after in situ S/S for analyses of physical and chemical characteristics. On-site DAS and analytical instrumentation was housed in temperature-controlled trailers on-site.

Process operation and off-gas monitoring--Auger position was monitored by a geollogger on the MecTool that was connected to the DAS and was monitored and recorded approximately every minute. The grout delivery rate was controlled and monitored by a flow meter on the grout pump, and the volume of grout added was controlled and monitored by the number and capacity of the grout delivery trucks. The off-gas flow rate was monitored using both a hand-held anemometer inserted in the 20 cm off-gas line and a vortex shedding meter inserted in a slipstream of the off-gas treatment system, both of which were connected to the DAS. Off-gas temperature was monitored with a thermocouple connected to the DAS. Off-gas monitoring for VOCs was conducted by directing a small sidestream of the off-gas leaving the shroud covering the mixed region through small-diameter tubing to a flame ionization detector (FID) (MSA Instruments, Model 1015B) connected to the DAS. The FID was calibrated before and after each column. To verify the FID response, discrete gas samples were collected at selected times during in situ processing and analyzed immediately by gas chromatography with an electron capture detector (Hewlett Packard Model 5890). Finally, particulates in the off-gas were qualitatively measured using an air sampling pump attached to the off-gas piping system. Time-composited air samples were collected and analyzed for total particulate concentrations, and gross alpha and gross beta activity.

Solid sample collection and analysis--A few days prior to the actual demonstration, pretreatment soil samples were collected from seven locations within the area to be treated by in situ S/S. Samples were taken at four (4) depth intervals, 0.3-0.6 m, 1.2-1.5 m, 2.4-2.7 m, and 3.9-4.2 m, using a truck-mounted Geoprobe<sup>®2</sup> sampler with a 0.3m long by 2.5 cm diameter soil sampling tool (Envirosurv, Inc., Arlington, VA). A subsample from each sample location was extruded from the sampler and screened on-site for radioactivity using hand-held detectors. Then additional subsamples were extruded into 40 mL VOA septum vials, labeled, and subjected to on-site and off-site laboratory analysis. Within hours of completion of the in situ S/S demonstration, samples of the uncured soil/grout mixture were collected using a drill rig, flight auger, and the Geoprobe sampling system. Soil samples were only collected at depths of 1.2-1.5 and 2.4-2.7 m. Additional samples of the soil/grout mixture were collected at 1.2-1.5 and 2.4-2.7 m and CWM conducted leaching tests according to the Toxicity Characteristic

<sup>2</sup>Geoprobe Systems, Salina, KS

Leaching Procedure (TCLP). In addition to the subsurface soil sampling just described, post-treatment soil samples were collected from the above-ground berms created within the shroud as a result of the soil mixing process. These samples were collected using a pre-cleaned stainless steel hand tool. Soil or soil/grout samples were collected from three locations within the berm above each treated column at a depth of approximately 0.3 m below the exposed surface of the berm. All solid samples were analyzed for seven target VOCs in an on-site mobile laboratory (Envirosurv, Inc.). VOC concentrations were determined using a heated (60°C) headspace procedure with a gas chromatograph equipped with an electron capture detector (Shumadzu 14A).

#### Post-Demonstration Core Sampling

After completion of the demonstration in May 1992, the berms over each solidified column were compacted and the temporary geomembrane cap was repaired. In August 1993, 15 months after the demonstration, additional sampling was performed to determine the physical and chemical characteristics of the grouted soil columns. Core samples were collected from the solidified columns by an NX coring drill (5.4 cm diameter) using water for cuttings removal. Two control cores were also collected from an area 3.1 m north of the test columns (Fig. 2). Due to a plant-wide ban on hoisting and lifting, the Z2 column was cored along its entire depth only in the eastern region which overlapped with the Z3 column (PST-SBZ2h), and in the center of the Z2 column (PST-SBZ2g) but only to a depth of 1.5 m. The core segments were removed from the core barrel, placed on plastic sheets, and photographed. Core sample(s) were collected at depth intervals of 0.3-0.6, 1.2-1.5, 2.4-2.7, and 3.9-4.2 m, consistent with those used for sampling before the demonstration. All core segments were packaged and shipped to ORNL for physical and chemical analyses by generally accepted methods [13-16]. Analyses were made or are in progress for micromorphology, mineralogy, moisture content, bulk density, compressive strength, hydraulic conductivity, pH, volatile solids (loss on ignition), elemental composition, and TCLP leachable substances. Physical and chemical properties and their uniformity can be used as indirect measures of S/S effectiveness. Thus, the visual observations and laboratory analyses not only illustrated differences between the treated and untreated samples, but also provided some indication of the relative uniformity and/or spatial variability of the grouted soil columns.

### **RESULTS**

#### In Situ S/S Operation and Performance

During shakedown tests at the onset of the demonstration, mechanical problems were encountered with grout delivery through the hopper/screen/pump to the Kelly Bar, and with the grout formulation and volume addition. Modifications were eventually made and all three test columns were completed within several hours. Grout delivery, injection, and in situ processing of the contaminated media were accomplished relatively smoothly and without mechanical difficulty. A typical plot of auger position with time is presented in Fig. 5, along with the total

VOC concentrations detected in the off-gas with time. As a result of mixing the dense clay soil and the injection of the grout, an above-ground berm was created above each solidified column (approximately 1 m high and equivalent to  $\approx 30\%$  v/v of the mixed region). The berms were eventually leveled out and compacted with vibratory equipment.

The vacuum within the shroud was approximately 155 Pa during processing of columns Z1 and Z2, but only 17 Pa during processing of Z3. It is possible that the lower vacuum observed for Z3 may have been due to a relatively poorer seal between the soil surface and the shroud caused by the berms created during the mixing of columns Z1 and Z2. The average temperature of the off-gas was 26.1°C with little variation.

During in situ processing, there was limited VOC mass removal during the S/S demonstration. The low VOC concentrations that were detected in the off-gas did not appear until after 10 minutes of treatment (Figs. 5,6). The immediate appearance of off-gas VOCs was likely hindered by the initial spraying of grout onto the ground surface prior to auger penetration. Unlike air-stripping processes also evaluated at the X-231 Unit in which  $>95$  wt% of the initial VOC mass was detected in the off-gas system, the total cumulative VOC mass in the off-gas from all three S/S columns (Fig. 6) represented  $<1$  wt% of the initial VOC concentration [9]. In fact, the concentrations detected in the S/S off-gas were below the regulatory exposure limits (8 hr TWA) for all the target VOCs listed in note "a" of TABLE 1. [17]. Thus, it appears that the application of grout onto the surface of the columns before mixing and the rapid addition of grout into the test columns may have effectively limited the transfer of the VOCs from the subsurface into the induced air flow caused by the vacuum on the shroud. The use of activated carbon as a stabilizing agent in the grout formula may have also enhanced the sorption of VOCs onto the soil/grout matrix.

The total VOC concentrations in the pre-treatment soil ranged from 0.1 to over 500 mg/kg, with TCE predominating ( $>80\%$  of initial VOC mass). The highest concentrations were found in the 1.2-2.7 m region (Table 3). In addition to the off-gas analyses, soil samples were collected from the berm and from the fresh soil/grout mixture (Table 3). Concentrations in the post treatment samples were markedly lower than the corresponding pre-treatment samples, even after accounting for a 20 wt% reduction due to the initial concentration being "diluted" by the grout addition. A mass balance computation revealed (Table 3) that only 8 wt% of the pre-treatment VOC mass could be accounted for from the post treatment analyses which were performed. Although some error is inherent when predictions of total VOC masses are based upon a few discrete sample concentrations, it is speculated that as much as 90% of the pre-treatment VOC mass was captured in the grout and immobilized, at least for the short-term. Furthermore, since the average VOC concentration in the berms was very low, it is probable that much of the VOC mass detected in the off-gas originated from the soil found in the berm.

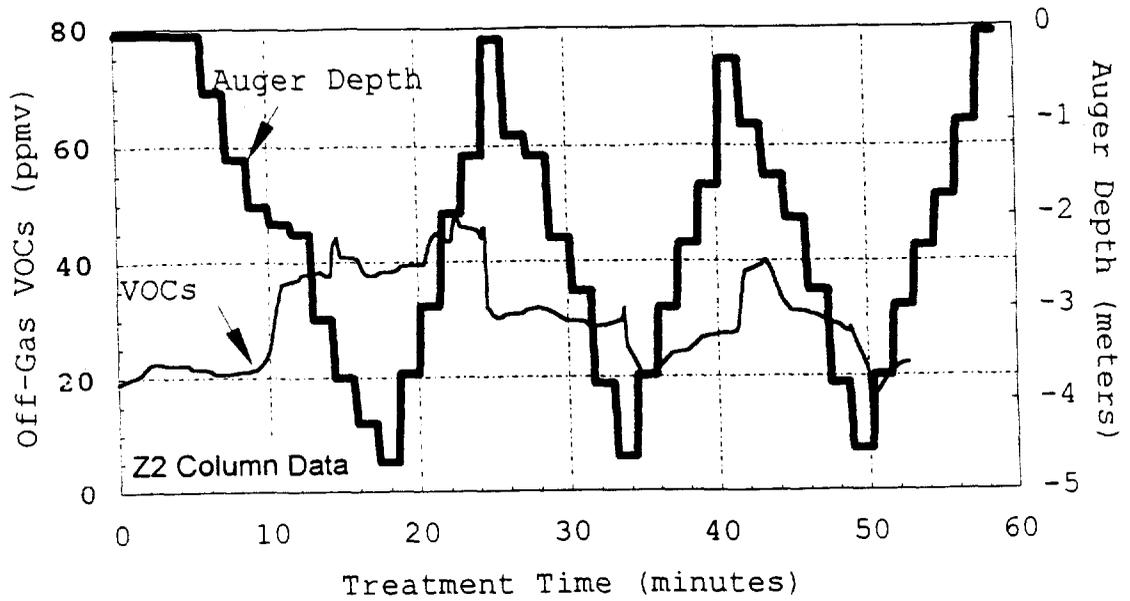


FIG. 5--Mixing operation and off-gas composition during in situ solidification of a 3.1-m diameter by 4.6-m deep region.

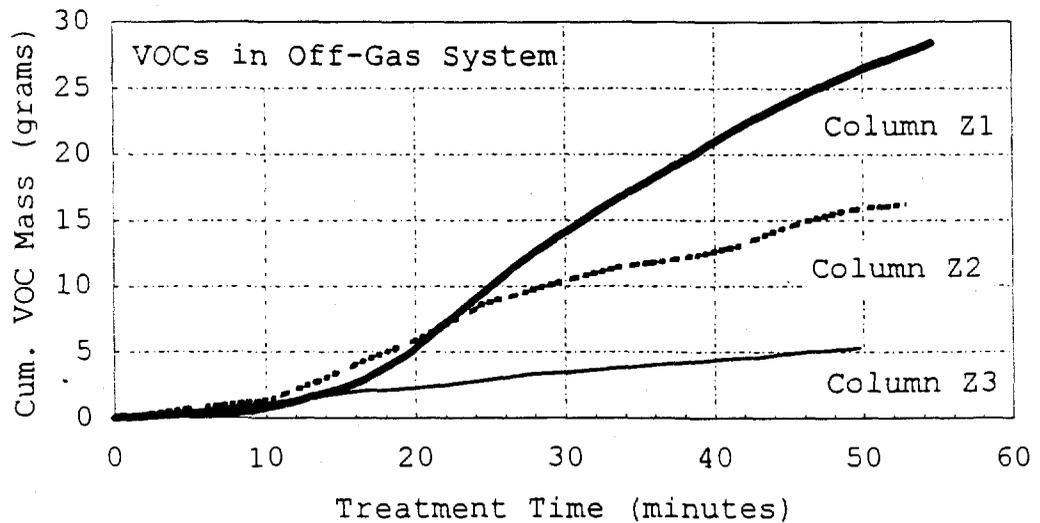


FIG. 6--Mass of VOCs in the off-gas during in situ solidification of each of three, 3.1-m diameter by 4.6-m deep soil regions.

TABLE 3--Pre- and post-treatment total VOC soil concentrations<sup>a</sup>.

| Depth Interval | Pre-treatment |                   | Post-treatment |                  | Initial |
|----------------|---------------|-------------------|----------------|------------------|---------|
|                | (mg/kg)       | (grams)           | (mg/kg)        | (grams)          |         |
| Off-Gas        | --            | --                | --             | 50 <sup>c</sup>  | 0.40    |
| Berm           | --            | --                | 2.39           | 70 <sup>d</sup>  | 0.55    |
| 0-0.9 m        | 48            | 1860 <sup>b</sup> | --             | 252 <sup>e</sup> | 2.0     |
| 0.9-2.1 m      | 84            | 4380 <sup>b</sup> | 5.04           | 252 <sup>e</sup> | 2.0     |
| 2.1-3.4 m      | 106           | 5515 <sup>b</sup> | 3.14           | 180 <sup>e</sup> | 1.40    |
| 3.4-4.6 m      | 20            | 1050 <sup>b</sup> | --             | 180 <sup>e</sup> | 1.40    |
|                | TOTAL:        | 12,805            | TOTAL:         | 984              | 7.75    |

<sup>a</sup> Total VOC concentration is defined as the summation of following seven volatile compounds: trichloroethylene; 1,1,1-trichloroethane; trans-1,2-dichloroethylene; cis- 1,2-dichloroethylene; 1,1-dichloroethylene; 1,1-dichloroethane; and methylene chloride. Values represent the average of all three soil columns in Fig. 2.

<sup>b</sup> Based on bulk density= 1.95g/cm<sup>3</sup> throughout untreated material. It is assumed that VOC concentrations were uniform throughout the given depth intervals for samples collected at the following locations: 0.3-0.6m, 1.2-1.5 m, 2.4-2.7 m, 3.9-4.2 m.

<sup>c</sup> Total VOC mass detected in all three S/S columns processed.

<sup>d</sup> Based on assumption that the berm volume is 50% soil @ bulk density= 1.4 g/cm<sup>3</sup> and 50% grout @ unit wt. =1515 kg/m<sup>3</sup>

<sup>e</sup> Based on mass of soil/grout left in column after creation of berm. Assumed that VOC concentrations were uniform throughout the given depth intervals for samples collected at the following locations: 1.2-1.5 m and 2.4-2.7 m.

Additional soil/grout samples were collected from the 1.2-1.5 m and 2.4-2.7 depth intervals of each test column immediately following the S/S demonstration for TCLP analysis. The analyses, performed by CWM at the Clemson Technical Center, Clemson, SC revealed that the post-treatment samples passed the accepted TCLP leaching limits for both inorganic and organic species.

#### Grouted Soil Characteristics After 15 months.

Physical Characteristics--During coring, substantial fines were "washed out" of the solidified media within the top portion of the first soil core until the water flow used in the drilling operation was adjusted. The core segments collected at the 0.3-0.6, 1.2-1.5, and 2.4-2.7 m depths were brownish yellow in color and tightly grouted with no loose soil fragments present. Discrete particles of activated carbon were clearly visible throughout the samples (Fig. 7). The 3.9-4.2 m depth sample, however, was fragile and clearly a heterogeneous mixture of soil and grout.

The average moisture content of the untreated soil core collected in August 1993 was approximately 18 wt%. Assuming a 0.25 stoichiometric water/cement ratio, the grout formula used (Table 2) would produce a cured specimen having a moisture content near 23.5 wt% (no soil

present). The moisture content of the soil/grout matrix (80 wt% soil) would be approximately 19.1 wt% if it is assumed that none of the 18 wt% moisture in the untreated soil was involved in the hydration of the cement ( $0.20 \times 23.5 \text{ wt\% grout} + 0.80 \times 18 \text{ wt\% soil} = 19.1 \text{ wt\% soil/grout}$ ). The average value determined for the soil/grout core samples was 19.5 wt%. Thus, it appears that little of the original soil moisture was involved in the hydration of the grout. Soil/grout matrices typically obtain higher strength and lower permeability if water is not added in excess (i.e., if free soil water could be available for the cementitious reactions). However, the workability of such a grout formulation would be poor.

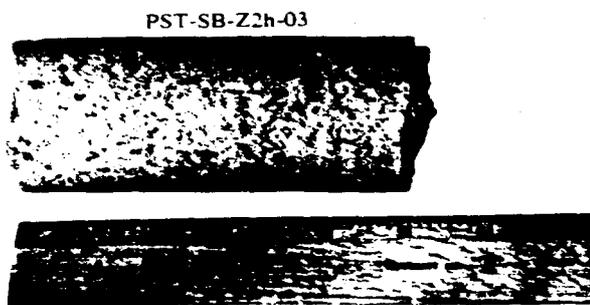


FIG. 7 --Photograph of a solidified clay soil core collected 15 months after completion of the demonstration. (ORNL Photo 7586-93)

Bulk densities of the untreated soil collected at depths of 1.2-1.5 m and 3.9-4.2 m yielded an average bulk density (at field moisture) of  $1.95 \text{ g/cm}^3$ . In contrast, the soil/grout specimens used in the compressive strength tests had an average bulk density of  $1.78 \text{ g/cm}^3$  (Std dev. = 0.15) (Table 5). Theoretically, the bulk density of each soil column should be near  $2.2 \text{ g/cm}^3$ . This value based on mass of soil in column after creation of a 1 m high berm (see footnote "d" of Table 3 for assumptions). In contrast to these results, others have reported bulk density increases of 21% (sandy soil) as a result of in situ S/S and an 8% increase in total volume rather than the 30% volume increase observed here due to the creation of the 1 m high berm. [2]. One would expect an increase in bulk density after in-situ grout injection, particularly when working with high permeability soils, as the grout fills the meso- and macropores present in the original soil matrix. However, the high bulk density of a clay deposit is markedly reduced as a result of mixing. The apparent reduction in bulk density may have also been due to the addition of "excess" water in the grout or to the entrapment and/or entrainment of air into the grout during the mixing operation.

The compressive strength values obtained from the 5 cm diameter core subsamples ranged from 390 to 5200 kPa (56 to 750 psig) and were inversely proportional to the depth of the respective sample locations (Table 5). Such a result was expected after physical inspection of the

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samples prior to the analysis. Again, samples from the upper part of the core appeared to be highly grouted, while the deepest sample (3.9-4.2 m) appeared very fragile with a relatively small amount of grout material present. All values obtained, however, were greater than the currently accepted guideline that grouted material have a compressive strength of at least 340 kPa (50 psig) [4]. Values similar to those obtained in this study were observed in another in-situ S/S project where the final compressive strength values ranged from 520-6000 kPa (75-866 psig) with an average value of 2800 kPa (410 psig) [2].

The hydraulic conductivity ( $K_{sat}$ ) of the cured soil/grout samples was two orders of magnitude greater than that of the untreated soil (Table 5). This is believed to be due to disruption of the dense clay deposit (increase in porosity and pore continuity) as a result of mixing and to the heterogeneity of the soil/grout product. In most S/S applications the final solidified product has a  $K_{sat}$  several orders of magnitude lower than that of the untreated soil. However, most S/S applications have involved soils with very high initial  $K_{sat}$ 's (e.g.,  $10^{-2}$  cm/sec).

TABLE 5--Physical properties of the untreated soil and S/S product.

| Sample description | Sample depth (m) | Water content (wt%) <sup>a</sup> | Bulk density (g/cm <sup>3</sup> ) | Compressive strength (kPa) | Hydraulic conductivity <sup>b</sup> (cm/sec) |
|--------------------|------------------|----------------------------------|-----------------------------------|----------------------------|--|
| Untreated          | 0.3-0.6          | 20.5 <sup>c</sup>                | ...                               | ...                        | ...  |
| Control Core       | 1.2-1.5          | 16.9 <sup>c</sup>                | 2.15                              | ...                        | 8.08x10 <sup>-8</sup>                        |
| (PRE-SB-SB15)      | 2.4-2.7          | 18.4 <sup>c</sup>                | 1.75                              | ...                        | ...  |
|                    | 3.9-4.2          | 16.5 <sup>c</sup>                | ...                               | ...                        | 8.09x10 <sup>-8</sup>                        |
| Treated Core       | 0.3-0.6          | 19                               | 1.73 <sup>c</sup>                 | 5200 <sup>d</sup>          | 8.88x10 <sup>-6</sup>                        |
| (PST-SB-Z2h)       | 1.2-1.5          | 23.5                             | 1.66 <sup>c</sup>                 | 3500                       | ...  |
|                    | 2.4-2.7          | 19                               | 1.72                              | 2600                       | ...  |
|                    | 3.9-4.2          | 13.6                             | 2.00 <sup>c</sup>                 | 390                        | 7.75x10 <sup>-6</sup>                        |

<sup>a</sup> Analyses performed at 60°C.

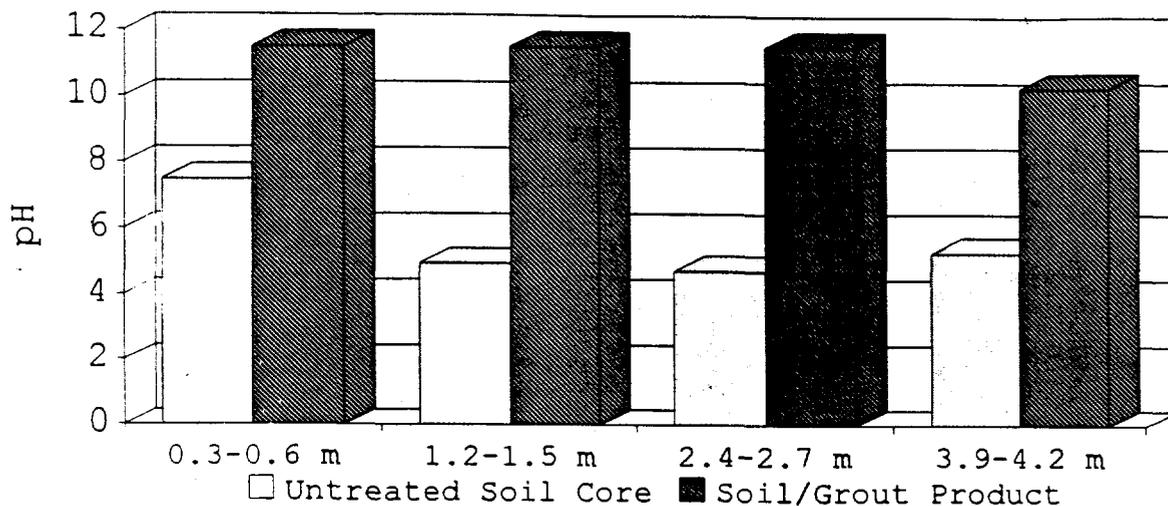
<sup>b</sup> All values represent at a minimum, the average of 6 replicate analyses. Data reported at 25°C per ASTM D5084. Permeant fluid: 0.005M CaSO<sub>4</sub>.

<sup>c</sup> Average of analyses of two samples.

<sup>d</sup> Sample taken at the 1-m depth.

Chemical Characteristics--The pH of the soil/grout product, a qualitative indicator of cement content, (10.3-11.5) was significantly higher than that of the untreated soil (5.3-7.5), presumably due to the high alkalinity of the cement-based grout (Fig. 8). High pH values such as those encountered here should reduce the possibility of excessive leaching induced by acid attack. Calcium Hydroxide [Ca(OH)<sub>2</sub>], which highly influences the pH of the matrix, is a byproduct of the cementitious reactions. The grout used in the S/S demonstration had a pH of 12.5. Such a high pH is indicative of cement rich grout that is very porous and has low durability due to the high production of Ca(OH)<sub>2</sub>. The flyash present in the grout serves to reduce the amount of cement needed, to react with the Ca(OH)<sub>2</sub> produced, and to physically fill in the porous structure of the grout matrix. All of the above

should reduce the pH of the grout to around 10. Thus, The pH values of the soil/grout product are slightly higher, indicating that the solidified matrix may be somewhat porous with low durability (as evidenced by the compressive strength data). The lower pH obtained for the 3.9-4.2 m sample (Fig. 8) is more likely due to an overall reduced cement content in this depth region (i.e., inadequate mixing with depth), rather than the possibility of having an abundance of flyash in this region.

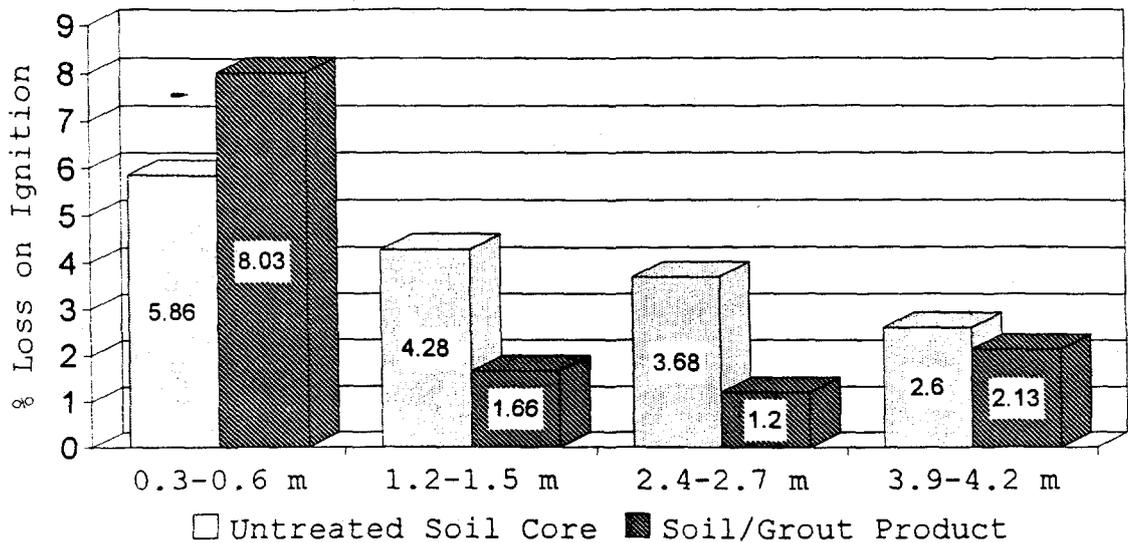


PRE-SB-SB15 and PST-SB-Z2h, respectively in Fig. 2.

FIG. 8--Soil pH with depth before and after in situ solidification.

Total volatile solids analyses (Percent loss on ignition) were performed to provide insight into the distribution of activated carbon within the grouted soil (Fig. 9). Based upon the same assumptions used in the mass balance computations (see Table 3), the soil/grout mixture within each column should contain about 2.7 wt% carbon if the grout were uniformly mixed throughout the soil column. The values obtained from the analyses (which measure all volatile components, not just activated carbon) ranged from 1.2 to 8.0 wt% loss on ignition. As expected, the region with the highest fraction of combustible material was the 0.3-0.6 m depth interval, where excess quantities of grout were discharged on the soil surface prior to auger penetration. For the remaining sample intervals, the soil/grout product yielded lower % wt losses than the corresponding samples from the untreated soil core. These discrepancies could have been due to spatial variability, or simply to analytical error(s) associated with differences in the two matrices evaluated.

Samples of the soil/grout core from column Z2 were also subjected to the TCLP test. The core was subsampled at the 1.2-1.5 m and 2.4-2.7 m depth intervals (same location as those TCLP samples evaluated immediately after grouting) Regulated VOCs were not detected, except for TCE from the 1.5-m depth sample. However, the reported TCE leach value of 0.08 mg/L was well below the 0.5 mg/L regulatory limit set for the compound. Similarly, the concentrations determined for uranium and the RCRA regulated metals were also significantly below EPA regulatory limits. Unfortunately, only one TCLP analysis was performed on the



PRE-SB-SB15 and PST-SB-Z2h, respectively in Fig. 2.

FIG. 9--Volatile matter content with depth before and after in situ solidification.

untreated soil (1.2-1.5 m depth) with an appreciable quantity of 1,1,1-trichloroethane present in the extract. Due to the insufficient number of TCLP samples (and depth intervals) collected for the untreated soil, predictions on the S/S product's leachability were difficult.

#### CONCLUSIONS AND RECOMMENDATIONS

There are many factors to consider in evaluating the effectiveness of in situ S/S, including operational factors (e.g., equipment requirements, processing rate, flexibility, reliability), contaminant capture and immobilization, and homogeneity and stability of the solidified media. Cost is also an important factor. Operationally, in situ S/S of dense clay soil was not straight-forward. Several shakedown tests were required due to both mechanical problems (e.g., a clogged pre-screen on the grout pump, and a broken hydraulic line) and difficulties in finding a proper grout formulation amenable to the dense clay soil at the site. Once corrective actions were made for these problems, difficulties in controlling the grout flow rate into the MecTool and the creation of the large berms were the two largest operational problems observed during the actual process demonstration on May 22, 1992. The flow meter on the grout pump was not calibrated for the grout formula used, resulting in a very wide range of grout flow rates. This lack of consistent control may have contributed to some of the heterogeneity/variability observed in the soil/grout product. The berms, created by mixing and grout injection, had to be compacted before the existing geomembrane was put back into place following the demonstration. Such operational problems encountered in the field illustrate the need to optimize the grouting process, the grout formulation in particular, for the specific subsurface deposit to be treated.

The rate at which in situ S/S proceeded is an important parameter to consider in assessing the overall effectiveness of the process.

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Including equipment set-up and disassembly time, the S/S demonstration was capable of mixing four soil columns to a depth of 4.6 m in one 10 h working day. Taking the overlap into account, the three 3.1 m diameter soil columns encompassed approximately 20 m<sup>2</sup> of surface area. In a comparable study of in situ S/S of PCB contaminated soil, a processing rate of 1.1 m<sup>3</sup> soil/day was achieved [2]. During the X-231B demonstration, however, the S/S demonstration proceeded at a rate of 3.4 m<sup>3</sup> soil/day. The increased processing rate observed here is mostly due to the use of a larger diameter mixing auger. In these two studies, the equivalency of mixing and the homogeneity of the solidified monoliths is uncertain.

Data obtained from core sampling 15 months after the demonstration suggest that the soil/grout matrix possessed the highest degree of homogeneity and overall integrity from 0-3.0 m. Compressive strength of the core sample decreased with depth. Elemental analyses and loss on ignition experiments conducted on the soil/grout core also suggest that the 0-0.6 m depth interval possessed the largest quantity of grout. The two (2) hydraulic conductivity analyses performed suggest that the permeability of the soil/grout product is fairly uniform with depth, although there was an increase in hydraulic conductivity was observed between the untreated soil and the soil/grout product.

Finally, there was an attempt to compare the total VOC concentrations from the pre-treatment sampling with the other VOC quantities obtained during and immediately after the S/S demonstration (mass balance) in order to evaluate the effectiveness of in situ S/S in terms of treatment efficiency. Data from the FID response curves, post-treatment samples, and the berm sample analyses account for only 8 wt% of the initial solidification pre-treatment VOCs. Since it is unlikely that significant VOC quantities escaped from the off-gas shroud or diffused into the tight, clay soil surrounding the mixed regions, it is believed that the remaining VOCs not detected in the test samples (i.e., ≈ 90 wt% initial VOC mass) was stabilized and captured in the soil/grout matrix. It is recommended that additional work be focused upon delivering larger quantities of S/S agents deeper into the subsurface. Although the samples obtained from the post-treatment product passed all TCLP criteria, it is recommended that future studies also include a greater number of TCLP analyses of the untreated media so that better comparisons of leachability can be made.

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