

TECHNOLOGY EVALUATION REPORT:

**SILICATE TECHNOLOGY CORPORATION, SOLIDIFICATION/STABILIZATION OF
PCP AND INORGANIC CONTAMINANTS IN SOILS; SELMA, CA**

**NATIONAL RISK MANAGEMENT RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OH 45268**

NOTICE

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FOREWORD

The Superfund Innovative Technology Evaluation (SITE) program was authorized by the 1986 Superfund Amendments and Reauthorization Act (SARA). The program is a joint effort between EPA's Office of Research and Development (ORD) and Office of Solid Waste and Emergency Response (OSWER). The purpose of the program is to assist the development of hazardous waste treatment technologies necessary to implement new cleanup standards that require greater reliance on permanent remedies. This assistance is accomplished through technology demonstrations that are designed to provide engineering and cost data on selected technologies.

This project was a field demonstration under the SITE program and was designed to analyze the Silicate Technology Corporation solidification/stabilization technology. The technology demonstration took place at a lumber treating facility in Selma, California. The demonstration effort was directed to obtain information on the performance and cost of the technology and to assess its use at this and other Superfund or uncontrolled hazardous waste sites. Documentation related to this technology consists of two reports: (1) an Applications Analysis Report that evaluates and discusses the potential applicability of the STC treatment process; and (2) this Technology Evaluation Report that describes field activities and laboratory results.

Copies of this report can be purchased from the National Technical Information Service, Ravensworth Building, Springfield, Virginia 22161, (703) 487-4650. Reference copies will be available at EPA libraries in the Hazardous Waste Collection.

E. Timothy Oppelt, Director
Risk Reduction Engineering Laboratory

ABSTRACT

This Technology Evaluation Report evaluates the solidification/stabilization process of Silicate Technology Corporation (STC) for the on-site treatment of contaminated soil. The STC immobilization technology uses a proprietary product (FMS Silicate) to chemically stabilize and microencapsulate organic and inorganic wastes, and to physically solidify contaminated soils.

The STC demonstration was conducted under EPA's Superfund Innovative Technology Evaluation (SITE) Program in November, 1990, at the Selma Pressure Treating (SPT) wood preserving site in Selma, California. The SPT site was contaminated with both organics, predominantly pentachlorophenol (PCP), and inorganics, mainly arsenic, chromium, and copper. Extensive sampling and analyses were performed on the waste both before and after treatment to compare physical, chemical, and leaching characteristics of raw and treated wastes. STC's contaminated soil treatment process was evaluated based on contaminant mobility, measured by numerous leaching tests; and structural integrity of the solidified material, measured by physical, engineering, and morphological examinations. This report provides a comprehensive description of the SITE demonstration and its results including a description of data collection activities, testing procedures, and quality assurance and quality control (QA/QC) results.

Conclusions resulting from this SITE demonstration evaluation are that: (1) the STC process chemically stabilized contaminated soils containing both inorganic and semivolatile organic contaminants; (2) PCP concentrations were reduced by 91 to 97 percent as determined by total waste analysis (SW-846, Method 8270); (3) arsenic and copper were immobilized based on various leach-test criteria; (4) chromium concentrations were very low prior to and after treatment, but showed slight to moderate increase in leachability after treatment; (5) PCP concentrations remained above California state regulatory threshold levels after treatment; and metal concentrations in the treated waste were not consistently below California state regulatory thresholds; (6) the short-term physical stability of the treated waste was good, with unconfined compressive strengths well above landfill solidification standards; (7) due to the addition of reagents, treatment resulted in a volume increase of 59 to 75 percent (68 percent average) and a slight bulk density increase; (8) six-month monitoring showed increased concentrations of the contaminants released from the treated waste; (9) eighteen-month monitoring showed improved percent reductions for arsenic and PCP relative to 6-month cured sample test results; (10) thirty-two-month monitoring showed comparable percent reductions for PCP relative to 18-month results, but arsenic leachability more closely resembled 6-month results; (11) chromium and copper showed slight to moderate increases in leachate concentrations over time, although copper concentrations at 32 months more closely resembled 28-day results than either 6-month or 18-month results; and (12) unconfined compressive strengths increased an average of 145 and 245 percent for 32-month and 18-month samples, respectively, relative to 28-day values.

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ABBREVIATIONS

ACI	American Concrete Institute
AEA	Atomic Energy Act
amp	ampere
ANS	American Nuclear Society
ARAR	applicable or relevant and appropriate requirement
ASA	American Society of Agronomy
ASTM	American Society for Testing and Materials
BSE	backscattered emissions
CAA	Clean Air Act
CEPA	California Environmental Protection Agency
CALWET	California Waste Extraction Test
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
cm	centimeter
CRWQCB	California Regional Water Quality Control Board
CWA	Clean Water Act
°C	degree Celsius
DOE	Department of Energy
DOT	Department of Transportation
EDX	energy dispersive X-ray
Eh	oxidation/reduction potential
EP	Extraction Procedure
EPA	Environmental Protection Agency
ESBL	Engineering-Science, Inc. Berkeley Laboratory
FIT	Field Investigation Team
ft	foot
FRTL	Federal Regulatory Threshold Limit
FTIR	Fourier transform infrared spectroscopy
g	gram
gal	gallon
gph	gallons per hour
HCP	Hazard Communication Program
HDPE	high-density polyethylene
hr	hour
HRS	Hazard Ranking System
HSL	Hazardous Substance List
HSWA	Hazardous and Solid Waste Amendments
kg	kilogram
kV	kilovolt
L	liter
lb	pound
LDR	Land Disposal Restriction
LI	leachability index
mA	milliampere

ABBREVIATIONS (continued)

MDL	method detection limit
meq	milliequivalent
mg	milligram
mo	month
mm	millimeter
MSDS	Material Safety Data Sheet
mV	millivolt
NC	not calculable
ND	not detected
NIOSH	National Institute of Occupational Safety and Health
NPDES	National Pollutant Discharge Elimination System
NPL	National Priorities List
NRC	Nuclear Regulatory Commission
ORD	Office of Research and Development
OSHA	Occupational Safety and Health Administration
OSWER	Office of Solid Waste and Emergency Response
PAH	Polycyclic aromatic hydrocarbon
PCB	Polychlorinated biphenyl
PCDD	Polychlorinated dibenzo-p-dioxin
PCDF	Polychlorinated dibenzofuran
PCP	Pentachlorophenol
POTW	Publicly Owned Treatment Works
PPE	personal protective equipment
ppb	parts per billion
ppm	parts per million
psi	pounds per square inch
QA/QC	Quality Assurance/Quality Control
QAPjP	Quality Assurance Project Plan
RCRA	Resource Conservation and Recovery Act
RFP	Request For Proposal
RI/FS	Remedial Investigation/Feasibility Study
RM	reagent mixture
ROD	Record of Decision
RPD	relative percent difference
SARA	Superfund Amendments and Reauthorization Act
SDWA	Safe Drinking Water Act
sec	second
SEM	Scanning Electron Microscopy
SI	International System of Units
SITE	Superfund Innovative Technology Evaluation
SPT	Selma Pressure Treating
STC	Silicate Technology Corporation
STLC	Solubility Threshold Limit Concentration
TCLP	Toxicity Characteristic Leaching Procedure
TCP	Tetrachlorophenol
TER	Technology Evaluation Report

ABBREVIATIONS (continued)

TMSWC	Test Methods for Solidified Waste Characterization
TPH	Total Petroleum Hydrocarbons
TSCA	Toxic Substances Control Act
TSDF	treatment, storage, and disposal facility
TTLC	Total Threshold Limit Concentration
TWA	Total Waste Analysis
UCS	unconfined compressive strength
UIC	Underground Injection Control
VER	volume expansion ratio
VOC	volatile organic compound
wk	week
yr	year
XRD	X-ray diffractometry

CONVERSION OF U.S. CUSTOMARY UNITS TO SI UNITS

Length

inches	x	25.4	=	millimeters
inches	x	2.54	=	centimeters
inches	x	0.0254	=	meters
feet	x	0.3048	=	meters

Volume

gallons	x	3.785	=	liters
cubic yards	x	0.7646	=	cubic meters

Weight

pounds	x	0.4536	=	kilograms
short tons	x	0.9072	=	metric tons

Temperature

$\frac{5}{9}$	x	(° Fahrenheit - 32)	=	° Celsius
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Note: 1000 liters = 1 cubic meter

1000 kilograms = 1 metric ton

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1.0 EXECUTIVE SUMMARY

1.1 INTRODUCTION

The Silicate Technology Corporation (STC) immobilization technology is a solidification/stabilization treatment process that was evaluated under the U.S. Environmental Protection Agency's (EPA) Superfund Innovative Technology Evaluation (SITE) Program. This immobilization technology is designed to treat organic and inorganic contaminants, thereby reducing the leaching potential of these constituents in contaminated soils and sludges. For purposes of this report, "solidification" refers to the physical consolidation of contaminated soil into a hard, rock-like material. "Stabilization" refers to the chemical immobilization of hazardous contaminants. Proprietary silicate mineral reagents bind the contaminants within a layered alumino-silicate mineral structure prior to encapsulating the waste in a cement-like material, thus producing a high-strength, leach-resistant monolith.

The STC technology demonstration was performed at the Selma Pressure Treating (SPT) site in Selma, California during November, 1990. In general, the STC SITE demonstration had the following four objectives:

- Assess the technology's ability to chemically stabilize both organic and inorganic contaminants.
- Assess the structural characteristics of the treated waste and the effectiveness of solidification/stabilization over a 3-year period.
- Determine the volume and density increases resulting from the treatment process.
- Develop information required to estimate the capital and operating costs for the treatment system.

The purpose of this report is to present technical information from the SITE demonstration that is useful for implementing the STC immobilization technology at Superfund, Resource Conservation and Recovery Act (RCRA), and uncontrolled hazardous waste sites. Section 2 presents an introduction and overview of the SITE program and describes the purpose of the Technology Evaluation Report (TER). Section 3 describes the STC technology, equipment necessary for

implementation of the technology, and relevant factors affecting the STC technology. Section 4 describes the SPT site contamination characteristics and gives a background discussion of the previous studies at the site. Section 5 gives a detailed account of the demonstration procedures, including an overview of the quality assurance/quality control plan. Section 6 discusses the analytical results for the various leaching, chemical, and physical tests used to evaluate the STC technology and includes a report on the QA results. Section 7 presents the demonstration costs for both the SITE contractors and the developer.

1.2 OVERVIEW OF THE SITE DEMONSTRATION

The SPT site was selected to evaluate the effectiveness of STC's immobilization technology for soils contaminated with both organic and inorganic constituents. The waste material was reported to contain pentachlorophenol (PCP; 1,900 to 8,400 mg/kg), arsenic (375 to 1,900 mg/kg), chromium (1,900 mg/kg), and copper (1,500 mg/kg). In addition, oil and grease levels ranged from 10,000 to 20,000 mg/kg. Prior to treatment, soil pH was slightly acid to neutral and moisture content ranged from 4 to 6 percent (CDM, 1989 and U.S. EPA, 1990a).

During the SITE demonstration, approximately 16 tons of contaminated soil material were treated. STC's proprietary alumino-silicate compounds were added to the waste to chemically fix, and thereby immobilize, heavy metals and semivolatile organic constituents. Addition of a silicate solidifying agent microencapsulated the adsorbed contaminants, thereby producing an additional physical barrier to leaching.

The STC SITE demonstration required 6 days to complete once all of the treatment equipment was mobilized. Initial processing consisted of treating clean sand with the proprietary reagent mixture used by STC. On each of the following 5 days, one 2.5-cubic-yard batch of contaminated soil was treated. Surface hardpan and sand from an unlined, dry waste-disposal pond was collected to a depth of 2 to 3 feet and thoroughly mixed prior to the addition of STC reagents and water. Significant inhomogeneity in the treated waste from Batch 2 resulted in pretreatment screening of the remaining batches (3 through 5), and led to the exclusion of Batch 2 from additional analytical evaluations. Detailed descriptions of waste samples collected from each of the batches are included in section 6.5. In general, Batch 1 consisted of contaminated waste from a soil horizon approximately 1 foot deep,

while Batch 2 consisted of a shallower hardpan surface horizon (down to 8 inches). Batches 3, 4 and 5 were composite blends of the first two soil horizons.

Samples of raw and treated waste were submitted for chemical and physical characterization after a 28-day cure period. Analytical testing was targeted towards selected inorganic constituents (arsenic, chromium, and copper) and organic contaminants (primarily PCP), using various leach tests plus total waste analysis (TWA) extraction procedures. EPA SW-846 Methods 8240 and 8270 were used for the chemical analyses of volatile and semivolatile organic compounds, respectively. Metals were analyzed using EPA SW-846 Methods 3010, 3020, 3050, 7060, 7421, 7740, 7841, 7471, and 7470 (U.S. EPA, 1986b). Leach tests included the EPA SW-846 Method 1311 Toxicity Characteristic Leaching Procedure (TCLP); a modified TCLP Distilled Water test; a TCLP-Cage test; the California Waste Extraction Test (CALWET) as described by the California Health and Safety Code, Section 66700; and a modified version of the American Nuclear Society (ANS) 16.1 leaching procedure (ANS, 1986). Additional chemical and physical characterization of the raw and/or treated waste included pH, Eh, loss on ignition, neutralization potential, particle size analysis, bulk density, permeability, unconfined compressive strength, wet/dry and freeze/thaw analyses, petrographic analysis, X-ray diffraction, scanning electron microscopy, and Fourier transform infrared spectroscopy (U.S. EPA, 1992).

1.3 SUMMARY OF SITE DEMONSTRATION RESULTS

The sampling and analysis conducted on the raw and treated wastes for the STC SITE demonstration can be grouped into four general classes: leaching tests, chemical tests, physical tests, and petrographic examinations. To constitute treatment under Superfund, immobilization (i.e., solidification/stabilization) technologies must chemically limit the solubility or mobility of the contaminants. Specifically, before a technology can be selected as a treatment alternative, EPA guidance suggests that an immobilization technology demonstrate a significant reduction (i.e., a 90 to 99 percent reduction) in the mobility of chemical constituents of concern (OSWER Directive No. 9200.5-220). The reduction in mobility is evaluated using the TCLP for inorganics and TWA for volatile and semivolatile organics. In addition, federal and state regulatory thresholds must be met to allow for legal disposal as nonhazardous wastes either on site or off site. The following conclusions about the effectiveness of STC's solidification/stabilization treatment process are based on results of

analytical data and general observations from the SITE demonstration as discussed in Section 6 of this report.

PCP (Targeted for treatment):

- TWA extract concentrations of PCP were reduced 91 to 97 percent.
- TWA extract concentrations of PCP were well above the California state regulatory total threshold limit concentration of 17 mg/kg prior to and after treatment.
- Percent reductions of TCLP leachate concentrations of PCP varied from negative percentages to greater than 81 percent.
- TCLP-Distilled Water leachate concentrations of PCP were reduced 80 to 97 percent.
- PCP concentrations were well below the federal regulatory threshold TCLP concentration of 100 mg/L prior to and after treatment.
- CALWET leachate concentrations of PCP were above California's solubility threshold limit concentration of 1.7 mg/L prior to and after treatment.
- Stabilization of semivolatile organic compounds (exclusive of PCP) and volatile organic compounds could not be evaluated due to the low concentrations of these analytes in the waste.

Arsenic (Targeted for treatment):

- TCLP leachate concentrations of arsenic were reduced 35 to 92 percent.
- TCLP-Distilled Water leachate concentrations of arsenic were reduced 98 percent or more.
- Arsenic concentrations were below the federal regulatory threshold TCLP concentration of 5.0 mg/L prior to and after treatment.
- CALWET leachate concentrations of arsenic were both over and under the California state regulatory solubility threshold limit concentration of 5.0 mg/L after treatment.
- TWA extract concentrations of arsenic were both above and below the California state regulatory total threshold limit concentration of 500 mg/kg prior to and after treatment.

Chromium (Not targeted for treatment):

- TCLP leachate concentrations of chromium were increased as a result of treatment.

- Percent reductions of TCLP-Distilled Water leachate concentrations of chromium varied from 42 to 54 percent.
- Chromium concentrations were below the federal regulatory threshold TCLP concentration of 5.0 mg/L prior to and after treatment.
- CALWET leachate concentrations of chromium were well below the California state regulatory solubility threshold limit concentration of 560 mg/L prior to and after treatment.
- TWA extract concentrations of chromium were below the California state regulatory total threshold limit concentration of 2,500 mg/kg prior to and after treatment.

Copper (Not targeted for treatment):

- TCLP leachate concentrations of copper were reduced 90 to 99 percent.
- TCLP-Distilled Water leachate concentrations of copper were reduced 86 to 90 percent.
- CALWET leachate concentrations of copper were both over and under California's solubility threshold limit concentration of 25 mg/L prior to and after treatment.
- TWA extract concentrations of copper were below the California state regulatory total threshold limit concentration of 2,500 mg/kg prior to and after treatment.

Long-Term Results:

- TCLP extracts for metals and TWA extracts for PCP of the 6-month cured samples showed increased concentrations of contaminants released from the treated waste.
- Analyses for the 18-month cured samples showed improved percent reductions relative to the 6-month cured sample test results, averaging 88 percent reduction for arsenic, and 96 percent reduction for PCP.
- Analyses for the 32-month cured samples showed TWA extract concentrations of PCP to be comparable to 18-month results. TCLP extract concentrations of arsenic at 32 months were comparable to 6-month results.
- Chromium and copper concentrations showed slight to moderate increases in the TCLP extracts over time, although copper concentrations at 32 months more closely resembled 28-day results than extracts at either 6 months or 18 months.

Physical Properties:

- Unconfined compressive strength (UCS) of the treated wastes was moderately high after 28 days, averaging 260 to 350 pounds per square inch (psi). Eighteen-month UCS tests showed an average 245 percent increase in physical strength with time.

UCS results from 32 months showed an average 27 percent decrease in strength from the 18-month results, but were still greater on average than the 28-day results by a factor of 2.5.

- Permeability of the treated waste was low ($< 1.7 \times 10^{-7}$ cm/sec).
- Relative cumulative weight losses from 12 wet/dry and 12 freeze/thaw cycles were less than 1 percent.
- Due to addition of reagents, treatment of the wastes resulted in volume increases ranging from 59 to 75 percent (68 percent average), with slight increase in bulk density.
- Petrographic and scanning electron microscopy examinations indicated good binder-to-aggregate bonding. Constituents comprising the reagent mix binder included calcium hydroxide, glass, portland cement, and black pigment. Soil constituents were predominantly quartz and feldspar with minor hornblende and trace mica.

Treatment Technology:

- No equipment-related problems occurred during the 6-day technology demonstration.
- The process equipment used during the demonstration was capable of mixing all components, including the waste material, into a homogeneous, solidified product, provided that pretreatment screening or size reduction of surface hardpan material was conducted.

2.0 INTRODUCTION

This section provides background information on the Superfund Innovative Technology Evaluation (SITE) program and the purpose of this report. In addition, this section also presents the objectives of the STC SITE demonstration program.

2.1 SITE PROGRAM

The Superfund Amendments and Reauthorization Act (SARA) of 1986 (Section 209(b)) amends Title III of the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) by adding Section 311. Section 311 directs the U.S. Environmental Protection Agency (EPA) to establish an "Alternative or Innovative Treatment Technology Research and Demonstration Program." In response to the SARA directive, EPA has established a formal program to accelerate the development, demonstration, and use of new or innovative treatment technologies. This program is called the SITE program, and is administered by the Office of Research and Development (ORD).

The primary purpose of the SITE program is to enhance the development and demonstration of innovative technologies applicable to Superfund sites, thereby establishing the commercial availability of these technologies.

The SITE program has four objectives:

- Identify and remove impediments to the development and commercial use of alternative technologies.
- Conduct a demonstration program of the more promising innovative technologies to establish reliable performance and cost information for site characterization and cleanup decision making.
- Develop procedures and policies that encourage selection of available alternative treatment remedies at Superfund sites.
- Structure a development program that nurtures emerging technologies.

One of the most important components of the SITE Program is the Demonstration Program, through which EPA evaluates field- or pilot-scale tested technologies that can be scaled up for commercial use. The Demonstration Program is the primary focus of the SITE Program because the innovative alternative technologies evaluated are close to being available for remediation of Superfund sites. The main objective of the Demonstration Program is to develop performance, engineering, and cost information for innovative technologies. With this information, potential users can make informed decisions on whether to use these technologies to remediate hazardous waste sites. Specifically, potential users can use this information to compare the technology's effectiveness and cost to other alternatives and make sound judgments regarding applicability of the technology for a specific use.

Each year the EPA solicits proposals to demonstrate innovative technologies for permanent, long-term site cleanups. The most promising technologies are chosen for participation in the SITE demonstration program. ORD, and EPA regional personnel match these technologies with a list of potentially appropriate sites.

The results of the demonstration identify possible limitations of the technology, the potential need for pre- and post-processing of wastes, the types of wastes and media to which the process can be applied, the potential operating problems, and the approximate capital and operating costs. The demonstrations also permit evaluation of long-term risks. Demonstrations usually occur at Superfund sites or under conditions that duplicate or closely simulate actual conditions found at Superfund sites to ensure the reliability of the information collected and acceptability of the data to users.

Developers are responsible for demonstrating their innovative systems at selected sites and are expected to pay the costs to transport equipment to the site, operate the equipment on site during the demonstration, and remove the equipment from the site. EPA is responsible for project planning, sampling and analysis, data quality assurance and quality control, report preparation, and information dissemination.

2.2

PURPOSE OF THE TECHNOLOGY EVALUATION REPORT

The Technology Evaluation Report (TER) is one of two reports resulting from the SITE demonstration. The TER provides a comprehensive description of the demonstration and its results, including a description of data collection activities, testing procedures, and QA/QC results. This report is intended for technical professionals making detailed evaluations of the technology for a specific situation. The information contained in the TER is used to produce conceptual designs in sufficient detail to prepare preliminary cost estimates for the demonstrated technology.

The second report resulting from the SITE demonstration is the Applications Analysis Report (AAR). The AAR presents estimates of the costs of implementing a technology and describes its Superfund applications based on available data. The AAR is intended to be used by decisionmakers responsible for implementing remedial actions and to help them determine whether a technology has merit as an option for a particular cleanup situation.

2.3

SITE DEMONSTRATION OBJECTIVES

The overall goal of the Silicate Technology Corporation (STC) demonstration at the Selma Pressure Treating (SPT) site was to evaluate the effectiveness of the STC immobilization technology as a long-term remedial measure at Superfund, Resource Conservation and Recovery Act (RCRA), and uncontrolled hazardous waste sites. The SPT site was selected for the demonstration based on its waste characteristics, the results of treatability testing, and site logistical considerations. STC's technology is designed for sites with moderate-to-high concentrations of both organic and inorganic contaminants, including polycyclic aromatic hydrocarbons (PAHs) and heavy metals as were reported by CDM at the SPT site (CDM, 1988a and b). *The primary objective of this demonstration was to determine if the STC technology could reduce the potential leachability and mobility of contaminants as measured by total waste analysis (TWA) for organics and the Toxicity Characteristic Leaching Procedure (TCLP) for inorganics. In particular, the principal contaminants for assessing the STC technology were pentachlorophenol (PCP) and arsenic.* Other objectives of this demonstration were to:

- Determine if the STC technology could reduce the leachability of contaminants as measured by other leaching methods.
- Determine if the STC technology could reduce leachate concentrations of PCP and metals below applicable regulatory limits to allow for legal disposal as a nonhazardous waste.
- Determine the homogeneity of mixing and structural characteristics of the waste treated by the STC technology.
- Determine the volume and density increase of the waste treated by the STC technology due to added reagents.
- Determine if the STC technology could treat contaminated soils to produce a monolithic block that would resist the effects of weathering.
- Determine whether the treated, solidified waste could maintain its structural properties and stabilization effectiveness over a 3-year period.
- Develop capital and operating cost models for the technology that can be used reliably in the Superfund and RCRA decision-making process.

3.0 TECHNOLOGY DESCRIPTION

STC's treatment process uses silicate compounds that chemically stabilize organic and inorganic constituents in contaminated soils and sludges. Proprietary silicate reagents adsorb organic and inorganic contaminants prior to solidifying the waste with a cementitious material, resulting in a high-strength, leach-resistant monolith. Treatability tests and site investigations were conducted to determine the necessary types and dosage of reagents according to the waste characteristics. The following section discusses the general STC treatment process chemistry, major process equipment needed for the STC technology, and major factors that can affect the technology. All information presented is provided by STC. Specific procedures used in the SITE demonstration are detailed in Section 5.

3.1 PROCESS CHEMISTRY

STC has developed two groups of reagents: SOILSORB HM for treating wastes with inorganic constituents and SOILSORB HC for treating wastes with organic constituents. These two groups of reagents can be combined to treat wastes containing both organic and inorganic contaminants. The specific proprietary reagents P-4 and P-27 were combined to treat contaminated soils from the SPT site.

Stabilization by the STC process of wastes with inorganic constituents involves silicate-forming reactions resulting in the incorporation of heavy metal ions into the crystal lattice structure of a highly insoluble calcium-alumino-silicate compound. The reactions effectively immobilize the contaminants, thereby reducing the potential for leaching. A silicate solidifying agent microencapsulates the alumino-silicate compound to form another physical barrier to leaching. The result is a very stable compound analogous to common rock-forming silicate minerals.

STC's technology for treating organic wastes uses a three-step process in which organic compounds in the waste are sequestered by a modified alumino-silicate mineral. The silicate is surface-modified with organic compounds, creating a layered structure that consists of organic layers sandwiched between the alumino-silicate layers. Upon mixing with the organic wastes, this modified silicate adsorbs organic contaminants into the layers of the organically surface-modified alumino-

silicate compound through a partitioning reaction. STC claims that the organic layers of the modified silicate can adsorb as much as 20 times their own weight of organic constituents. In addition, STC also claims that the organics adsorbed cannot be physically squeezed out of the layered silicate structure.

The first step of the contaminant immobilization process involves partitioning similar to a liquid/liquid extraction. If a waste containing a polynuclear aromatic compound such as anthracene is mixed or combined with a water-immiscible oil, the anthracene will migrate into the oil phase and remain there. STC's technology is based on this concept, except that it uses a solid organic phase instead of oil. This partitioning follows basic laws of physical chemistry and can in general terms be predicted for any organic compound based on its water solubility.

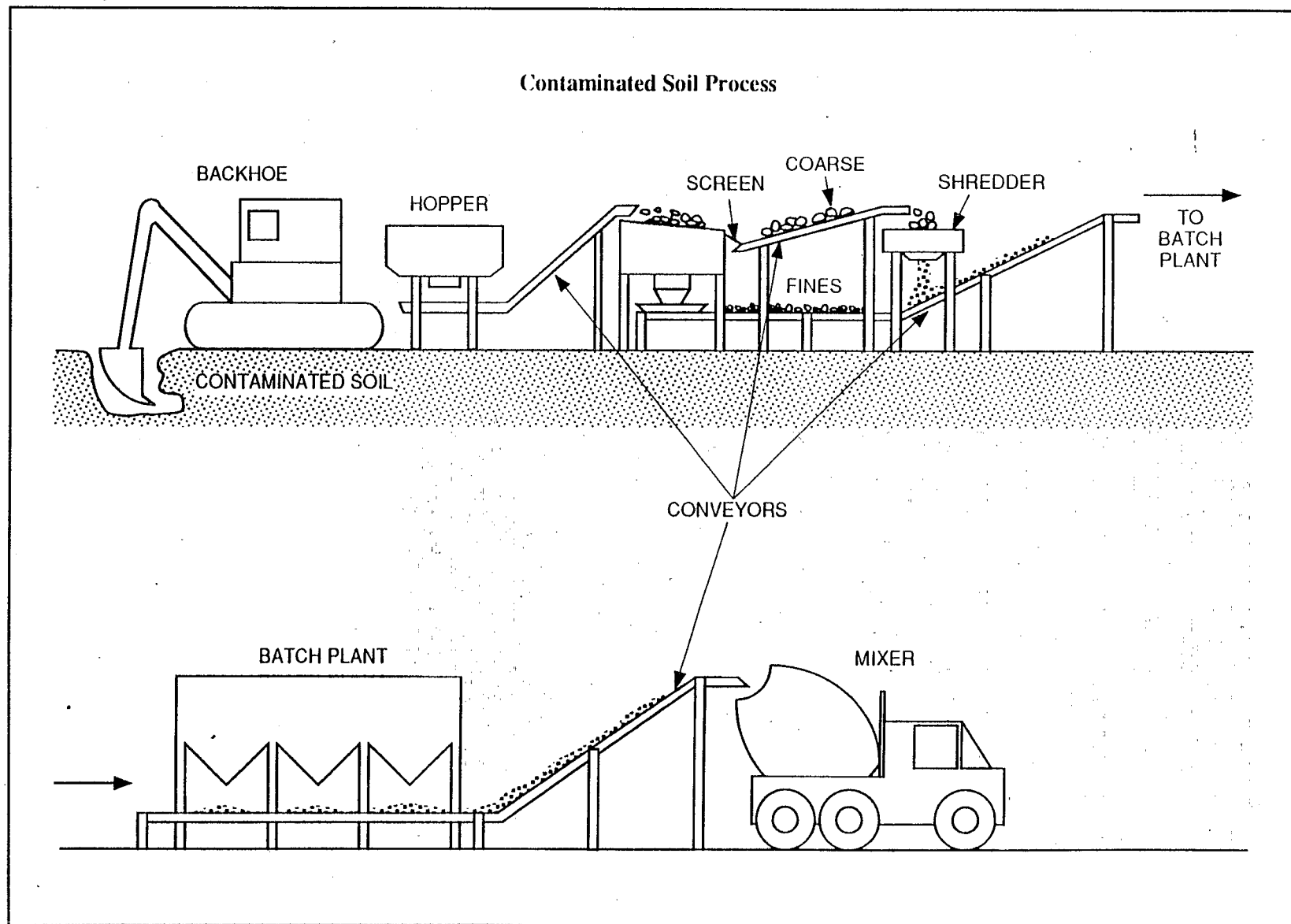
The second step of the stabilization process involves the morphology of the alumino-silicate structure. As the organic waste constituents partition to the organic layers of the surface-modified silicate, the layered alumino-silicate plates tend to bond with the surface of the waste, thereby creating a physical barrier and thus reducing leachability.

Finally, the third step of the stabilization process is the addition of STC's proprietary silicate solidifying agent, which microencapsulates the layered alumino-silicate structure and bonds the solidifying agent to the exposed layered-silicate surfaces. This microencapsulation of the adsorbed organics further reduces leachability by forming another physical barrier to leaching. STC claims that the alumino-silicates used for the organic partitioning reaction and the silicates used for the microencapsulation reaction can be shown to be thermodynamically stable compounds, analogous to common, rock-forming silicate minerals.

3.2 TREATMENT PROCESS

Treatment of contaminated soil typically begins with the separation of coarse material from fine material in a mechanical separator (Figure 3-1). This separation is accomplished by using a shaker screen to isolate the coarse material greater than 3/8 inch in diameter. This coarse material is sent through a shredder or crusher, which reduces it to the size required for the STC technology.

Figure 3-1. Schematic Diagram for STC Treatment Process



The screened waste is loaded into a batch plant where it is weighed and the appropriate amount of the STC proprietary reagents -- SOILSORB: P-4 and P-27 -- as determined during treatability testing are added. This mixture is conveyed to a pug mill mixer (or equivalent, such as a ready-mix cement truck) where water is added and the mixture is thoroughly blended. When this technology is used to treat sludges, the waste is placed directly into the pug mill for addition of reagents and mixing. Reagents are then added in amounts that can be adjusted according to variations in organic and inorganic contaminant concentrations determined during treatability testing. The mixing process continues until the operator determines that the materials are thoroughly homogenized, up to approximately 60 minutes per batch. The treated material is then placed in confining pits for on-site curing, or cast into molds for curing and subsequent transport and disposal off site.

Hardware for the treatment process includes processing and materials-handling equipment. With the exception of STC's liquid reagent metering equipment, conventional construction equipment readily available for purchase or rental in most areas can be used. Such equipment typically would have the capacity to treat up to 40 cubic yards of contaminated soil per day; however, only 2.5 cubic yards per day were processed during the demonstration.

Process equipment for soil treatment using STC's technology includes the following:

- Pretreatment screen -- Pretreatment screening is normally accomplished with a shaker screen to separate fine material (< 3/8-inch diameter) from coarse material (> 3/8-inch diameter). Pretreatment screening down to 0.04 - 0.08 inch (1-2 mm) diameter was required for the STC demonstration since a crusher was not used, and it was necessary to ensure that individual aggregates of untreated waste did not bias the chemical analyses.
- Crusher or shredder -- A crusher or shredder is used to further reduce waste aggregate size prior to mixing, if necessary.
- Weight conveyor -- The weight conveyor is used to weigh and transfer screened material to the pug mill.
- Pug mill -- A pug mill, cement-mixer, or other conventional construction equipment can be used as a mixing vessel.
- Liquid reagent metering equipment -- Liquid reagent metering is accomplished with STC's mobile liquid meter, which is mounted on a 20-foot bed trailer. This equipment includes two 500-gallon tanks.

Materials handling equipment for soil treatment includes the following:

- Front-end loader/backhoe for excavation and transport of waste material on site.
- All-terrain forklift for moving 3-ton forms filled with treated waste.

3.3 FACTORS AFFECTING THE PERFORMANCE OF THE TECHNOLOGY

Several factors can influence the performance and cost effectiveness of the STC solidification/stabilization technology, including (1) waste characteristics, (2) volume/density increase, (3) operating conditions, and (4) climate and curing conditions. The following subsections discuss these factors in detail.

3.3.1 Waste Characteristics

The ability of the STC solidification/stabilization technology to solidify and chemically stabilize organic and inorganic contaminants in a waste depends on the characteristics of the waste. The characteristics that may affect the performance of the STC technology include clay content, coal and lignite content, moisture content, oil and grease content, pH of the waste, volatile organic concentrations, and aggregate size of the waste. Wastes with high clay content (> 50 percent) may release clay into the mixing water, which may result in a large concentration of these clay particles near the surface of the solidified matrix, producing an inferior quality matrix. Coal and lignite in excess of 1 percent may also impair the quality of the solidified waste mixture. Wastes with very high moisture content should be treated as sludges, and may therefore require larger amounts of reagents for solidification.

Oils and grease (and other nonpolar organics) may have deleterious effects on the ability of the matrix to set and thus may reduce the unconfined compressive strength of the treated waste. STC reports that levels of up to 60 percent oil and grease have been successfully treated. Low pH wastes (e.g., acid sludges) may react with the relatively higher pH materials used in the reagent mixture, resulting in incomplete solidification. Such wastes must be neutralized prior to treatment.

For wastes with large aggregate sizes, incomplete mixing can occur, which may result in pockets of untreated waste within an otherwise homogeneous waste/reagent mixture. Well-graded raw wastes (i.e., wastes with several different particle sizes) will form more stable monolithic blocks than poorly-graded (one-sized) raw wastes. Additional screening and size reduction of the SPT contaminated soil aggregates down to 0.04-0.08 inch (1-2 mm) diameter was necessary to ensure that individual aggregates did not bias the chemical analyses.

Wastes containing volatile organics may release these organics during the mixing process, resulting in artificially high percent reductions for these constituents. In addition, the concentrations of metals or semivolatile organics in the waste may impair the ability to meet desired levels of these constituents in the treated waste. For example, if the objective of the treatment is to render a waste nonhazardous, the higher the contaminant concentration in the raw waste, the higher the concentration in the treated waste, and even after 90 percent reduction in TWA or leachate concentrations, the technology may not be appropriate for some wastes because the wastes may still be considered hazardous after treatment.

3.3.2 Volume/Density Increase

The STC technology, like any immobilization technology, may be less desirable for use in treating wastes as the ratio of the volume and/or density of the treated waste to the volume and/or density of the raw waste increases. Off-site disposal of treated wastes becomes more difficult and costly with increasing volume, since disposal costs are usually on a unit volume or total weight basis. Based on the results of this demonstration, volume increases of 59 to 75 percent can be expected after treatment. The volume increase depends on the characteristics of the waste being treated and the desired performance specifications. The bulk densities of the wastes showed only minor increases of 0.6 to 11 percent, with an average increase of 5.5 percent resulting from the addition of reagents during treatment. For on-site disposal, the above increases may be desirable in situations where additional soil material is needed for backfilling and leveling depressions. The increased volume would reduce the costs of purchasing, transporting, and placing fill material at the site.

3.3.3 Operating Conditions

Several operating conditions for the STC stabilization/solidification technology can be varied to account for differences in the characteristics of the waste to be treated. Such operating parameters for the STC process include mixer power, mixing time, and the additives ratio for the reagents used to treat the raw waste, as shown in Table 3-1. Any of these operating conditions can be modified to accommodate differences in waste characteristics. Operating conditions can also be modified to yield treated waste better suited for a particular disposal option or use.

The power delivered to the mixer can have a significant effect on the degree of mixing of the waste. Wastes that are exceptionally viscous or that have larger particle or aggregate sizes may require a larger power output by the mixer.

Variations in mixing time can result in differences in the chemical properties of the treated waste through processes such as oxidation/reduction reactions. Arsenic in Batch 4 is thought to have been reduced from arsenate (V) to the more mobile species arsenite (III) during the inordinately long pretreatment mixing time (4.5 hours). Supplemental analyses for selected samples from Batches 3, 4, and 5 indicate that the raw waste from Batch 4 contained higher quantities of the arsenic ion-species arsenite (205 mg/kg) and lower amounts of arsenate (20 mg/kg) relative to Batches 3 and 5, which had high arsenate (61 mg/kg and 260 mg/kg, respectively) and low arsenite values (<2 mg/kg in both batches). The net result in this case was that the arsenic in Batch 4 was more easily leached under the acid TCLP conditions, thus the anomalously low percent reduction for the TCLP extract in Batch 4.

Table 3-1. Operating Parameters for the STC SITE Demonstration

Parameters	Batch					
	RM	1	2	3	4	5
Waste soil weight (lbs)	0	5,000	5,000	4,000	4,000	4,464
Silica sand weight (lbs)	1,972	0	0	0	0	0
Dry reagent weight (lbs)	695	1,732	1,723	1,382	1,413	1,638
Water added (lbs)	422	2,172	3,850	1,713	1,760	1,759

Table 3-1. Operating Parameters for the STC SITE Demonstration (continued)

Parameters	Batch					
	RM	1	2	3	4	5
Water lost during curing (lbs)	NA	97	NA	41	71	67
Mixer power (hp)	29	29	29	29	29	29
Current to mixer (amp-hr)	17	83	77	77	248	79
Pretreatment mixing time (min)	0	50	60	60	270	60
Treatment mixing time (min)	22	60	40	40	60	45
Additives ratio ^a	NC	0.761	NC	0.764	0.776	0.746

RM = Reagent mixture

NA = Not analyzed

NC = Not calculated

a = The additives ratio is the sum of the weight of additives, including water of hydration, divided by the weight of wastes.

b = Batch 2 was not analyzed due to inhomogeneity of the treated waste.

The additives ratio for the process can be varied to account for differences in the composition of certain wastes. For example, the volume of water added in the process should be adjusted to account for the moisture content of the waste. Certain waste streams with high moisture content may not be easily treated using solidification/stabilization procedures. The amount of water used in the process should be decreased with increasing moisture content of the waste to be treated.

3.3.4 Climate and Curing Conditions

The curing temperature and the curing time will have an effect on the physical characteristics of the treated waste. Solidified wastes should be allowed to cure for several weeks; The American Concrete Institute (ACI) and the American Society for Testing Materials (ASTM) guidelines for construction materials recommend a cure time of 28 days (ACI Standard 318) at 16° to 27°C (ASTM, Method C31-90). In general, treated wastes cured at higher temperatures will cure faster, but may have lower structural integrity compared to that of wastes cured at lower temperatures. (An exception to this correlation is waste that is cured at temperatures at or below freezing.) In general, treated wastes cured at constant room temperature will become increasingly stable with increasing

time, although blocks of treated waste that are exposed to the effects of weather for an extended period of time may begin to break down as a result of weather conditions, including precipitation and freeze/thaw cycles.

Below-freezing temperatures and heavy rain could have an adverse impact on the operation of the STC immobilization technology. If sub-freezing temperatures are expected, the mixer and water source should be insulated or heated to avoid freezing of the water used in the process. The mixer should also be covered during periods of heavy rain, because such precipitation could interfere with the solidification of the treated waste. Raw materials, including the reagents, should also be protected from precipitation.

4.0 DESCRIPTION OF THE DEMONSTRATION SITE

The Selma Pressure Treating (SPT) site in Selma, California was selected for demonstrating the STC technology based on its waste characteristics, the results of treatability testing, and site logistical considerations. The following section provides background information about the SPT site and information regarding contamination at this site.

4.1 SITE BACKGROUND

The SPT site has been used for chemical treatment of lumber since 1942. The original wood-preserving process consisted of dipping the lumber into a mixture of pentachlorophenol (PCP) and oil, and allowing the excess fluid to drip off as the wood dried on open storage racks. In 1965, the site operators converted their process to a pressure-treating process that consisted of two steps:

- (1) conditioning the lumber to reduce moisture content and increase permeability, and
- (2) impregnating the wood with chemical preservatives.

Federal and state agencies have been jointly involved in regulatory and enforcement actions at the site since the 1970s. The California Regional Water Quality Control Board (CRWQCB) was first to impose discharge standards, monitor water quality, and require the owners to submit operational reports. On January 13, 1981, the following agencies conducted an Uncontrolled Hazardous Waste Site Investigation: EPA's Field Investigation Team (FIT), California Environmental Protection Agency (CEPA), and CRWQCB. SPT site owners filed for bankruptcy on April 13, 1981, and the plant closed its operations in June 1981. On September 4, 1981, CRWQCB issued a Cleanup and Abatement Order for the SPT site. The landowner, Selma Leasing Company, indicated that it could not comply with the Cleanup and Abatement Order; however, an attorney for the company indicated to CRWQCB that Selma Leasing Company would accept responsibility for the geotechnical investigation. In February 1982, Sawmill Properties, Inc. acquired the facility, but stipulated that Selma Leasing Company continue to accept responsibility for the investigations of contamination caused by past operations. Sawmill Properties, Inc., reopened the plant in Summer 1982, as the Selma Treating Company. In August 1983, EPA scored the SPT site at 48.83 using the Hazard Ranking System (HRS). Based on this information, the site was placed on the Superfund National Priorities List (NPL) in September 1983. Following a remedial investigation/feasibility study

(RI/FS), a Record of Decision (ROD) was signed on September 24, 1988, and a Pre-Remedial Design Soil Boring Report was completed in June 1989 (CDM, 1989).

4.2 SITE DESCRIPTION

The SPT site is located approximately 15 miles southeast of Fresno, California, adjacent to the southern city limits of Selma, California (Figure 4-1). The site is situated in the center of the San Joaquin River Valley, an area that contains numerous vineyards. The entire SPT site covers 18 acres; however, the actual wood-treatment area of this site covers only 3 to 4 acres. While zoned for heavy industrial use, the site is located in a transition zone between agricultural, residential, and industrial areas with approximately 12 residences and businesses located within 1/4 of a mile. The CRWQCB has classified the ground-water resources in the vicinity of the SPT site as a beneficial use, sole-source aquifer. This resource provides the necessary domestic water supply for the surrounding communities and scattered county residences. Surface-water irrigation systems are also supplemented by this ground-water resource.

4.3 SITE CONTAMINATION CHARACTERISTICS

From 1942 to 1971, wastes from the lumber treatment plant were disposed of in various ways: (1) runoff into drainage ditches and a percolation ditch; (2) drainage into dry wells; (3) spillage on open ground; (4) placement into an unlined pond and a sludge pit; and (5) disposal in an adjacent vineyard through pipelines (Figure 4-2). Known chemical preservatives used at the site include:

- Fluor-chromium-arsenate-phenol (1966 to 1973)
- Woodtox 140 RTU (1974 only)
- Heavy oil penta solution (1977 only)
- LST concentrate (1970 to 1979)
- Copper-8-quinolinoate (1977 to 1980)
- PCP (1970 to present)
- Chromated-copper-arsenate (CCA) (1953 to present).

Figure 4-1. Regional Location Map - SPT Site, Selma, California

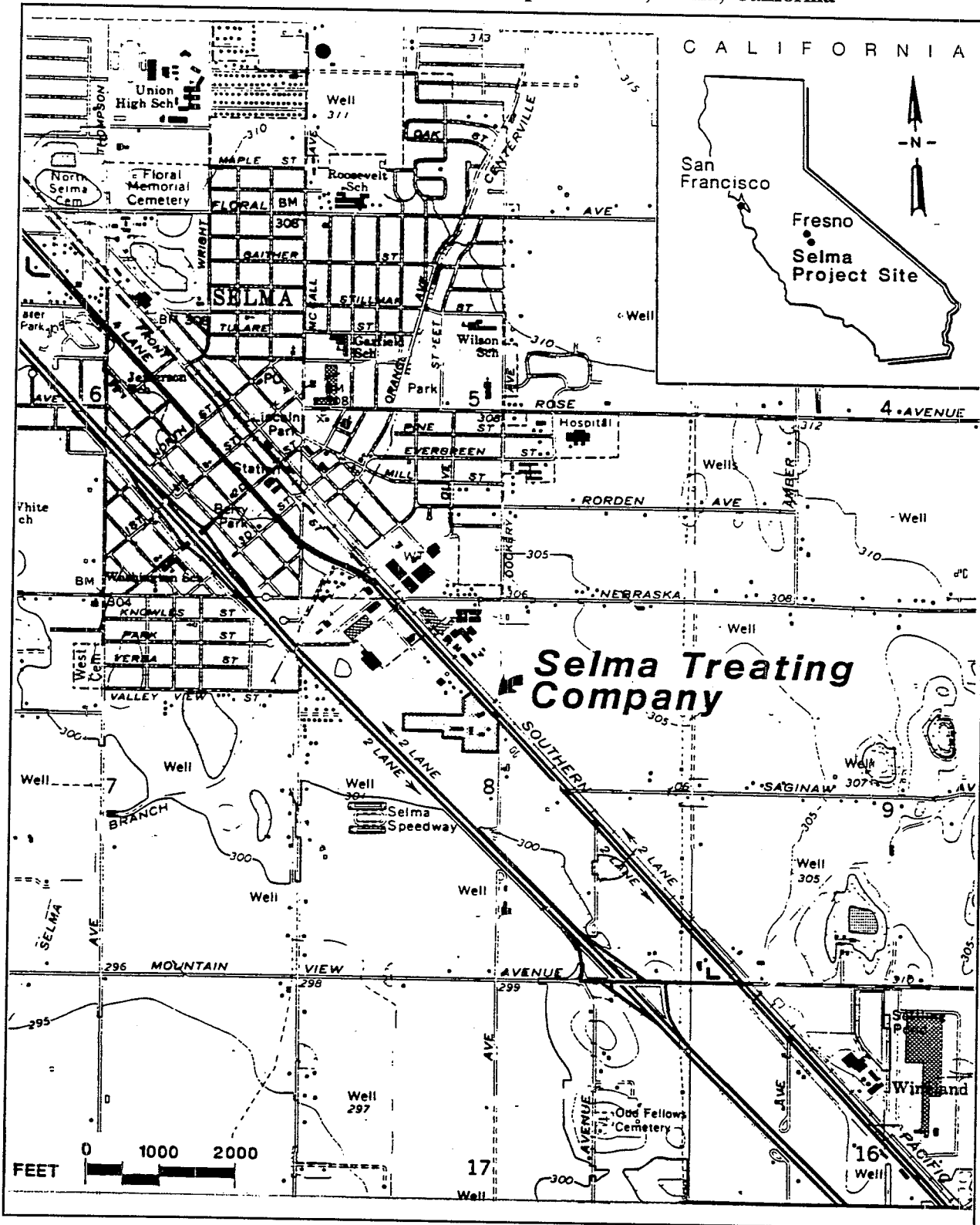
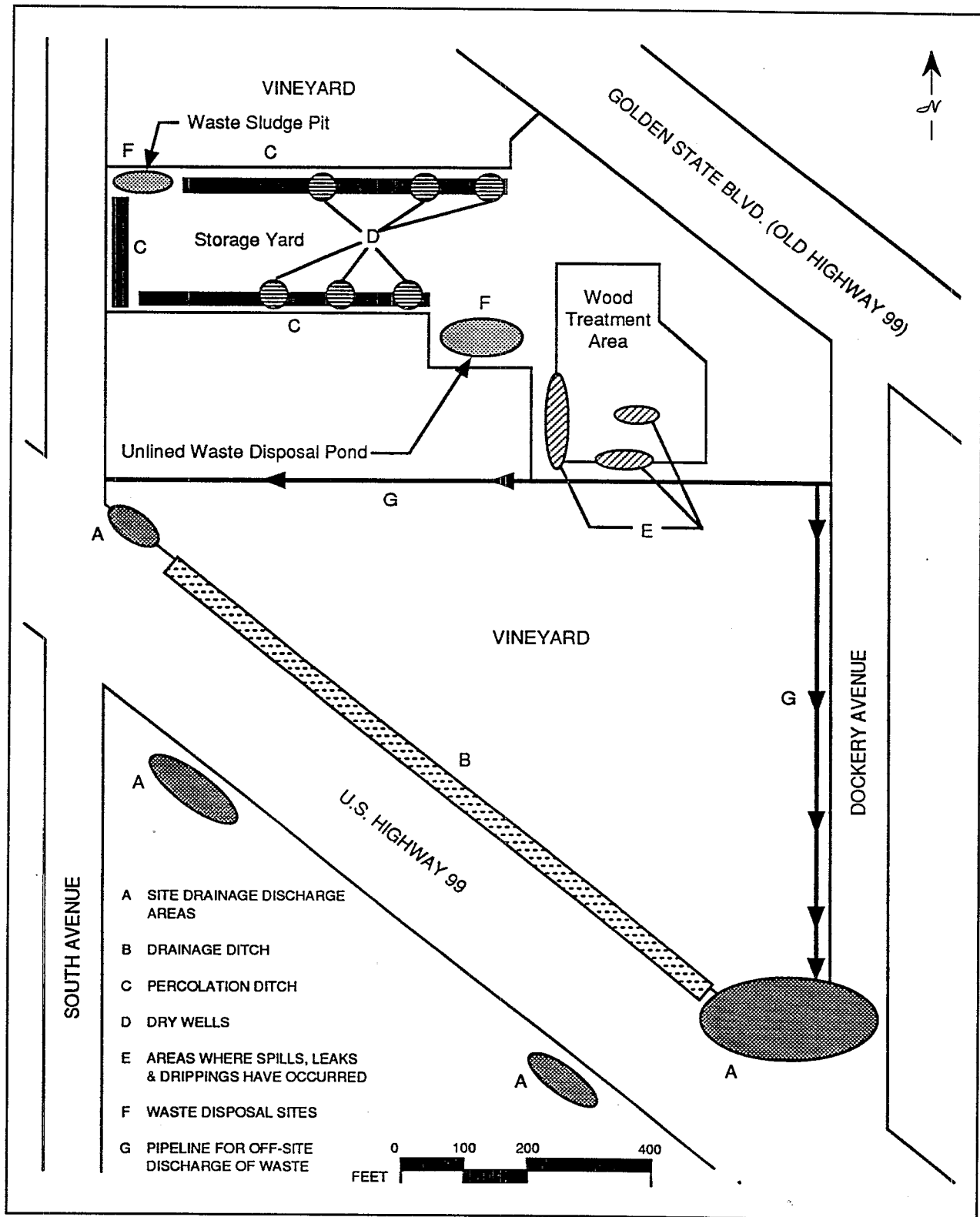


Figure 4-2. Areas of Contamination at the SPT Site



A contaminated ground-water plume emanating from the site has been identified in addition to pervasive soil contamination beneath the SPT site. The Pre-Remedial Design Soil Boring Report (CDM, 1989) for the site indicates that the primary metal contaminants are arsenic (375 to 1,900 mg/kg), chromium (1,900 mg/kg), and copper (1,500 mg/kg). PCP was also reported (1,900 to 8,400 mg/kg), along with associated degradation and impurity products including polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), and chlorinated phenols. Hydrocarbon-related constituents were reported at the site and are thought to have resulted from the use of diesel fuel as a carrier for the PCP. The hydrocarbon-related constituents include volatile organic compounds such as benzene, toluene, and xylene, plus polycyclic aromatic hydrocarbons (PAHs) such as naphthalene and pyrene. Results from the Final Remedial Investigation Report and the Pre-Remedial Design Soil Boring Report (CDM, 1988a and 1989) confirm that the highest levels of contamination occur in the first 5 feet of the soil material.

5.0 DEMONSTRATION PROCEDURES

The procedures that were used during the STC immobilization technology demonstration were developed to test the technology using contaminated soils from the Selma Pressure Treating (SPT) site near Fresno, California. Based on the SPT site characteristics and the results of treatability testing, a demonstration plan was prepared that detailed the proposed sampling, analytical, quality assurance, and health and safety procedures (U.S. EPA, 1990a and b). This section summarizes the actual demonstration procedures, including waste characterization and treatability testing, field activities, laboratory analyses, and the quality assurance/quality control plan.

5.1 WASTE CHARACTERIZATION AND TREATABILITY TESTING

The SPT site was considered for demonstration of the STC technology because its waste contained elevated mixtures of organic and inorganic contaminants. Preliminary waste samples from the SPT site were collected in September, 1989 to characterize the waste, to confirm contaminants of concern for the SITE demonstration, and to conduct treatability studies to optimize the STC process.

Four primary objectives for the preliminary sampling for waste characterization were to 1) obtain data to determine if STC's processes could stabilize organic and inorganic contaminants; 2) obtain data to determine if STC's processes could effectively treat wastes found at the SPT site; 3) identify potential waste feed areas for STC's demonstration; and 4) obtain data to determine if the testing and analytical methods used in the preliminary sampling were effective so that appropriate methods could be selected for the full-scale demonstration. The objectives of the preliminary sampling were accomplished through the collection and analysis of area characterization and treatability samples. Area characterization samples were site samples that were analyzed to determine the concentration of contaminants in the raw samples and were not subjected to treatment. Treatability samples were site samples that were subjected to the STC's treatment process and subsequently analyzed to determine the effectiveness of the technology. All samples were collected from an unlined waste disposal pond known to contain the major contaminants desired to test the ability of the STC treatment process to stabilize organic and inorganic contaminants.

5.1.1

Preliminary Treatability Study

Table 5-1 presents chemical and leaching data for the area characterization samples as determined by TWA and TCLP (using standard EPA SW-846 methods), and CALWET (as described in the California Health and Safety Code, Section 66700). Detectable concentrations of volatile organic contaminants by TWA included methylene chloride and toluene. Base neutral contaminants included naphthalene and phenanthrene. Acid extractable compounds detected by TWA included 2,3,4,6-tetrachlorophenol (400 mg/kg) and PCP (8,400 mg/kg). No pesticides or PCBs were detected. Arsenic (1,900 mg/kg), chromium (1,900 mg/kg), and copper (1,500 mg/kg) were also detected by TWA. TCLP leachates were analyzed for volatile and semivolatile organics and metals, but no significant amounts of leachable organics (above TCLP guidelines) were detected in the sample extract. Low concentrations of metals were detected in the TCLP extract sample; however, the levels did not exceed the TCLP Waste Characteristic Test guidelines. The area characterization sample was further subjected to the CALWET method and was determined, based on California regulations, to its concentrations of PCP (3.2 mg/L), arsenic (20 mg/L), and copper (60 mg/L).

The treatability samples were processed by STC in November, 1989. Treatment entailed mixing the waste with a proprietary solidification reagent in three different reagent-to-soil ratios: minimum (50 percent of presumed optimum); presumed optimum; and maximum (150 percent of presumed optimum). These reagent-to-soil ratios correspond to additives ratios of 0.22, 0.42, and 0.62 respectively. The treated material was poured into molds and allowed to cure for 28 days.

Four samples of the treated waste were submitted for analysis, one for each reagent concentration plus a duplicate of the optimum reagent concentration. Extracts of the treated wastes were generated using the TCLP and the extracts were analyzed for volatile organics, semivolatile organics, and metals. Table 5-2 presents the TCLP analytical results for the treatability samples.

Analysis of the TCLP extracts from the treatability samples for organics indicated that no sample contained significant amounts (above TCLP guidelines) of leachable organics. Several compounds were detected at higher concentrations in the treatability samples compared to the TCLP extracts of the area characterization samples. Methylene chloride was detected in several samples at greater concentrations than those detected in the area characterization samples: Acetone was detected

Table 5-1. Area Characterization Sample Data for SPT Site

Analyte	Concentrations (ppm)		
	TWA	TCLP	CALWET
Methylene Chloride	0.067	<0.005 ^d	NA
Toluene	0.058	<0.005 ^d	<0.005
Total Xylenes	<0.025	0.011 ^d	<0.005
Naphthalene	34 ^a	0.057 ^d	0.016 ^a
Phenanthrene	86	0.006 ^{a,d}	NA
2-Methylnaphthalene	<50	0.008 ^{a,d}	<0.020
Pentachlorophenol (PCP)	8,400	4.3 ^c	3.2
2,3,4,6-Tetrachlorophenol	400	0.440 ^d	NA
Arsenic	1,900	1.7	20
Cadmium	8 ^b	<0.01	0.1
Chromium	1,900	0.06	4.0
Copper	1,500	7.5 ^d	60
Nickel	8	0.04 ^d	<0.4
Lead	21	<0.1	<1
Zinc	48	0.24 ^d	0.7

NA = Not analyzed.

a = Estimated concentration (below reporting limit).

b = Spiked sample recovery not within control limits.

c = Calculated from a diluted analysis.

d = Analyte not applicable to the toxicity characteristic.

Table 5-2. TCLP Analyses of Treatability Samples of STC-Treated Wastes

Analyte	Concentrations (mg/L)		
	Minimum Reagent Mixture	Optimum Reagent Mixture ^a	Maximum Reagent Mixture
Methylene Chloride ^e	0.034	<0.005/ <0.005	<0.005
Acetone ^e	0.130	0.140/0.130	0.170
Toluene ^e	<0.005	<0.005/ <0.005	<0.005
Total Xylenes ^e	<0.005	<0.005/ <0.005	<0.005
Phenol ^e	0.027	0.021/0.023	0.038
Benzoic Acid ^e	<0.050	0.007 ^b / <0.050	<0.050
Naphthalene ^e	0.008 ^b	<0.010/ <0.010	0.009 ^b
2,4,6-Trichlorophenol	0.005 ^b	<0.010/ <0.010	<0.010
Pentachlorophenol (PCP)	26 ^c	0.033 ^b /2.3 ^c	13 ^c
Phenanthrene ^e	<0.010	<0.010/ <0.003 ^b	<0.010
Bis(2-ethylhexyl)phthalate ^e	<0.010	0.044/ <0.010	<0.010
Arsenic	0.11 ^d	0.035/0.049	0.077 ^d
Cadmium	<0.01	<0.01/ <0.01	<0.01
Chromium	<0.05	0.63/0.64	<0.05
Copper ^e	0.18	4.2/4.3	0.17
Nickel ^e	<0.04	0.089/0.085	<0.04
Lead	<0.1	<0.1/ <0.1	<0.1
Zinc ^e	0.027	0.27/0.28	0.095

a = Duplicate analysis of treatability sample.

b = Estimated concentration (below reporting limit).

c = Calculated from a diluted analysis.

d = Post digestion spike for Furnace AA analysis is out of control limits (85-115 percent), while sample spike absorbance is less than 50 percent of spike absorbance.

e = Analyte not applicable to the toxicity characteristic.

in several samples when it was not detected in the area characterization samples. Phenol was detected in all treatability samples at higher concentrations than in the area characterization samples. Pentachlorophenol was detected at greater concentrations than the area characterization samples for the minimum- and maximum-reagent-mixture treatability samples. PCP was present in the sample duplicate at a level approximately two orders of magnitude higher than in the original optimum-reagent-mixture treatability sample. 2,4,6-Trichlorophenol was detected in the minimum-reagent-mixture treatability sample, but was not detected by either the TWA or the TCLP extract analysis of the area characterization samples. Bis(2-ethylhexyl) phthalate was detected in one of the optimum-reagent-mixture treatability sample when it was not detected in the area characterization samples. In general, the sample duplicate of the optimum-reagent-mixture treated sample showed good corroboration with its sample counterpart.

Analysis of the TCLP extracts for metals indicated that several metals (chromium, nickel, and zinc) were elevated in concentration in the optimum-reagent-mixture treatability sample waste and the corresponding duplicate sample as compared with the area characterization samples.

Based on the analytical results of STC's waste treatment at three different reagent-to-soil ratios, treatability samples with the least leaching ability (optimum treatment dose) were chosen for further analyses, and additional characterization and treatability studies. In March 1990, the treatability samples were analyzed by TWA, CALWET-Distilled Water, and TCLP-Cage tests for volatile organics, semivolatile organics, and 32 Hazardous Substance List (HSL) metals. The results for these analyses are presented in Table 5-3. Acetone, PCP, and phenanthrene were the only organics detected in the treatability samples. Acetone, however, was also detected in the associated laboratory blank. Results of the TWA for metals indicated that levels of arsenic, chromium, copper, and cadmium were above typical background levels. Cobalt was not detected in the TWA of the characterization samples.

PCP was detected in the CALWET-Distilled Water leachate of the treatability sample. Small amounts of arsenic, chromium, copper, and zinc were also detected. Methylene chloride and acetone were detected in the CALWET-Distilled Water leachate of the treatability sample, but also in the associated laboratory blanks.

**Table 5-3. Analytical Results of TWA, CALWET-Distilled Water, and
TCLP-Cage for Treatability Samples**

Analyte	Concentrations (ppm)		
	TWA	CALWET-Distilled Water	TCLP-Cage
Methylene Chloride	< 1.3	0.010 ^b	0.006 ^{b,c,i}
Acetone	2.8 ^{a,b}	0.190 ^b	0.130 ^{b,c,i}
Phenol	NA	0.037 ^a	0.050 ⁱ
2,4,6-Trichlorophenol	NA	0.010 ^a	NA
Pentachlorophenol (PCP)	840	40 ^d	31 ^{c,d}
Phenanthrene	54 ^a	NA	NA
Arsenic	1,300 ^{e,f,g}	0.0441 ^{c,g}	0.01 ^{c,g}
Cadmium	6	NA	0.073 ^c
Chromium	860	0.067	< 0.05
Cobalt	5.7 ^h	NA	NA
Copper	810 ^{e,g}	0.37	< 0.03 ⁱ
Lead	21 ^g	< 0.1	41 ^c
Nickel	6.9	NA	0.15 ^{c,i}
Zinc	45 ^g	0.038	6.6 ^{c,i}

NA = Not analyzed

a = Estimated concentration (below reporting limit).

b = Compound was detected in blank.

c = Detected at a higher concentration than in original area characterization analysis.

d = Calculated using a greater dilution than the primary analysis.

e = Spiked sample recovery not within control limits.

f = Post digestion spike for Furnace AA analysis out of control limits (85-115 percent), while sample absorbance is less than 50 percent of spike absorbance.

g = Duplicate analysis not within control limits.

h = Not detected in original area characterization analysis.

i = Analyte not applicable to the toxicity characteristic.

The only volatile organic compounds detected in the TCLP-Cage leachate of the treatability sample were methylene chloride and acetone; both also were detected in the associated laboratory blank. PCP was the only semivolatile compound detected. Lead concentrations exceeded the regulatory threshold for the TCLP, thereby classifying the sample as a hazardous waste. Small amounts of cadmium, nickel, and zinc were also detected in the TCLP-Cage leachate of the treatability sample.

5.1.2 Arsenic-Spiked Treatability Study

An additional characterization and treatability study was conducted in February, 1990 to determine the ability of STC's treatment process to chemically stabilize organic and inorganic contaminants in the presence of high concentrations of arsenic. Samples of raw waste collected in September, 1989 (area characterization samples) were homogenized and spiked with arsenic. Spiked samples were then characterized by TWA, TCLP, and TCLP-Distilled Water analyses. The spiked samples were also sent to STC. STC treated the spiked waste samples with a predetermined optimum amount of solidification reagents, poured the mixture into 250-mL Teflon molds and allowed the molds to cure for 28 days. The treated samples were analyzed in the same manner as the raw waste spiked with arsenic.

Table 5-4 presents the analytical results for the arsenic-spiked raw and treated waste. Arsenic was detected in the raw waste sample at 5,100 mg/kg, considerably higher than the addition of approximately 500 mg/kg during the spiking procedure. (The concentration of arsenic in the raw waste collected at the SPT site in September, 1989 had previously been determined to be 1,900 mg/kg by TWA.) Several factors may have contributed, solely or in combination, to the unexpectedly high concentration of arsenic in the spiked raw waste. For example, the arsenic solution may not have mixed evenly with all soil particles, or may not have chemically bonded evenly throughout the soil. Uneven mixing or chemical binding could result in areas of higher or lower arsenic concentration within the waste sample. Treated samples had somewhat less elevated concentrations of arsenic. PCP was present in the raw waste but was not detected by TWA in the duplicate treated waste samples.

Table 5-4. Analytical Results for Arsenic-Spiked Raw and Treated Waste

Analyte	Concentrations (ppm)					
	TWA		TCLP		TCLP-Distilled Water	
	Raw Waste	Treated Waste ^a	Raw Waste	Treated Waste ^a	Raw Waste	Treated Waste ^a
Pentachlorophenol (PCP)	1,140 ^b	<580/ ^c 580	4.8 ^b	41 ^b /120 ^b	14 ^b	48 ^b /53 ^b
Phenanthrene	<100	100 ^c /99 ^c	NA	NA	NA	NA
Benzoic Acid	NA	NA	<250 ^f	<2,500 ^f /240 ^{c,f}	NA	NA
Arsenic	5,100 ^d	4,400 ^e /3,300 ^e	22 ^{d,e}	3.1/3.0	22 ^{d,e}	0.72/0.58
Cadmium	31	31/18	0.051	0.014/0.014	0.065	<0.01/ ^c 0.01
Chromium	3,500	2,900/3,500	0.076	1.0/1.1	0.13	0.051/ ^c 0.05
Copper	2,400 ^{d,e}	1,600/2,100	10 ^f	<0.03 ^f / ^c 0.03 ^f	2.7	<0.03/ ^c 0.03
Lead	37 ^e	54/40	NA	NA	NA	NA
Nickel	9.7	9.1/8.4	NA	NA	NA	NA
Zinc	53 ^e	47/46	0.3 ^f	0.094 ^f /0.079 ^f	0.17	<0.02/ ^c 0.02

NA = Not analyzed.

a = Duplicate analysis of treated waste sample.

b = Calculated using a greater dilution than the primary analysis.

c = Estimated concentration (below reporting limit).

d = Spiked sample recovery not within control limits.

e = Duplicate analysis not within control limit.

f = Analyte not applicable to the toxicity characteristic.

Besides the expected increased concentration of arsenic, other metals were also detected in the spiked raw and treated waste at concentrations higher than those detected in the original area characterization and treatability samples. These metals include: cadmium, chromium, copper, lead, and zinc. Lead increased in concentration for the treated waste.

Raw and treated waste samples were analyzed for semivolatile organic compounds and metals following TCLP extraction. Elevated concentrations of PCP were detected in all samples. Concentrations of PCP in the treated waste samples were greater than levels detected in the raw waste TCLP analysis. In fact, the concentration of the duplicate exceeds the TCLP regulatory threshold, classifying that sample as hazardous waste even though the raw waste was not classified as a hazardous waste. TCLP extracts of raw and treated waste in the previous study (not spiked with arsenic) contained consistently lower concentrations of PCP than treated samples in this treatability study (spiked with arsenic). PCP concentrations for TCLP extracts in the previous study were not in excess of the TCLP guidelines.

In general, detected concentrations of metals in the TCLP leachates were lower in the treated waste samples than in the raw waste sample. However, concentrations of chromium were higher in the treated waste TCLP leachate than in the raw waste TCLP leachate. Neither of the treated waste samples exceeded the TCLP regulatory threshold for arsenic even though the spiked concentration of arsenic as measured by TWA in the raw waste (5,100 mg/kg) was unexpectedly high compared to the experimental protocol (500 mg/kg added). All other concentrations of metals in the raw and treated leachate were below TCLP regulatory thresholds.

As compared with the previous study, elevated concentrations of PCP were detected in all samples of the raw and treated waste TCLP-Distilled Water leachates. Additionally, concentrations of PCP in the treated waste TCLP-Distilled Water leachate were higher than the concentration in the raw waste sample. In general, concentrations of metals were lower in the treated waste TCLP-Distilled Water leachates than in the raw waste TCLP-Distilled Water leachate.

5.1.3 Reagent Mixture and Ratio Selection

Finally, a small-scale treatability study was conducted just prior to the pilot-scale demonstration to fine-tune the reagent-to-waste ratio for the technology demonstration at the SPT site. In September, 1990, treatability samples were collected from the unlined waste pit area. Waste samples were shipped separately for analysis and to STC for treatment. The raw waste sample was subjected to TWA for PCP and extraction by TCLP followed by analysis for arsenic.

STC treated the waste sample by mixing the waste with proprietary solidification reagents (identified as P3, P4, P5, P27, and S6) in five different batch mixes. The contents of each of the five batches are listed in Table 5-5. The treated material was poured into 125-mL PVC bottles (molds) and allowed to cure for 28 days. Following curing, the five treated samples were analyzed in duplicate for the same parameters as the raw waste sample.

Table 5-5. STC-Treatability Study Batch Contents

Treated Waste Constituents (grams)	Batch				
	1	2	3	4	5
Waste	800	400	400	400	400
Water	120	120	80	80	80
P27	200	100	80	100	80
P3	80	0	0	0	10
P4	0	40	0	40	10
P5	0	0	0	0	10
S6	0	0	40	0	10

Results of the TWA for PCP and TCLP extraction followed by analysis for arsenic are presented in Table 5-6. PCP was detected in the raw waste sample at a concentration of 17,000 ppm, considerably higher than concentrations of PCP detected in the samples of the previous characterization and treatability studies using waste from the same unlined pit area. Concentrations of PCP in all samples and duplicates from the five separate treated waste batches were all less than the

concentration detected in the raw waste sample. Treated waste from Batch 2 contained the least amount of PCP.

Table 5-6. Analytical Results for PCP and Arsenic Treatability Study

Analyte	Concentrations (ppm)					
	Raw Waste	Treated Wastes (Batch) ^c				
		1	2	3	4	5
PCP ^a	17,000 ^d	1,500/640	640/770	980/960	4,100 ^d /2,500 ^d	2,200 ^d /3,600 ^d
Arsenic ^b	2.1	0.13 ^e /0.095 ^e	0.073 ^e /0.090 ^e	0.38 ^e /0.038 ^e	0.028 ^e /0.038 ^e	0.33 ^e /0.31 ^e

a = TWA

b = TCLP

c = Duplicate analysis of treated waste sample.

d = Calculated from a greater dilution than the primary dilution.

e = Spiked sample recovery not within control limits.

Arsenic was detected in the TCLP extract of the raw waste sample at a concentration of 2.1 mg/L. This result is consistent with results of the previous studies where the concentrations of arsenic in the TCLP extract of a raw waste sample and duplicate from the same site were 1.6 and 1.8 mg/L. Results of the arsenic analyses for the five treated waste samples and duplicates show that Batch 4 had the lowest concentration of arsenic, followed by Batch 2, 1, 3, 5 in order of increasing concentrations of leachable arsenic. Neither the raw nor any of the treated waste samples exceeded the TCLP regulatory threshold for arsenic. Based on the results of this treatability study, the reagents used in Batch 2 were selected for the pilot-scale demonstration.

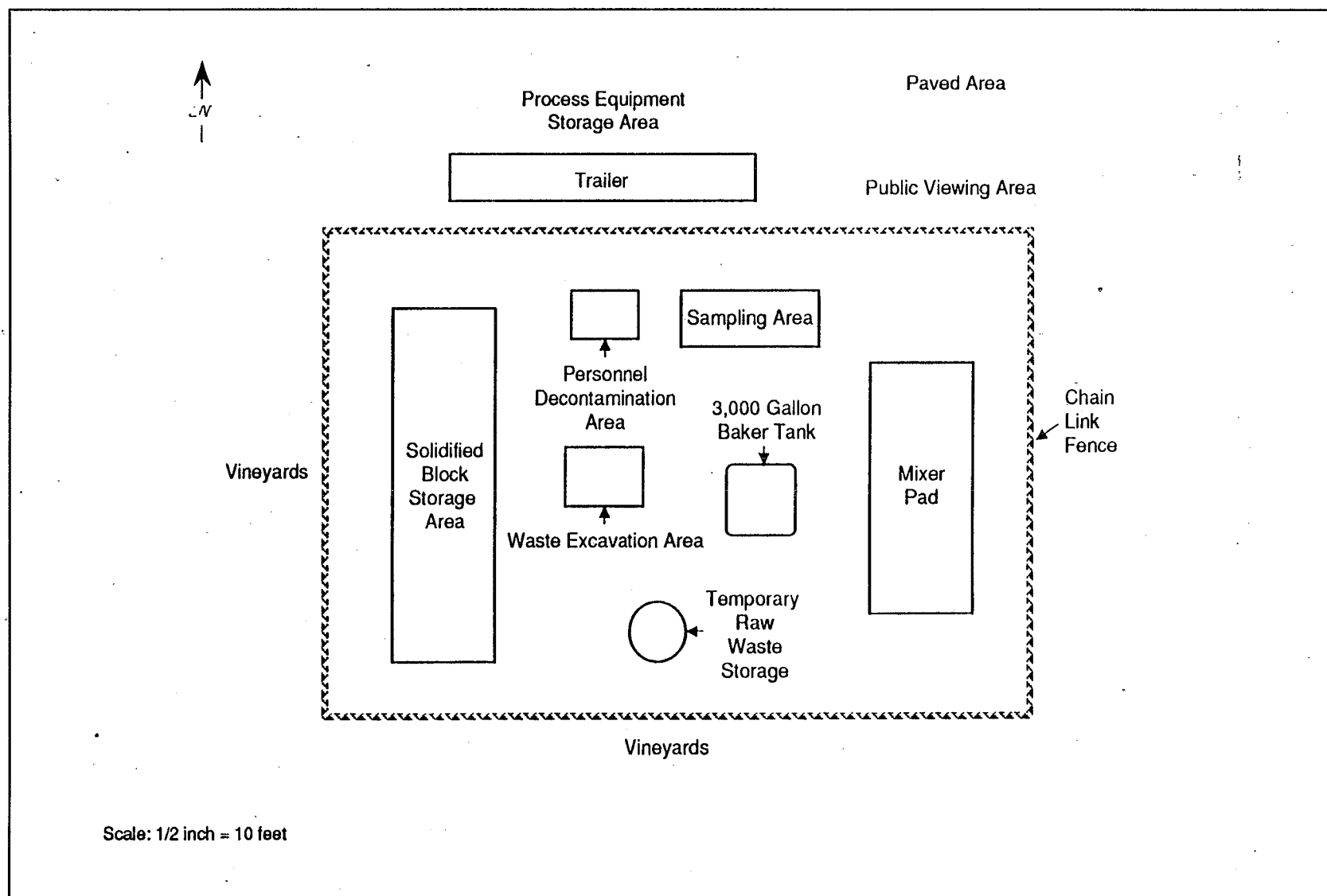
5.2 PILOT-SCALE DEMONSTRATION FIELD ACTIVITIES

Following EPA's approval of the demonstration plan, site preparation and equipment mobilization for the demonstration began in mid-November, 1990. The demonstration was divided into three stages, including site preparation, technology demonstration, and site demobilization. Site preparation began 1 week prior to the treatment technology demonstration. EPA and its contractors established a waste treatment area, staging and storage areas, a decontamination zone, and a public

viewing area, as depicted in Figure 5-1. To implement the technology at the SPT site and to meet the demonstration objectives, the EPA SITE team and STC personnel used the following on-site provisions:

- A 50- by 100-foot mixer pad area for the STC process equipment and temporary accumulation of waste and treatment reagents. The mixer pad area was constructed with a plastic liner and berm.
- A 45- by 6-foot gravel and compacted soil area for an office and sampling trailer. An area appropriate for parking and equipment staging was also provided.
- A 15- by 50-foot area lined with 20-mil high-density polyethylene (HDPE) liner to place and store the solidified waste. The treated waste was discharged into cardboard forms mounted on pallets and placed in the storage area. The storage area was graded in such a way that a low point in the liner existed for collection of any rainwater runoff from the solidified waste.
- A dumpster for containment and disposal of all nonhazardous waste.
- A diesel electric generator to supply 480-volt, 3-phase, 500-amp service for the STC process equipment. In addition, standard electric power was provided by a portable generator for the support trailer, equipment, and miscellaneous needs.
- Process and wash water for the treatment unit and decontamination. This water was obtained from the facility's potable water. Approximately 220 gallons of water were needed per treated batch.
- A scale for weighing reagents and raw wastes.
- A heavy equipment decontamination area bermed and lined with 20-mil plastic for cleaning large equipment. This area was also provided with a pump for the collection of wash water.
- A personnel decontamination station adjacent to the equipment decontamination area. The station was supplied with appropriate basins, brushes, water, and soap. This area also included several tables to function as an equipment drop, a first-aid station, and emergency eye-wash facilities.
- A 3,000-gallon Baker wastewater tank used to contain decontamination water.
- A gasoline-powered, high-pressure cleaner to clean the STC process equipment and other heavy equipment.
- Three 55-gallon drums to contain contaminated clothing, supplies, and other materials that could not be disposed of in the dumpster. These drums were disposed of at appropriate off-site facilities.

Figure 5-1. SPT SITE Demonstration Layout



- A 45-foot office and sampling trailer for EPA personnel, contractors, and STC personnel.
- A portable telephone for ordering supplies, scheduling deliveries, and emergency communications.
- Sanitary facilities for personnel involved with the demonstration.
- A public viewing area for the demonstration.
- A locked chain-linked fence constructed around the work area upon completion of the demonstration. Entry to the SPT property was restricted during the demonstration between 5:00 p.m. and 8:00 a.m.

A backhoe/front-end loader was used to excavate contaminated waste from the unlined waste disposal pond. To ensure that the soil with the highest concentrations of contaminants was tested, the first 2 to 3 feet of soil from the disposal pond were used for the demonstration. Therefore, it was necessary to excavate an approximately 300-square-foot area to provide the total amount of contaminated soil needed for the demonstration. The excavation was lined with a layer of 20-mil HDPE and backfilled with 1 foot of sand overlain by 1 foot of crushed stone (1-inch diameter) and clean soil at the conclusion of the demonstration.

Contaminated soils from the unlined waste disposal pond were transported to the processing area, where temporary storage piles covered by 10-mil HDPE were set up as necessary prior to batch processing.

Upon completion of the equipment setup, STC conducted a startup and test run to ensure that the equipment was operating properly and that all SITE team members understood the sampling procedures. During this procedure, a small batch of clean silica sand (1,972 lbs) was treated with STC's reagents (695 lbs). This initial "reagent mixture" run constituted a treatment process blank.

Waste treatment began with the transport of approximately 5,000 lbs of raw waste material to the 5-cubic-yard high-intensity batch mixer. The contaminated soil was blended in the mixer until STC personnel determined that the waste was adequately homogenized. Pretreatment grab samples were taken directly from the discharge from the mixer at three separate time intervals and placed into sample containers prior to the addition of treatment reagents. Mixing continued for up to 1 hour

following the addition of the reagents. The treated material was then discharged into three 1-cubic-yard cardboard forms. Samples were collected from the forms immediately after the treated waste was discharged from the mixer. For each batch run, complete records were maintained of pertinent operating parameters including weight of the contaminated soils, STC reagents, and water added; mixer power; and mixing time (see Table 3-1).

Certain contaminated soils (PCP-encrusted "hardpan") treated during the demonstration were not well mixed after treatment; the treated waste contained numerous large (0.5 inch) inclusions of untreated waste aggregates. These pockets of untreated waste resulted in the exclusion of Batch 2 from analytical evaluation. The problem was solved in subsequent batches by passing the raw waste through a series of screens prior to treatment, reducing the raw waste aggregate size to approximately 0.04 to 0.08 inch (1-2 mm) diameter. This pretreatment allowed for adequate mixing to occur; the subsequent batches (i.e., Batches 3 through 5) appeared to be homogeneous mixtures.

The generation of large amounts of contaminated dust from the movement of equipment, supplies, and site personnel caused fouling of the intake to the photoionization device that was used for air monitoring. This dust problem was remedied through the application of a light spray of water to the site from a water truck. An additional problem was a dust cloud of reagents created upon initially mixing the dry reagents with the waste in the mixer. Consequently, a tarp was secured over the top of the mixer after adding (but before mixing) the dry reagents. Although no downwind residents or receptors appeared to be affected by the small dust cloud "puff" of finely divided dry reagents during the demonstration, slurring the reagents prior to addition is desirable for future uses of the technology.

Prior to waste excavation for subsequent treatment, all STC equipment that would come in contact with raw waste materials was decontaminated. In addition, all process equipment was decontaminated between batch runs and at the conclusion of the demonstration. A portable high-pressure cleaner was used to decontaminate the equipment. Water and wastes generated from the cleaning of equipment were pumped to a 3,000-gallon Baker wastewater tank and stored on site for subsequent disposal. Personnel decontamination wash water and wastes were collected from wash basins and also placed in this tank for off-site disposal. All sampling equipment was cleaned with a

nonphosphate detergent and triple rinsed with distilled water before reuse. The resulting wash water was stored on site for disposal.

Once all test runs were completed and equipment decontaminated, all test equipment was demobilized and removed from the SPT site. Decontamination and demobilization took approximately 2 weeks. Residual wastes from the demonstration included 1,000 gallons of water and wastes from decontamination, three 55-gallon drums of contaminated clothing and disposable sampling supplies, and a 30-cubic-yard dumpster containing miscellaneous nonhazardous trash. The decontamination wastes, drums, and dumpster wastes were disposed of by EPA personnel and contractors at appropriate facilities.

The 1-cubic-yard cardboard form containing treated clean sand and 15 similar forms filled with treated wastes were placed on wooden pallets in the western section of the demonstration site. After 28 days, the cardboard forms were removed and disposed. The exposed monoliths of treated waste were inspected periodically for 32 months. After the 32 months monitoring period, EPA disposed of these wastes according to the cleanup criteria selected for the SPT site.

5.3 LABORATORY ANALYSES

The contaminants of regulatory concern at the SPT site were arsenic and PCP; however, other target analytes of secondary importance to the pilot-scale demonstration were chromium, copper, nickel, and lead, as well as other semivolatile organic compounds such as phenanthrene, tetrachlorophenol, phenol, and naphthalene. The corresponding critical measurements for the demonstration were TCLP for arsenic (and other inorganic analytes) and TWA for PCP (and other organic analytes). Noncritical measurements included TCLP for organic analytes, and TCLP-Distilled Water and CALWET leach procedures for both organic and inorganic analytes. Additional noncritical measurements for the demonstration included the TCLP-Cage and the modified American Nuclear Society (ANS) 16.1 leach tests, analysis for PCDDs and PCDFs, engineering and geotechnical tests, and petrographic examinations. In addition, chemical characterization of the raw and treated waste included pH, Eh, loss on ignition, and neutralization potential. Acid neutralization capacity tests originally planned for the untreated wastes could not be completed due to the acidic nature of the raw

waste samples. Instead, neutralization potential measurements were conducted on both the raw waste and treated waste samples.

For critical measurements, six or more field replicate samples were collected, depending on data variability as determined in the initial treatability tests on waste samples from the SPT site. Field replicate samples for metal contamination were analyzed for arsenic, chromium, copper, nickel, and lead only. Replicate samples were also analyzed for semivolatile organic compounds including PCP. In addition, field replicate geotechnical/engineering samples were collected to determine unconfined compressive strength, permeability, and petrographic examination, but not for particle size, water content, bulk density, wet/dry, or freeze/thaw testing.

5.4 QUALITY ASSURANCE/QUALITY CONTROL PLAN

EPA-approved sampling, analytical testing, and quality assurance and quality control (QA/QC) procedures were followed to obtain data of known quality. Details on QA/QC procedures are presented in the demonstration plan (U.S. EPA, 1992). Quality assurance results are presented in Section 6.8 of this report.

6.0 ANALYTICAL PROCEDURES AND RESULTS

6.1 PURPOSE

Performance data were collected from the STC solidification/stabilization treatment process demonstration at the SPT site to evaluate the overall performance of the STC immobilization technology as it was applied to wastes from the SPT site. The following sections describe these results in detail and include results from the leaching tests, chemical tests, physical tests, and petrographic examinations. Table 6-1 summarizes the analytical and measurement methods used for each of the various analyses. This section also presents a discussion of the mass balance between raw and treated waste, long-term test results, and quality assurance (QA) results.

6.2 MATERIALS BALANCE

This section describes materials balances, the additives ratio, and volume expansion. The purpose of performing these analyses is to provide information on the extent of dilution of wastes and the increase in volume that occurred as a result of treatment. When comparing the concentrations of contaminants in the TCLP extracts or total contaminant concentrations in the wastes before and after treatment, it is important to consider the dilution that occurred as a result of the addition of reagents as part of the treatment process. In addition, this analysis provides information on the quantity of material that will require land disposal capacity if this process is used. The volume expansion ratio can be used to estimate final disposal capacity needed for a known quantity of raw waste.

6.2.1 Materials Balance

Table 6-2 presents materials balance information for each of the four batches that were sampled and analyzed as part of demonstration. The data in this table are based upon information collected during the demonstration and information obtained from laboratory analyses.

Approximately 4,000 to 5,000 pounds of contaminated soil were treated for each test batch. Dry reagents were added for each batch in amounts that varied from 34 to 37 percent of the raw waste by weight, with an average value of 35 percent. The amount of water added ranged from 39 to 44 percent, with an average value of 42 percent, of the contaminated soil weight. Each test batch produced approximately 3 to 4 cubic yards of treated material.

Table 6-1. Analytical and Measurement Methods

Title	Method	Raw Waste	Treated Waste	Reagent Mixture	Clean Sand	Process Water	Leachate	Digestates	6-Month Long-Term	6-Month Long-Term Leachate	18- and 32-Month Long-Term	18- and 32-Month Long-Term Leachate
TCLP	TCLP	•	•	•	•				•		•	
TCLP-Distilled Water	TCLP	•	•	•								
TCLP-Cage	TCLP-Cage		•	•								
CALWET	CALWET	•	•	•								
Modified ANS 16.1	Modified ANS 16.1		•	•								
Metals: Sb, Be, Cd, Cr, Cu, Ni, Ag, Zn, Pb, Na, Al, Ba, Ca, Co, Fe, Mg, Mn, Mo, K, V	3050	•	•	•	•							
	3010						•			•		•
	6010							•				
Pb,Tl,As,Se	3050	•	•	•	•							
	3020						•			•		•
Pb	7421							•				
Tl	7841							•				
As	7060						•	•		•		•
Se	7740						•	•		•		•
Hg	7471	•	•	•	•							
	7470						•			•		•
Volatile Organic Compounds	TWA 8240	•	•	•			•			•		•
Semivolatile Organic Compounds	TWA 8270	•	•	•			•	•		•		•
PCDDs and PCDFs	TWA 8280	•	•									

Table 6-1. Analytical and Measurement Methods (Continued)

Title	Method	Raw Waste	Treated Waste	Reagent Mixture	Clean Sand	Process Water	Leachate	Digestates	6-Month Long-Term	6-Month Long-Term Leachate	18- and 32-Month Long-Term	18- and 32-Month Long-Term Leachate
pH	EPA 9045	•	•	•	•							
	EPA 9040					•	•			•		
Eh	Modified EPA 9045	•	•	•	•							
	ASTM D-1498					•	•			•		
Neutralization Potential	Sobeck, 1978		•	•								
Loss On Ignition	ASTM C-114	•	•									
Oil and Grease	Modified EPA 413.2	•	•	•								
Particle Size	ASTM D-422	•										
Moisture Content	ASTM D-2216	•										
	TMSWC-4		•	•								
Bulk Density	ASA-13.2	•										
	TMSWC-2		•	•								
Permeability	TMSWC-13		•	•								
Unconfined Compressive Strength	ASTM D-1633		•	•							•	
Wet/Dry Weathering	TMSWC-12		•	•								
Freeze/Thaw Weathering	TMSWC-11		•	•								
Petrographic	Modified ASTM C457/C856	•	•	•								

Table 6-2. Summary of Materials Balance Data

Parameters	Batch			
	1	3	4	5
Raw waste weight (lbs)	5,000	4,000	4,000	4,464
Dry reagent weight (lbs)	1,732	1,382	1,413	1,638
Water added (lbs)	2,172	1,713	1,760	1,759
Water lost during curing (lbs)	97	41	71	67
Additives ratio	0.761	0.764	0.776	0.746
Bulk density of raw waste (lb/ft ³)	88.6	96.1	96.1	96.1
Bulk density of treated waste (lb/ft ³)	98.0	96.8	98.6	101.1
Volume expansion ratio	1.59	1.75	1.73	1.66
Volume increase (%)	59	75	73	66

6.2.2 Additives Ratio

Reductions in leachable and total PCP, arsenic, chromium, and copper concentrations in the treated wastes may result from diluting the raw wastes with binders used in the treatment process. Therefore, the additives ratio was calculated for each treatment batch, to correct for the dilution of the raw waste by the addition of the process reagents. The additives ratio was determined using the following equation:

$$AR = \frac{W_r + (W_w - W_c)}{W_s}$$

where:

AR = Additives Ratio

W_r = Weight of dry reagents

W_w = Weight of water added

W_c = Weight of water lost during curing

W_s = Weight of raw waste

For example, the weight of raw waste for Batch 1 was 5,000 pounds. The weight of the STC dry reagents for Batch 1 was 1,732 pounds. The weight of water added was 2,172 pounds and the weight of the water lost during curing was 97 pounds. Therefore, the calculation of the additives ratio for Batch 1 is as follows:

$$\text{Additives Ratio} = \frac{1,732 + (2,172 - 97)}{5,000} = 0.761$$

The additives ratios determined for each treatment batch are shown in Table 6-2.

6.2.3 Volume Expansion Ratio

The volume expansion ratio (VER) between treated waste and raw waste was calculated for each treatment batch using the following equation:

$$VER = \frac{(W_s + W_a) B_s}{(W_s) (B_t)}$$

where:

VER = Volume expansion ratio

W_s = Weight of raw waste

W_a = Weight of dry reagents and water, minus water lost during curing

B_s = Bulk density of raw waste (88.6 to 96.1 lb/ft³)

B_t = Bulk density of treated waste (96.8 to 101.1 lb/ft³)

For example, the variables for Batch 1 are as follows:

W_s = 5,000 pounds

W_a = 3,807 pounds

B_s = 88.6 lb/ft³

B_t = 98.0 lb/ft³

Therefore, the VER for Batch 1 is 1.59. The VER for each treatment batch is shown in Table 6-2.

These ratios indicate that the contaminated soils from the SPT site increased in volume from 59 to 75 percent. The VER may allow prospective users of the STC technology to estimate the volume of treated waste that will need to be disposed of, transported, or stored if it is known how much raw waste is present at a site. It should be noted, however, that the VER is a site-specific value, requiring an estimate of the in-situ bulk density of the raw waste.

6.3 LEACHING TESTS

For purposes of comparison and to assess the ability to meet state and federal regulations, several leach tests were chosen to evaluate the STC immobilization technology. These tests include the Toxicity Characteristic Leaching Procedure (TCLP) and two modified versions of the TCLP: (1) TCLP-Distilled Water and (2) TCLP-Cage. In addition, the California Waste Extraction Test (CALWET) and the modified American Nuclear Society (ANS) 16.1 leach test were utilized. An additional modified TCLP test, TCLP-pH 12, which used a 0.1 M borate buffer solution as the leaching medium, was conducted as well (see Section 6.4). These methods and the results are described in the following subsections.

Leaching tests were performed on both raw and treated wastes cured for 28 days, as well as the reagent mixture. Analytical results presented in this report are limited to contaminants of regulatory concern, including PCP and arsenic, which were targeted for treatment during the demonstration. Analytical results for chromium and copper, although not target analytes for treatment, are also reported. For each of the analytes the results are reported as average values for six or more samples of raw and treated waste, and include sample standard deviation values (that is, an *estimate* (s) of the universe standard deviation (σ), if an infinite number of measurements could be made). In addition, the results include calculated percent reductions accounting for dilution effects of added reagents by incorporating the additives ratio for each batch tested (see Section 6.2.2). The additives ratio is the weight of additives, including water of hydration, divided by the weight of wastes. Thus, percent reduction was calculated using the following formula:

$$\text{Percent Reduction} = \left[1 - (1 + \text{Additives Ratio}) \times \frac{\text{Concentration of Treated Waste}}{\text{Concentration of Raw Waste}} \right] \times 100.$$

In general, when a constituent was not detected in the treated waste, the reporting limit for the treated waste was used to calculate a minimum value for the percent reduction (indicated by ">"). Reporting limits are calculated by multiplying the method detection limit by a dilution factor specific for each analysis. If a constituent was not detected in the raw waste, the percent reduction was not calculable. It should be mentioned that as a result of the dilution associated with the STC treatment, negative percent reduction values may be expected even if the concentrations show a decrease in values from the raw to the treated waste. Table 6-3 summarizes federal and California regulatory thresholds for the critical analytes. These thresholds classify wastes as hazardous or nonhazardous, depending on whether the concentration of a contaminant exceeds the given threshold value.

6.3.1 Toxicity Characteristic Leaching Procedure (TCLP)

The TCLP determines the concentrations of extractable organic and inorganic constituents using an acidic extraction solution and is designed to evaluate a waste's potential for leaching contaminants when the waste is codisposed with municipal waste in a landfill. This test is the basis for EPA's regulation of solid waste containing arsenic and other heavy metals as hazardous wastes. Because the TCLP is a regulatory test for RCRA hazardous wastes, it is likely to be considered in determining the success of future applications of the STC technology to other wastes.

The sample material was prepared by crushing, cutting, or grinding the waste if the surface area of the solid sample was less than 3.1 cm² or the aggregate size was larger than 9.5 mm diameter. This step, however, was not necessary with the contaminated soils from the SPT site. The leaching procedure involved a liquid-to-solid ratio of 20 to 1, maintained at a specified pH using an acetate buffer. The waste-leaching medium mixture was agitated in a rotary extractor for 18 ± 2 hours and was filtered through a 0.6 to 0.8 mm glass-fiber filter. The pH and type of leaching medium used depended on the alkalinity of the solid phase of the waste.

Table 6-4 presents a summary of PCP, arsenic, chromium, and copper concentrations in TCLP extracts for the raw and treated wastes. In addition, Figures 6-1 through 6-4 graphically show concentrations of target analytes in the raw and treated wastes for PCP, arsenic, chromium, and copper, respectively. Average concentrations are depicted by solid symbols for the raw waste and open symbols for the treated wastes. Error bars associated with each symbol represent the standard deviation for each average of six analytical results.

Table 6-3. Regulatory Thresholds for Critical Analytes of the SPT Waste

Constituents	Federal	State of California	
	FRTL (mg/L) ^a	TTLC (mg/kg) ^b	STLC (mg/L) ^c
Pentachlorophenol (PCP)	100	17	1.7
Arsenic	5.0	500	5.0
Chromium (hexavalent)	---	500	5.0
Chromium (total)	5.0	2,500	560
Copper	---	2,500	25

a = Federal Regulatory Threshold Limit, based on TCLP.

b = Total Threshold Limit Concentration, based on TWA.

c = Solubility Threshold Limit Concentration, based on CALWET.

Table 6-4. Analytical Results for TCLP Leachates

Constituent: PCP			
Batch	Concentrations (mg/L)		Average Percent Reduction ^{a,b}
	Raw Waste	Treated Waste	
1	1.4	3.9 ^c	-300
	1.6	5.2 ^c	
	1.4	0.73	
	1.5	3.0	
	1.7	3.7	
	1.4	4.0	
Average	1.5 ± 0.13	3.4 ± 1.5	
3	2.8 ^c	0.25 ^{f,g}	>81
	2.1	0.25 ^{f,g}	
	2.0	0.25 ^{f,g}	
	2.4	0.25 ^{f,g}	
	2.4	NA	
	1.9	NA	
Average	2.3 ± 0.33	<0.25	
4	1.5	5.6	-460
	2.5	5.7	
	2.1	5.3	
	1.5	6.0	
	0.89	5.4	
	2.0	5.1	
Average	1.75 ± 0.57	5.5 ± 0.32	
5	1.1	3.0	31
	0.085 ^c	0.12 ^c	
	3.8 ^c	1.9	
	3.0	0.16 ^c	
	2.6	0.12 ^c	
	3.1	0.079 ^c	
Average	2.3 ± 1.4	0.9 ± 1.25	

Table 6-4. Analytical Results for TCLP Leachates (continued)

Constituent: Arsenic			
Batch	Concentrations (mg/L)		Average Percent Reduction ^{a,b}
	Raw Waste	Treated Waste	
1	1.3	0.048 ^d	92
	2.2	0.096 ^d	
	1.3	0.051 ^d	
	2.2	0.044 ^d	
	2.3	0.19 ^{d,q}	
	1.6	0.084 ^d	
Average	1.8 ± 0.47	0.086 ± 0.055	
3	1.1	0.091 ^d	83
	0.99	0.081 ^d	
	0.97	0.10 ^d	
	0.88	0.16 ^{d,s}	
	0.93	0.097 ^d	
	1.5 ^a	0.078	
Average	1.1 ± 0.23	0.10 ± 0.030	
4	2.0	0.91 ^d	35
	2.3	0.76 ^d	
	2.3	0.67 ^d	
	2.2	1.1 ^d	
	3.6 ^s	0.84 ^d	
	2.0	0.97	
Average	2.4 ± 0.60	0.88 ± 0.15	
5	3.1	0.56	71
	3.6	0.47	
	3.3	0.61	
	3.3	0.65	
	3.8	0.60	
	2.9	0.40	
Average	3.3 ± 0.33	0.55 ± 0.095	

Table 6-4. Analytical Results for TCLP Leachates (continued)

Constituent: Chromium			
Batch	Concentrations (mg/L)		Average Percent Reduction ^{ab}
	Raw Waste	Treated Waste	
1	0.051	0.24	-230
	0.21	0.25	
	0.053	0.24	
	0.20	0.25	
	0.22	0.24	
	0.052	0.25	
Average	0.13 ± 0.087	0.245 ± 0.0055	
3	0.05	0.19	NC
	<0.05	0.20	
	<0.05	0.18	
	<0.05	0.17	
	<0.05	0.18	
	<0.05	0.20	
Average	<0.05	0.19 ± 0.012	
4	0.071	0.29	-390
	0.073	0.28	
	0.077	0.29	
	0.083	0.27	
	0.23 ^a	0.27	
	0.084	0.27	
Average	0.10 ± 0.062	0.28 ± 0.0098	
5	0.24	0.32	-110
	0.25	0.31	
	0.24	0.33	
	0.28	0.29	
	0.37 ^a	0.29	
	0.23	0.38	
Average	0.27 ± 0.053	0.32 ± 0.033	

Table 6-4. Analytical Results for TCLP Leachates (continued)

Constituent: Copper			
Batch	Concentrations (mg/L)		Average Percent Reduction ^{a,b}
	Raw Waste	Treated Waste	
1	2.2	0.087	95
	4.5	0.085	
	2.4	0.090	
	4.4	0.089	
	4.5	0.089	
	2.5	0.099 ^a	
Average	3.4 ± 1.2	0.090 ± 0.0048	
3	1.5	0.074	90
	1.5	0.077	
	1.4	0.070	
	1.2	0.067	
	1.2	0.071	
	1.5	0.088	
Average	1.4 ± 0.15	0.0745 ± 0.0074	
4	6.2	0.10	97
	6.1	0.10	
	6.2	0.11	
	6.5	0.10	
	8.7 ^a	0.10	
	5.5	0.11	
Average	6.5 ± 1.1	0.10 ± 0.0052	
5	9.2	0.055	99
	9.3	0.058	
	8.8	0.085 ^a	
	9.5	0.054	
	12.0 ^a	0.057	
	7.8	0.064	
Average	9.4 ± 1.4	0.062 ± 0.012	

Table 6-4. Analytical Results for TCLP Leachates (continued)

NA = Not analyzed
NC = Not calculable

$$a = \text{Percent Reduction} = \left[1 - (1 + \text{Additives Ratio}) \times \frac{\text{Concentration of Treated Waste}}{\text{Concentration of Raw Waste}} \right] \times 100.$$

b = The additives ratio is the weight of additives, including water of hydration, divided by the weight of raw wastes. Values are 0.761, 0.764, 0.776, and 0.746 for Batches 1, 3, 4, and 5, respectively.

c = Reported value was calculated using a greater dilution than the primary analysis.

d = Spiked sample recovery not within control limits.

e = Estimated concentration; compound was detected but at a concentration amount less than the reporting limit.

f = Laboratory reporting limit; compound was not detected.

g = Sample failed surrogate recovery criteria.

q = Fails Q test (Shoemaker, 1974); value may be disregarded with 90% confidence (although it has been used to calculate the average value).

s = Value may be disregarded with 95% confidence (although it has been used to calculate the average value).

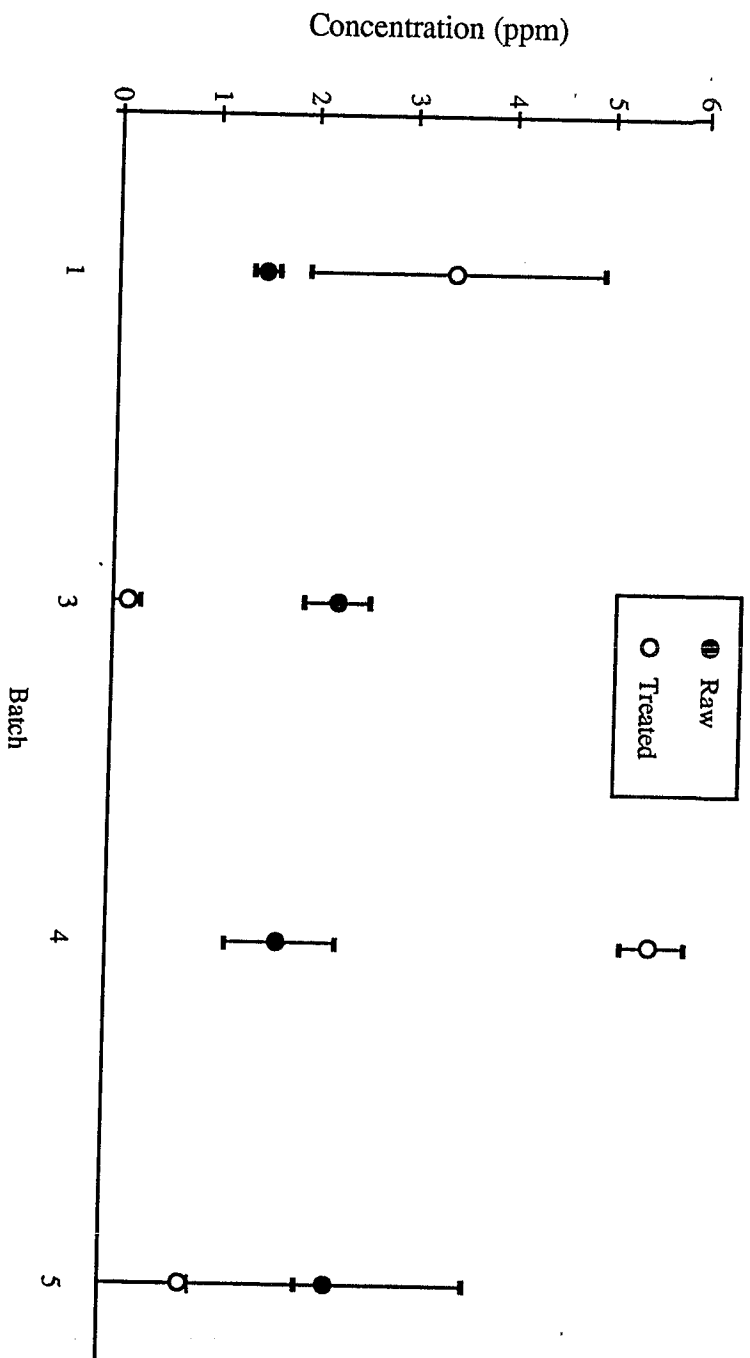


Figure 6-1. TCLP -- PCP

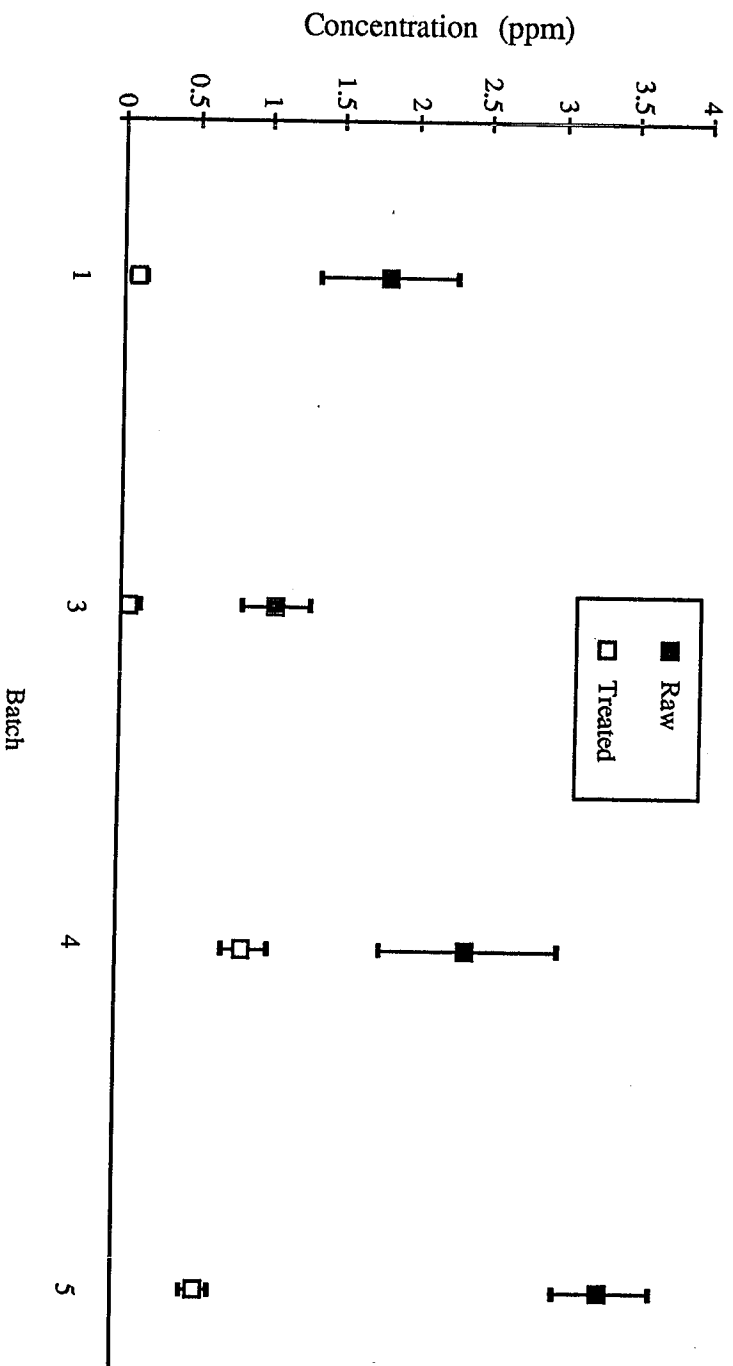


Figure 6-2. TCLP -- Arsenic

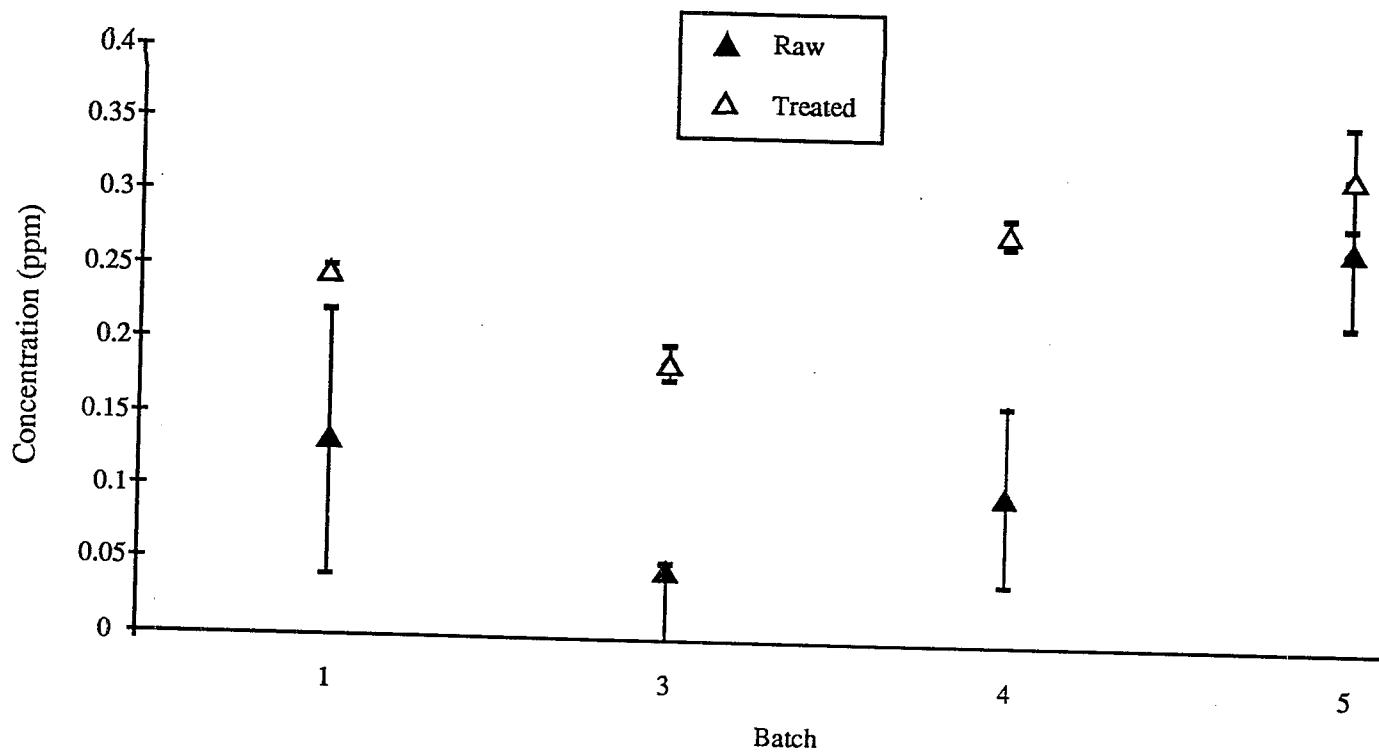


Figure 6-3. TCLP -- Chromium

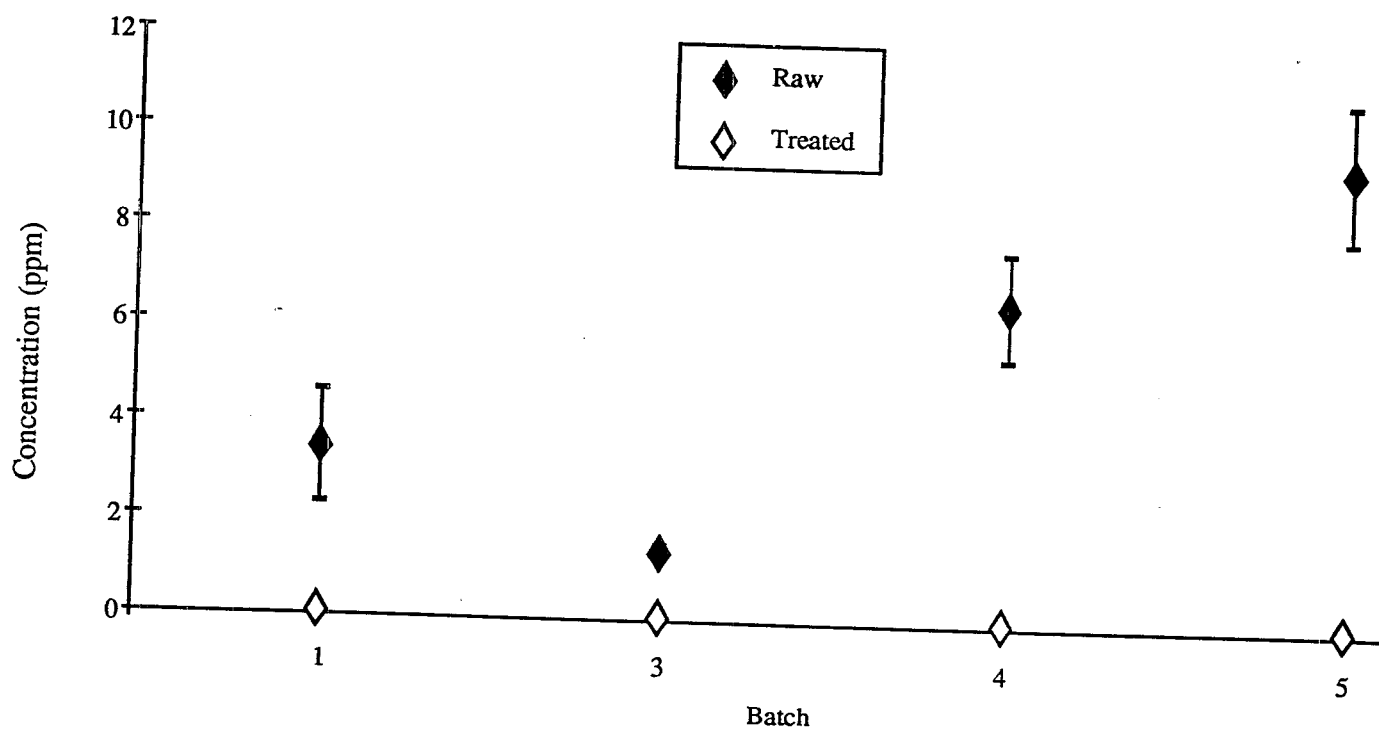


Figure 6-4. TCLP -- Copper

The TCLP test showed mixed results for PCP, with percent reductions ranging from -460 to greater than 81 percent. Batches 1 and 4 showed increases in extractable PCP after treatment. However, extractable concentrations of PCP in both the raw and treated wastes were well within the Federal Regulatory Threshold Limit (FRTL) of 100 mg/L for PCP.

Results for arsenic were variable among the four batches evaluated using the TCLP, with percent reductions ranging from 35 to 92 percent. Batch 4 depicts an anomalously low percent reduction of only 35 percent. This poorer-than-expected performance may be attributed to the inordinately long raw-waste mixing time for this batch (4.5 hours). Arsenic concentrations in the TCLP extracts were below the FRTL for both the raw and treated wastes.

Table 6-5 presents supplemental ion-chromatography analyses from TWA-extracts of arsenic for selected samples from Batches 3, 4, and 5. These results indicate that the raw waste from Batch 4 contained higher quantities of the arsenic ion-species arsenite (205 mg/kg) and lower values of arsenate (20 mg/kg), relative to Batches 3 and 5, that had higher arsenate (61 mg/kg and 260 mg/kg, respectively) and lower arsenite values (<2 mg/kg in both batches). It is likely that most of the Batch 4 arsenic was reduced from arsenate (V) to arsenite (III) during the long raw-waste-mixing process, thereby rendering the arsenic contaminant more mobile and easily leached under acid TCLP conditions. Alternatively, the STC process was not effective in converting arsenite to arsenate or a species which could be chemically stabilized; small amounts of both arsenite and arsenate were detected in the Batch 4 treated waste.

Chromium was not a target analyte for treatment because of very low leachable concentrations in the raw waste. Treated waste samples consistently leached greater quantities than the raw waste. TCLP tests for chromium in the treated waste showed large negative values for percent reduction ranging from -110 to -394 percent. However, both raw and treated waste concentrations of chromium were well below the FRTL of 5 mg/L for total chromium. Leachate concentrations of chromium from the raw waste of Batch 3 were below detection limits and therefore percent reductions were not calculable.

Copper concentrations in the TCLP extracts showed the greatest percent reduction following the STC treatment, even though copper was not a target analyte for treatment. Percent reductions ranged from 90 to 99 percent. Initial concentrations in the raw waste extracts were also low.

Table 6-5. Arsenic Ion-Speciation for STC-Treated Wastes

Constituent	Batch	Concentrations (mg/kg)		Average Percent Reduction ^{a,b}
		Raw Waste	Treated Waste	
Arsenate (V)	3	43	<2.0	>94
		78	<2.0	
		Average 60.5	<2.0	
	4	21	40	-91
		18	<2.0	
		Average 19.5	21	
	5	240	<2.0	>99
		280	<2.0	
		Average 260	<2.0	
Arsenite (III)	3	<2.0	<2.0	NC
		<2.0	<2.0	
		Average <2.0	<2.0	
	4	140	<2.0	94
		270	13	
		Average 205	7	
	5	<2.0	<2.0	NC
		<2.0	<2.0	
		Average <2.0	<2.0	

NC = Not calculable

$$a = \text{Percent Reduction} = \left[1 - (1 + \text{Additives Ratio}) \times \frac{\text{Concentration of Treated Waste}}{\text{Concentration of Raw Waste}} \right] \times 100.$$

b = The additives ratio is the weight of additives, including water of hydration, divided by the weight of raw wastes. Values are 0.761, 0.764, 0.776, and 0.746 for Batches 1, 3, 4, and 5, respectively.

6.2.2 TCLP-Distilled Water (Modified TCLP)

The TCLP-Distilled Water test is a modification of the standard TCLP designed to determine the mobility of contaminants present in the waste under neutral pH conditions. The test determines extractable constituents using water in place of an acid extraction solution, and is used to detect amphoteric metals and other constituents mobilized by matrix pH. All other steps of the TCLP (including liquid-to-solid ratio and duration of extraction time) remain the same. Table 6-6 depicts the analytical results for the various analytes in the TCLP-Distilled Water extracts. Figures 6-5 through 6-8 graphically show the concentrations of target analytes in the raw and treated wastes for PCP, arsenic, chromium, and copper, respectively. Average concentrations are depicted by solid symbols for the raw wastes and open symbols for the treated wastes. Error bars associated with each symbol represent the standard deviation for each average of six analytical results. In each case, concentrations of contaminants in the leachates from the treated wastes were less than concentrations in the leachates from the raw wastes. In the case of chromium, however, for Batches 4 and 5 there was considerable overlap between raw and treated waste concentrations.

PCP showed better stabilization characteristics under the neutral leaching conditions of this procedure than under the standard acid TCLP. Percent reductions following the STC treatment ranged from 80 to 97 percent based on this modified TCLP. Raw waste leachate concentrations from the TCLP-Distilled Water test, however, are an order of magnitude greater than those generated under the acid leaching conditions of the TCLP.

Arsenic depicted a high percent reduction level of 98 percent or greater for all four treatment batches. In addition, arsenic concentrations in the leachates of the raw waste were low (<1.5 mg/L), below the FRTL.

Chromium, although not a target analyte for treatment because of very low concentrations in the initial treatability TCLP raw waste leachates, showed mixed results following the STC treatment based on the TCLP-Distilled Water test. Percent reductions ranged from -42 percent to greater than 54 percent. Chromium was not detected in the treated waste leachates of Batches 1 and 3, although the raw waste leachates for these batches contained more chromium than the leachates for either the raw or treated wastes from Batches 4 and 5.

Table 6-6. Analytical Results for TCLP-Distilled Water Leachates

Constituent: PCP			
Batch	Concentrations (mg/L)		Average Percent Reduction ^{a,b}
	Raw Waste	Treated Waste	
1	13	2.5	80
	34 ^c	5.5	
	23	3.6 ^d	
	61	2.5	
	36	3.0	
	41	6.8	
Average	35 ± 16	4.0 ± 1.8	
3	69	0.69	97
	48	0.64	
	28	0.59	
	15	0.47	
	43	0.60	
	37	0.50	
Average	40 ± 18	0.58 ± 0.083	
4	49 ^e	3.9	83
	49 ^e	3.5	
	50 ^e	3.2	
	32	4.1	
	36	4.0	
	26	4.5	
Average	40 ± 10	3.9 ± 0.46	
5	68	2.5	93
	65	2.3	
	71	2.2	
	110	4.3	
	93	3.4	
	71	3.6	
Average	80 ± 18	3.05 ± 0.85	

Table 6-6. Analytical Results for TCLP-Distilled Water Leachates (continued)

Constituent: Arsenic			
Batch	Concentrations (mg/L)		Average Percent Reduction ^{a,b}
	Raw Waste	Treated Waste	
1	0.95 ^c	<0.01 ^f	>98
	1.0 ^c	<0.01	
	0.71 ^c	<0.01	
	0.82 ^c	<0.01	
	0.91 ^c	<0.01	
	0.42	<0.01	
Average	0.80 ± 0.21	<0.01	
3	0.66 ^c	<0.01	>98
	0.72 ^c	<0.01	
	0.75 ^c	<0.01	
	0.80 ^c	<0.01	
	0.77 ^{c,f}	<0.01	
	0.65	<0.01	
Average	0.725 ± 0.060	<0.01	
4	1.3	<0.010	98
	1.3	0.010 ^f	
	1.3	0.013	
	1.1	<0.010 ^f	
	1.4	<0.010 ^f	
	1.1	<0.010	
Average	1.25 ± 0.12	0.0115 ± 0.0021	
5	1.2	0.013	98
	1.1	0.013	
	0.97	0.011	
	1.1	0.011	
	0.96	0.013	
	1.1	<0.010	
Average	1.1 ± 0.091	0.012 ± 0.0011	

Table 6-6. Analytical Results for TCLP-Distilled Water Leachates (continued)

Constituent: Chromium			
Batch	Concentrations (mg/L)		Average Percent Reduction ^{a,b}
	Raw Waste	Treated Waste	
1	0.21	<0.05	>54
	0.31	<0.05	
	0.091	<0.05	
	0.16	<0.05	
	0.20	<0.05	
	0.17	<0.05	
Average	0.19 ± 0.072	<0.05	
3	0.20	<0.05	>48
	0.18	<0.05	
	0.18	<0.05	
	0.066 ^s	<0.05	
	0.22	<0.05	
	0.19	<0.05	
Average	0.17 ± 0.054	<0.05	
4	0.079	0.066	-42
	0.060	0.055	
	0.10	0.055	
	0.067	0.052	
	0.066	0.050	
	0.067	0.060	
Average	0.073 ± 0.0145	0.056 ± 0.0058	
5	0.15	0.078	-25
	0.11	0.077	
	0.063	0.079	
	0.057	0.076	
	0.10	0.084 ^a	
	0.15	0.079	
Average	0.105 ± 0.040	0.079 ± 0.0028	

Table 6-6. Analytical Results for TCLP-Distilled Water Leachates (continued)

Constituent: Copper			
Batch	Concentrations (mg/L)		Average Percent Reduction ^{a,b}
	Raw Waste	Treated Waste	
1	0.49	0.030	88
	0.73	0.030	
	0.25	0.031	
	0.37	<0.030	
	0.45	<0.030	
	0.43	0.032	
Average	0.45 ± 0.16	0.031 ± 0.00096	
3	0.41	0.030	>86
	0.37	0.030	
	0.36	<0.030	
	0.21 ^a	<0.030	
	0.45	<0.030	
	0.41	<0.030	
Average	0.37 ± 0.084	<0.030	
4	0.98	0.056	90
	1.1	0.053	
	0.97	0.055	
	0.97	0.055	
	1.0	0.055	
	0.92	0.051	
Average	0.99 ± 0.060	0.054 ± 0.0018	
5	0.71	0.032	90
	0.54	0.031	
	0.45	0.032	
	0.50	0.031	
	0.50	0.034	
	0.63	<0.030	
Average	0.555 ± 0.097	0.032 ± 0.0012	

Table 6-6. Analytical Results for TCLP-Distilled Water Leachates (continued)

- a* = Percent Reduction = $\left[1 - (1 + \text{Additives Ratio}) \times \frac{\text{Concentration of Treated Waste}}{\text{Concentration of Raw Waste}} \right] \times 100$.
- b* = The additives ratio is the weight of additives, including water of hydration, divided by the weight of raw wastes. Values are 0.761, 0.764, 0.776, and 0.746 for Batches 1, 3, 4, and 5, respectively.
- c* = Duplicate analysis not within control limits.
- d* = Estimated concentration; compound was detected but at a concentration less than the reporting limit.
- e* = Reported value was calculated using a greater dilution than the primary analysis.
- f* = Post digestion spike for Furnace AA analysis of control limits (85-115%), while sample absorbance was less than 50% of spike absorbance.
- q* = Fails *Q* test (Shoemaker, 1974); value may be disregarded with 90% confidence (although it has been used to calculate the average value).
- s* = Value may be disregarded with 95% of confidence (although it has been used to calculate the average value).

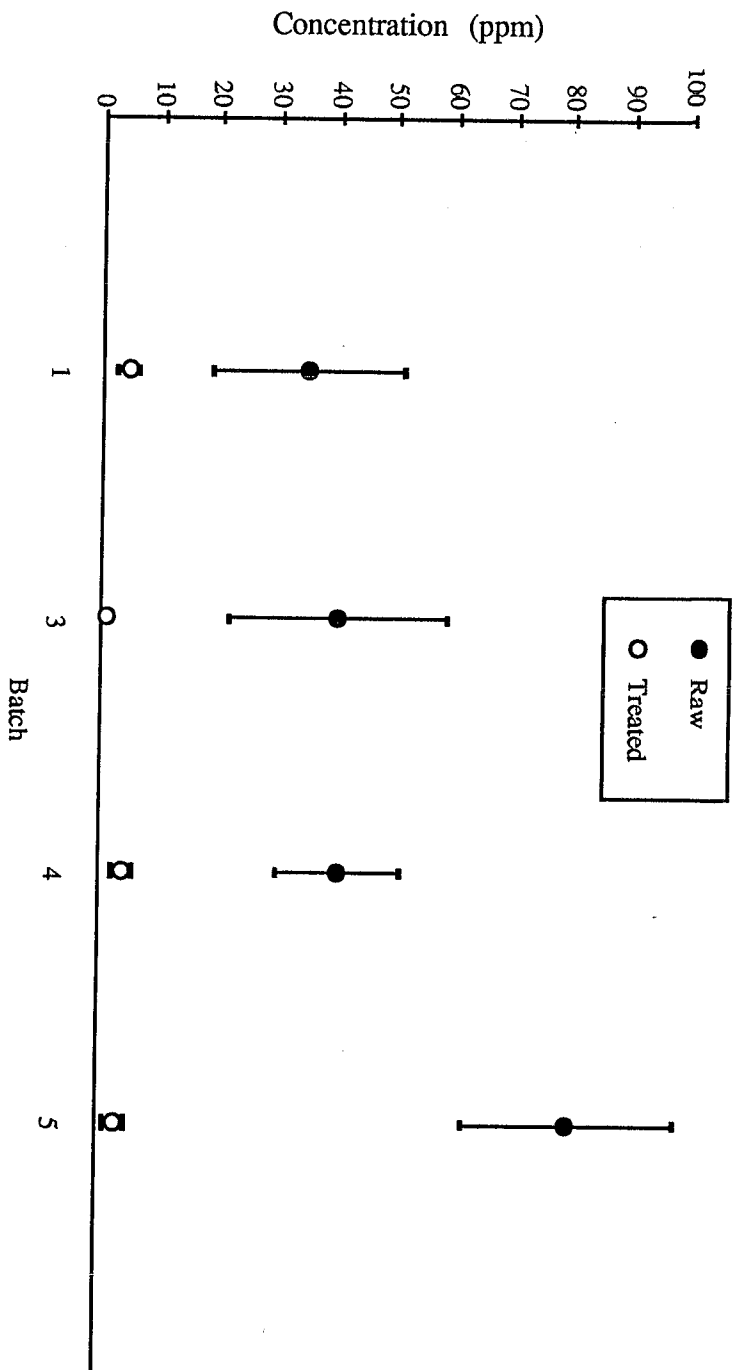


Figure 6-5. TCLP-Distilled Water -- PCP

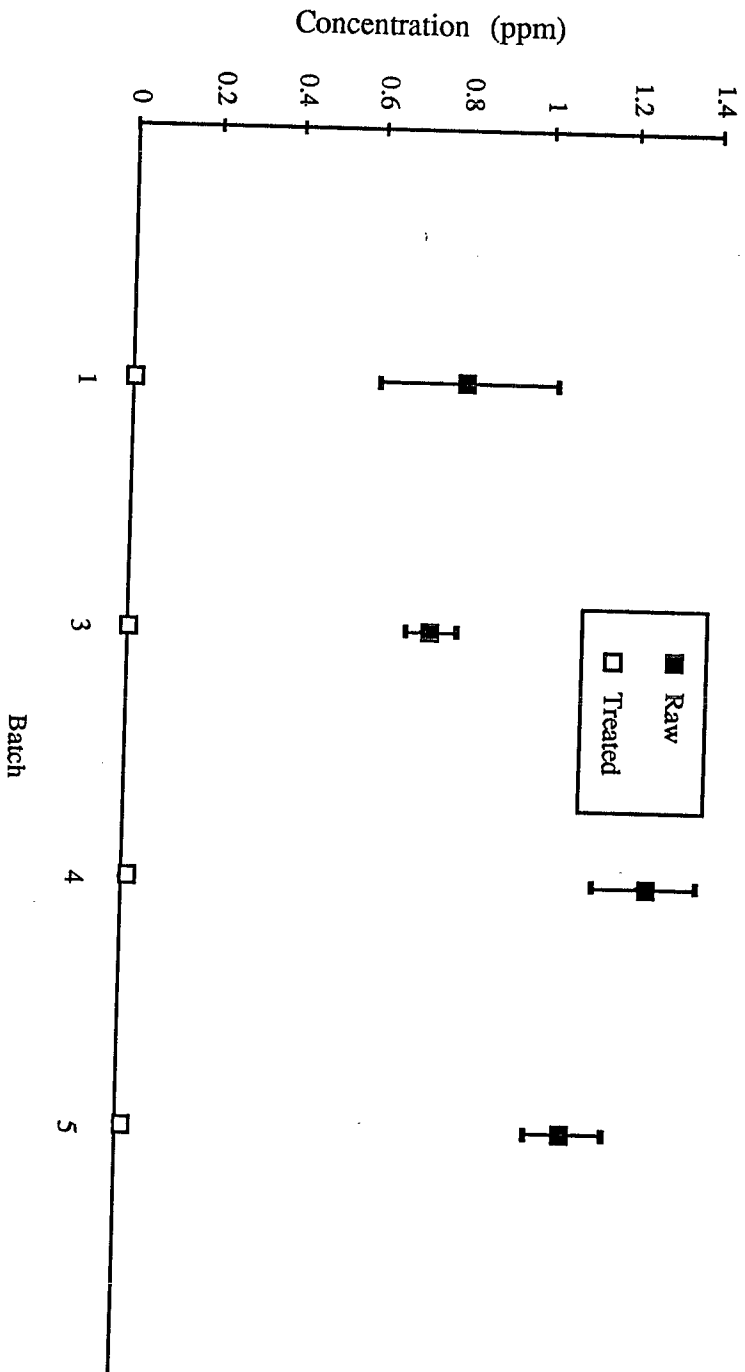


Figure 6-6. TCLP-Distilled Water -- Arsenic

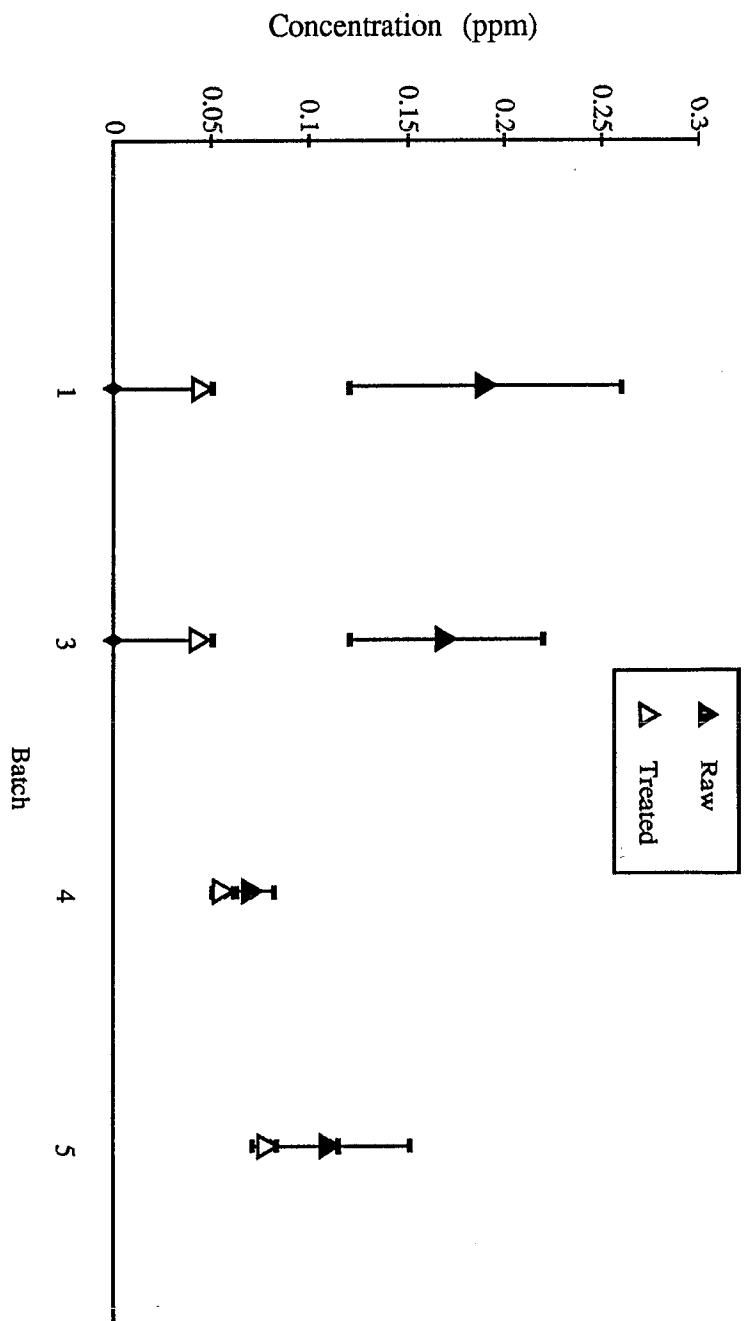


Figure 6-7. TCLP-Distilled Water -- Chromium

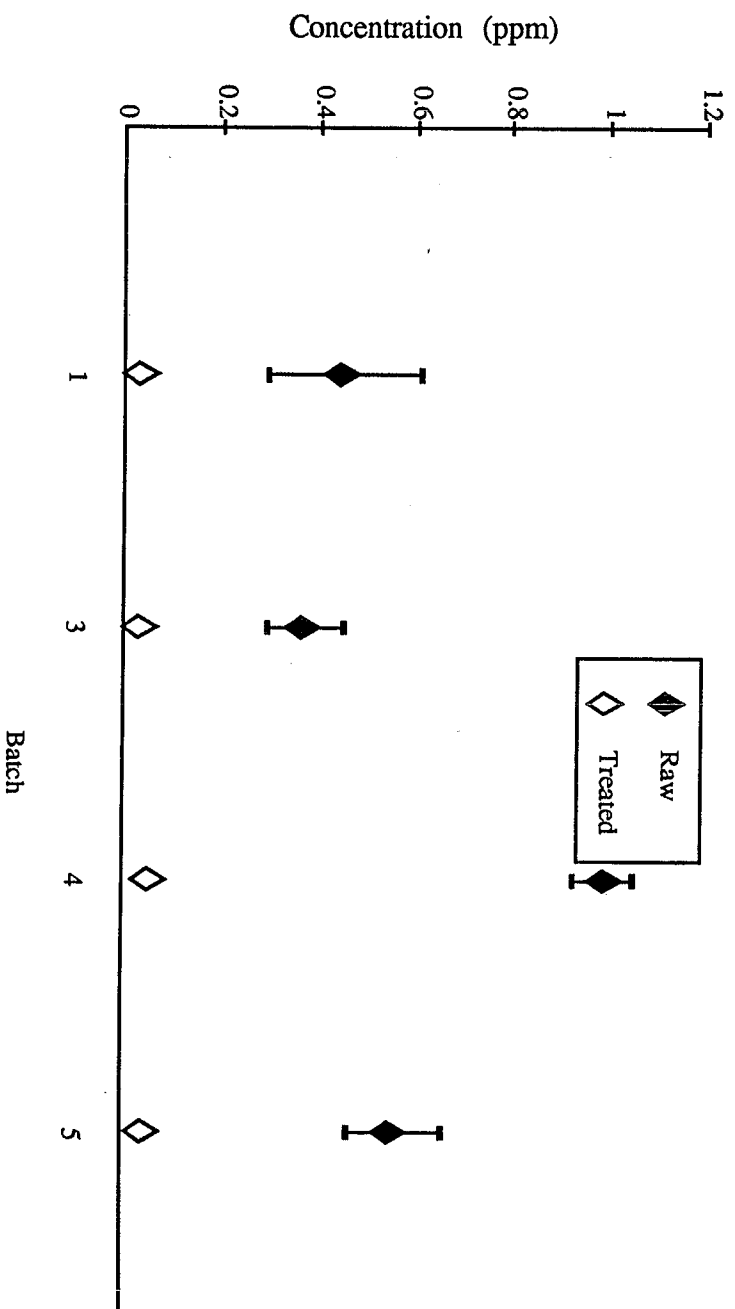


Figure 6-8. TCLP-Distilled Water -- Copper

Copper, also not a target analyte for treatment, showed slightly lower percent reductions under the neutral TCLP-Distilled Water leach than under the acetic acid TCLP, with values ranging from 86 to 90 percent. Copper concentrations for the raw waste leachates were, however, much lower than for the standard acid TCLP.

6.3.3 TCLP-Cage Test (Modified TCLP)

The TCLP-Cage test is a modification of the standard TCLP designed to determine the mobility of contaminants in treated wastes. Its distinguishing characteristic is that the treated sample is not crushed or ground before leaching. Rather, a monolith or bulk sample of solidified/stabilized waste is used for the leaching procedure. All other steps of the TCLP (including selection of extraction fluid) remain the same. Results for the TCLP-Cage test are presented in Table 6-7. Figures 6-9 through 6-12 graphically show the analytical results with open symbols representing average concentrations of the treated wastes. Error bars associated with each symbol represent the standard deviation for each average of three analytical results.

One would expect greater leaching under standard TCLP test conditions than under the conditions of the TCLP-Cage test, due to the increased exposed surface area resulting from crushing the solidified waste; however, this result was not typically the case for the metals. For the metals that were calculated, all but three cases showed higher concentrations in the TCLP-Cage leachates when compared to the TCLP leachates; however, the results were highly variable, especially for chromium and copper in Batches 4 and 5 where standard deviation values exceeded mean values. For PCP, on the other hand, lower leachate concentrations from the treated wastes were obtained for the TCLP-Cage test than the TCLP in all batches, except Batch 3 for which PCP was not detected using either the TCLP or the TCLP-Cage test. All TCLP, TCLP-Cage, and TCLP-Distilled Water leachate concentrations for PCP, arsenic, and chromium from the treated waste samples were below the regulatory levels for the TCLP test. However, leachates from raw waste samples were also below these levels.

6.3.4 CALWET

The CALWET method consists of an extraction similar to the TCLP extraction, except that the CALWET uses a citric (triprotic) acid solution for leaching solid material over a 48-hour period, at a liquid-to-solid ratio of 10 to 1. Following the leaching period, separation of the

Table 6-7. Analytical Results for TCLP-Cage Leachates

Constituent	Concentrations (mg/L)			
	Batch			
	1	3	4	5
PCP	0.077 ^a	<0.250 ^b	0.053 ^a	0.075 ^a
	5.5 ^a	<0.250 ^b	0.033 ^a	0.073 ^a
	0.28	<0.250 ^b	0.25 ^b	NA
Average	2.0 ± 3.1	<0.250	0.11 ± 0.12	0.074 ± 0.001
Arsenic	0.30	0.11 ^c	0.95	0.23
	0.35	0.11	0.55 ^c	0.30
	0.33	0.13	0.72	0.23
Average	0.33 ± 0.025	0.12 ± 0.0115	0.74 ± 0.20	0.25 ± 0.040
Chromium	0.73	0.15	0.10	0.31
	0.33	0.14	0.28	0.11
	0.76	0.13	1.60	1.30
Average	0.61 ± 0.24	0.14 ± 0.010	0.66 ± 0.82	0.57 ± 0.64
Copper	1.9	0.096	0.13	0.91
	0.82	0.10	0.16	0.08
	2.1	0.092	4.00	4.40
Average	1.6 ± 0.69	0.096 ± 0.0040	1.4 ± 2.2	1.8 ± 2.3

NA = Not Analyzed

a = Value reported is an estimated concentration; compound was detected, but at a concentration less than the reporting limit.

b = Laboratory reporting limit; compound was not detected.

c = Post digestion spike for Furnace AA analysis out of control limits (85-115%), while sample absorbance was less than 50% of spike absorbance.

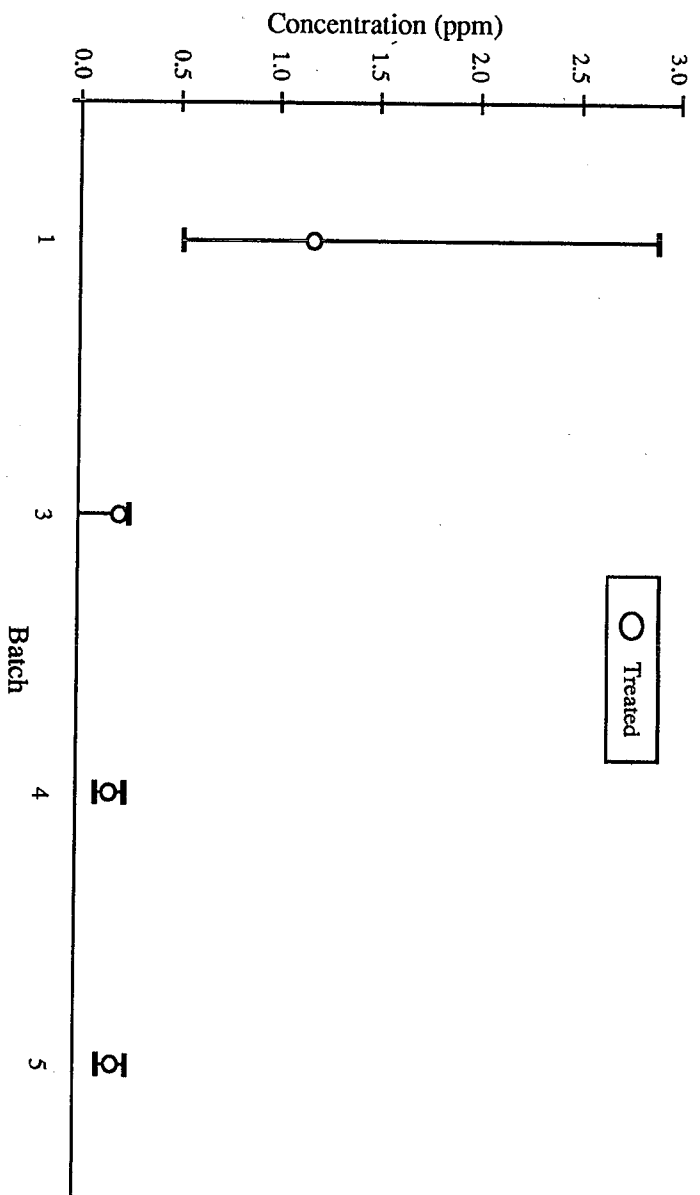


Figure 6-9. TCLP-Cage -- PCP

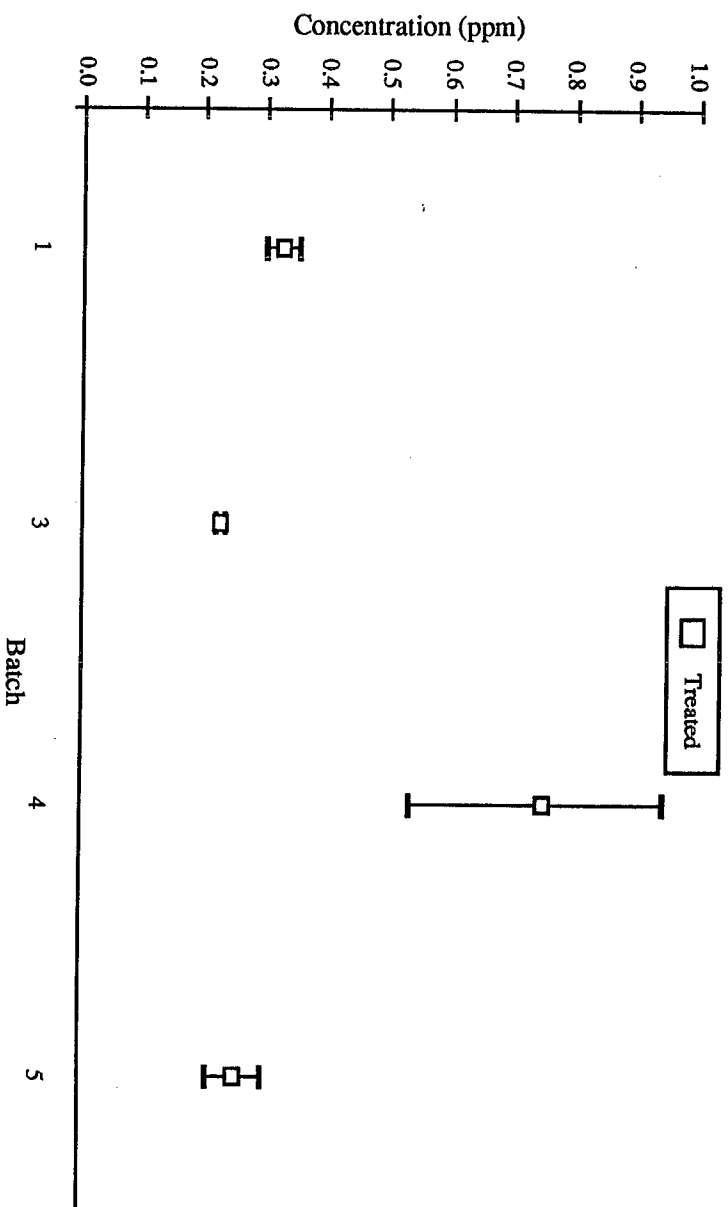


Figure 6-10. TCLP-Cage -- Arsenic

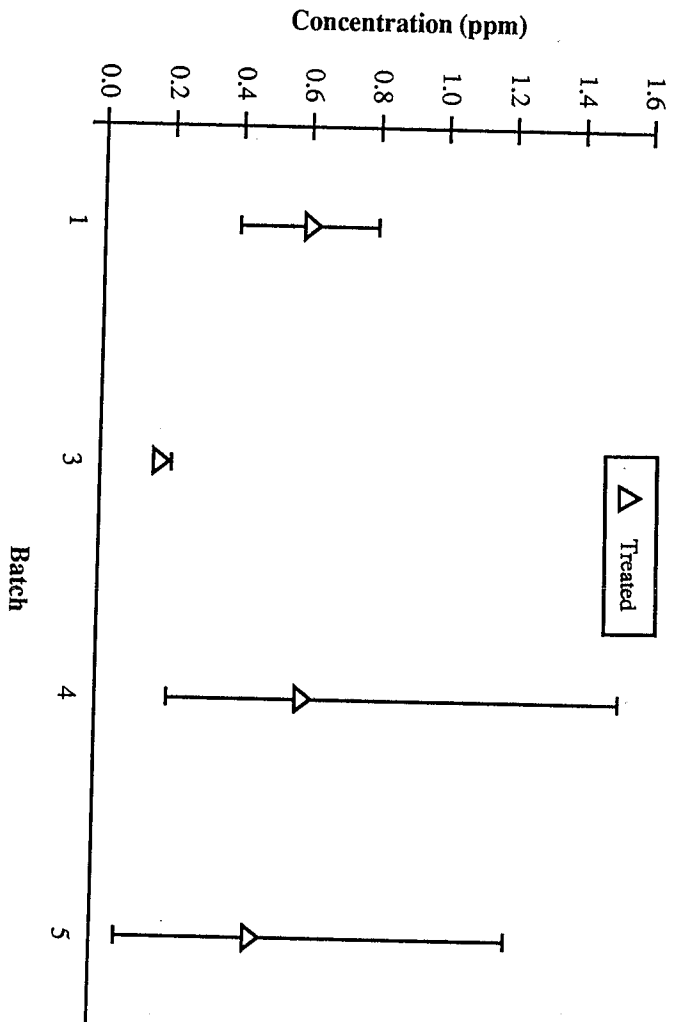


Figure 6-11. TCLP-Cage -- Chromium

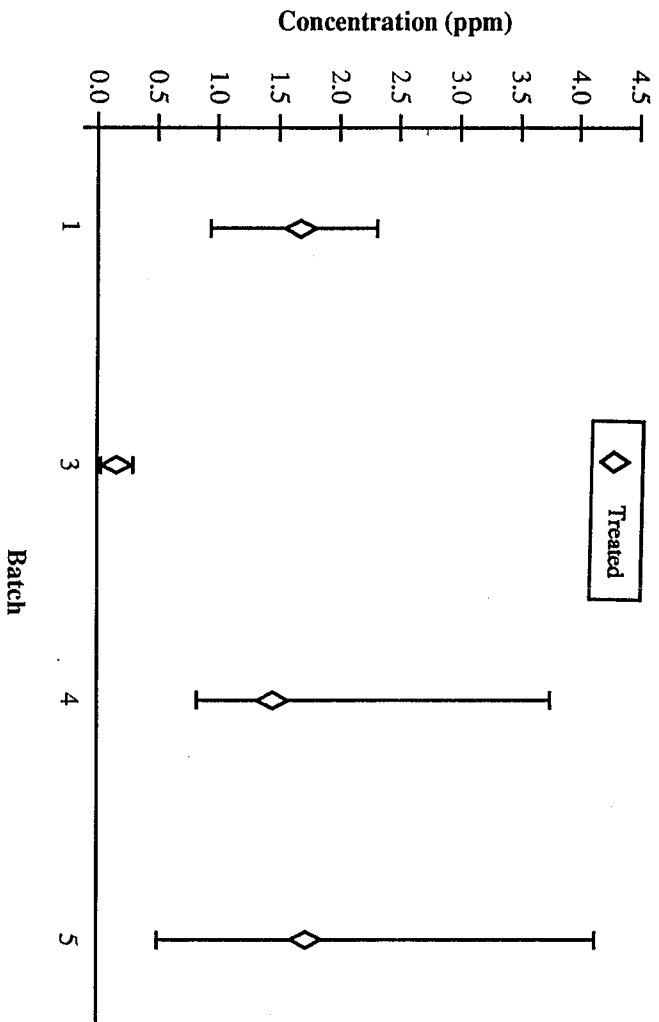


Figure 6-12. TCLP-Cage -- Copper

extracts is achieved by filtration through a 0.45- μ m membrane filter, centrifuging prior to filtration, if necessary. As a result of its greater acid concentration, longer leaching time, and greater buffering capacity, the CALWET is a more aggressive leach procedure than the TCLP. Analytical results for the CALWET are presented in Table 6-8. Figures 6-13 through 6-16 depict the data graphically with solid symbols representing raw waste average concentrations of three analytical results and open symbols representing treated waste average concentrations of three samples for PCP and six samples for each of the metals. Error bars associated with the average concentrations of arsenic in the raw and treated wastes overlap for Batch 5.

Raw waste leachate concentrations of PCP and arsenic were above the Solubility Threshold Limit Concentrations (STLC) of 1.7 and 5 mg/L respectively for the CALWET, a regulatory criteria used by the state of California (see Table 6-3). Leachable chromium concentrations in the raw waste were well below the total chromium STLC of 560 mg/L. Copper in the raw waste leachate from Batch 3 was below the STLC of 25 mg/L and leachate from soil used in Batches 1, 4, and 5 were above this limit.

STC treated wastes were not effectively stabilized based on results from the CALWET procedure. PCP concentrations in the treated waste leachates increase with respect to the raw waste leachate concentration, thus resulting in negative percent reductions ranging from -135 to -1,829 percent. Arsenic values showed mixed results, with percent reductions ranging from -44 to 37 percent. All batches showed reductions in arsenic leachate concentrations after treatment; however, Batches 4 and 5 resulted in average leachate concentrations above the STLC of 5 mg/L. Chromium concentrations in the CALWET leachates, like PCP, increased following the STC treatment, resulting in negative percent reductions ranging from -205 to -375 percent. Chromium leachate concentrations, however, remained below the STLC limit of 560 mg/L. Copper concentrations were slightly reduced upon treatment; Batch 1 was brought below the STLC of 25 mg/L for the treated CALWET leachates. Batch 3 was below this threshold prior to treatment. Overall, percent reductions for copper ranged from 2 to 22 percent.

6.3.5 ANS 16.1 (Modified)

The ANS 16.1 leach test is used by the nuclear industry to identify the mechanisms that control leaching and to indicate the amount of possible leaching from a monolithic solid. This leach test is used to simulate contact with rapidly flowing groundwater by using a static sequential leaching method. A 10-week modification of ANS 16.1 for solidified wastes was used to approximate leaching from treated waste by demineralized water that flowed around the sample.

Table 6-8. Analytical Results for CALWET

Constituent: PCP			
Batch	Concentrations (mg/L)		Average Percent Reduction ^{a,b}
	Raw Waste	Treated Waste	
1	1.7	13	-842
	2.4	10	
	2.8	14	
Average	2.3 ± 0.56	12 ± 2.1	
3	2.3	2.8	-135
	2.4	4.8 ^a	
	3.1	2.8	
Average	2.6 ± 0.44	3.5 ± 1.15	
4	3.1	34 ^a	-1,493
	3.2	26	
	3.3	26	
Average	3.2 ± 0.10	29 ± 4.6	
5	2.7	30	-1,829
	3.0	30	
	2.9	35 ^a	
Average	2.9 ± 0.15	32 ± 2.9	

Table 6-8. Analytical Results for CALWET (continued)

Constituent: Arsenic			
Batch	Concentrations (mg/L)		Average Percent Reduction ^{a,b}
	Raw Waste	Treated Waste	
1	11	3.6	37
	13	5.2	
	14	5.2	
	NA	5.9	
	NA	4.4	
	NA	3.1	
Average	13 ± 1.5	4.6 ± 1.1	
3	9.4	6.8	9
	8.3	5.2	
	8.6	4.2	
	NA	4.4	
	NA	2.6	
	NA	4.1	
Average	8.8 ± 0.56	4.55 ± 1.4	
4	28	23	-44
	29	26	
	29	21	
	NA	23	
	NA	23	
	NA	24	
Average	29 ± 0.58	23 ± 1.6	
5	29	18	-25
	29	16	
	26 ^a	19	
	NA	21	
	NA	20	
	NA	26	
Average	28 ± 1.7	20 ± 3.4	

Table 6-8. Analytical Results for CALWET (continued)

Constituent: Chromium			
Batch	Concentrations (mg/L)		Average Percent Reduction ^{a,b}
	Raw Waste	Treated Waste	
1	2.9	3.9	-205
	3.1 ^a	5.1	
	2.9	6.0	
	NA	5.7	
	NA	6.1	
	NA	4.1	
Average	3.0 ± 0.115	5.15 ± 0.96	
3	2.1	4.4	-224
	2.2	3.9	
	1.9	3.6	
	NA	3.8	
	NA	3.3	
	NA	3.8	
Average	2.1 ± 0.15	3.8 ± 0.36	
4	7.2	19	-375
	6.5	19	
	7.6	19	
	NA	18	
	NA	18	
	NA	21 ^a	
Average	7.1 ± 0.56	19 ± 1.1	
5	6.9	18	-355
	7.2	18	
	6.6	18	
	NA	17	
	NA	17	
	NA	20 ^a	
Average	6.9 ± 0.30	18 ± 1.1	

Table 6-8. Analytical Results for CALWET (continued)

Constituent: Copper			
Batch	Concentrations (mg/L)		Average Percent Reduction ^{a,b}
	Raw Waste	Treated Waste	
1	27	11	22
	28	12	
	28	13	
	NA	14	
	NA	14	
	NA	10	
Average	28 ± 0.58	12 ± 1.6	
3	18	8.1	12
	18	9.4	
	17	8.8	
	NA	9.0	
	NA	8.6	
	NA	9.1	
Average	18 ± 0.58	8.8 ± 0.45	
4	58	32	2
	54	33	
	61	31	
	NA	31	
	NA	31	
	NA	33	
Average	58 ± 3.5	32 ± 0.98	
5	63	33	6
	65	33	
	56	32	
	NA	32	
	NA	33	
	NA	35 ^a	
Average	61 ± 4.7	33 ± 1.1	

Table 6-8. Analytical Results for CALWET (continued)

NA = *Not analyzed*

$$a = \text{Percent Reduction} = \left[1 - (1 + \text{Additives Ratio}) \times \frac{\text{Concentration of Treated Waste}}{\text{Concentration of Raw Waste}} \right] \times 100.$$

b = *The additives ratio is the weight of additives, including water of hydration, divided by the weight of raw wastes. Values are 0.761, 0.764, 0.776, and 0.746 for Batches 1, 3, 4, and 5, respectively.*

q = *Fails Q test (Shoemaker, 1974); value may be disregarded with 90% confidence (although it has been used to calculate the average value).*

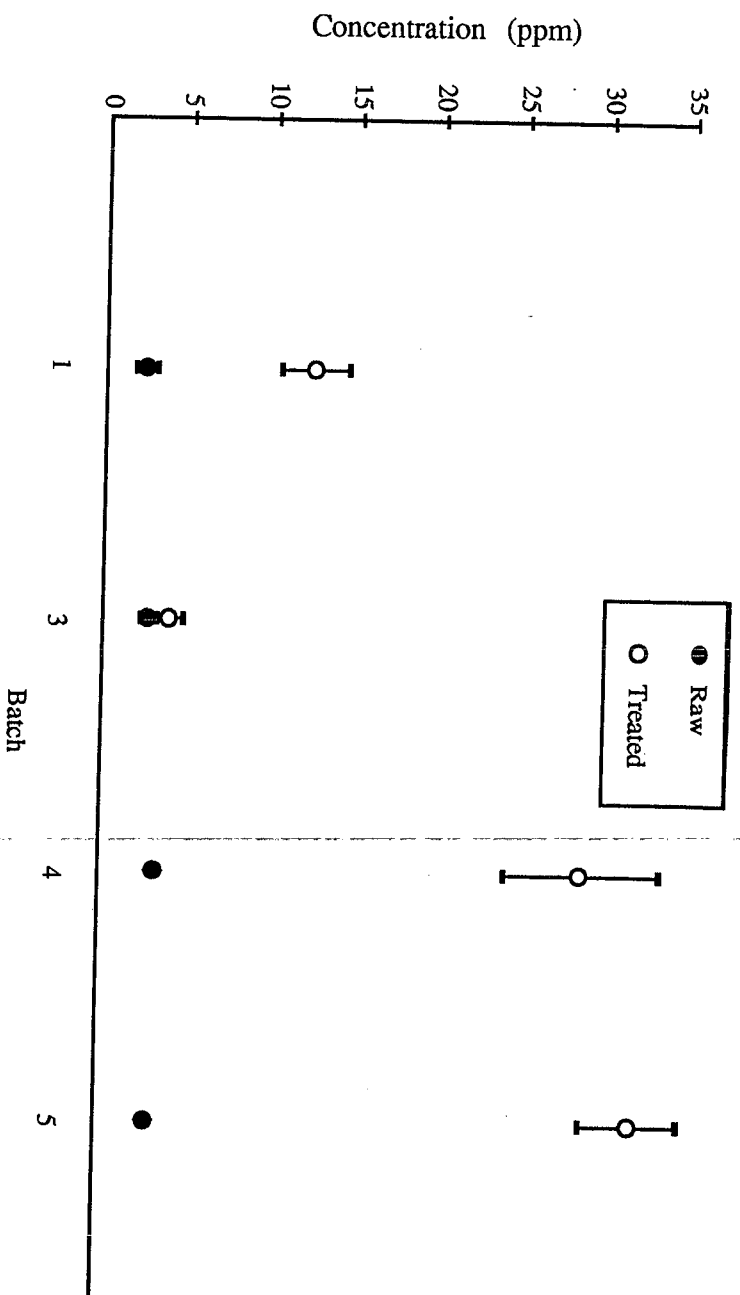


Figure 6-13. CALWET -- PCP

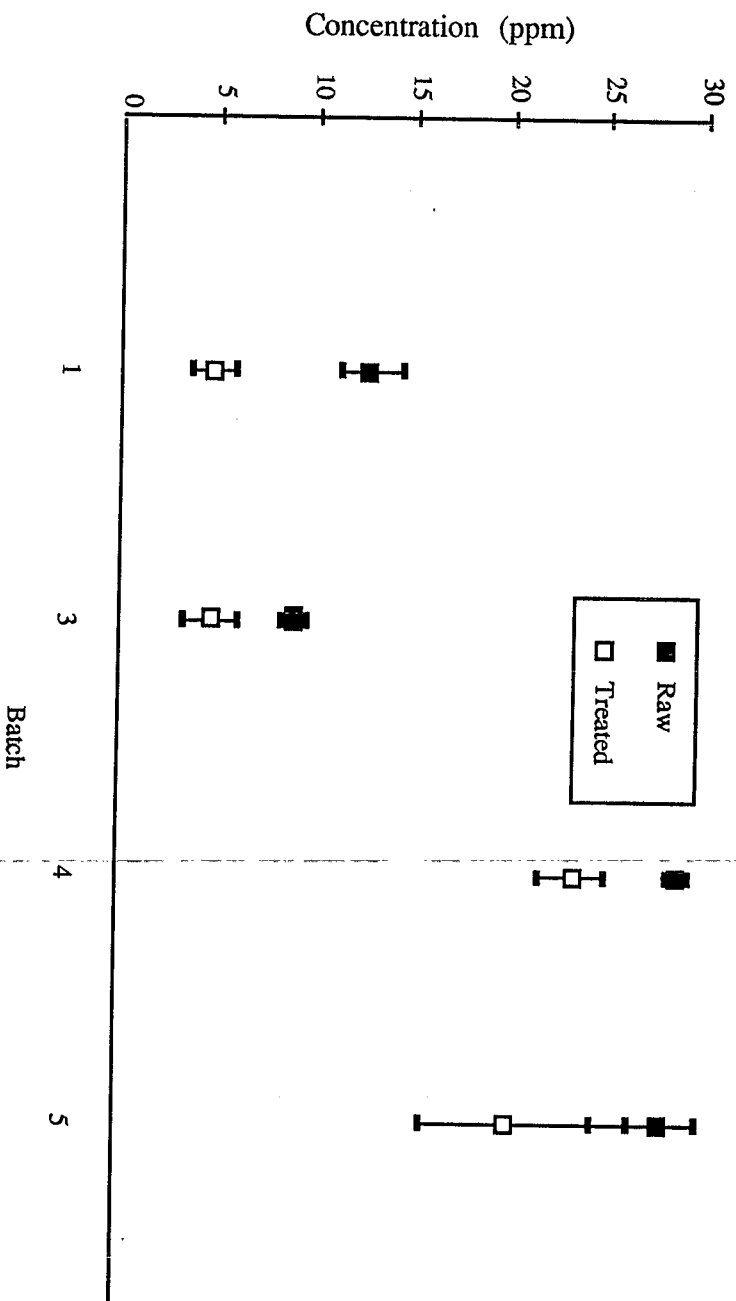


Figure 6-14. CALWET -- Arsenic

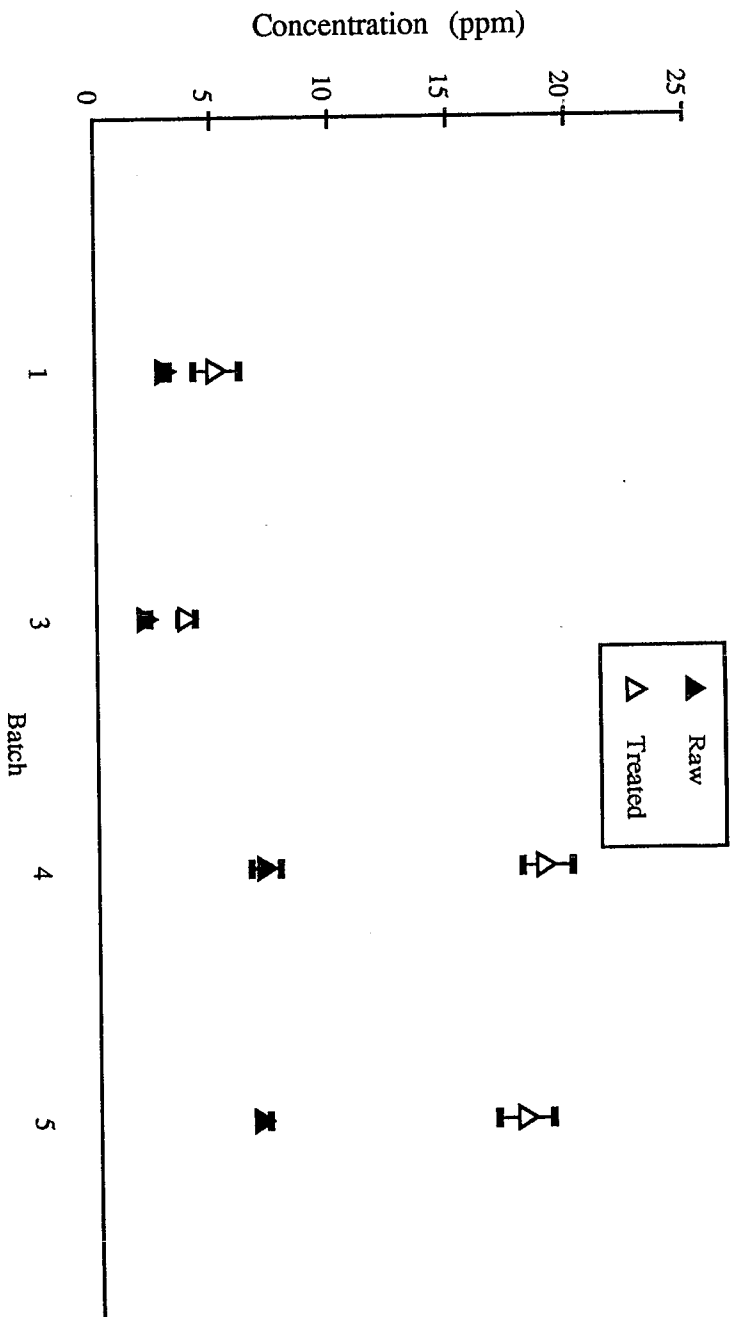


Figure 6-15. CALWET -- Chromium

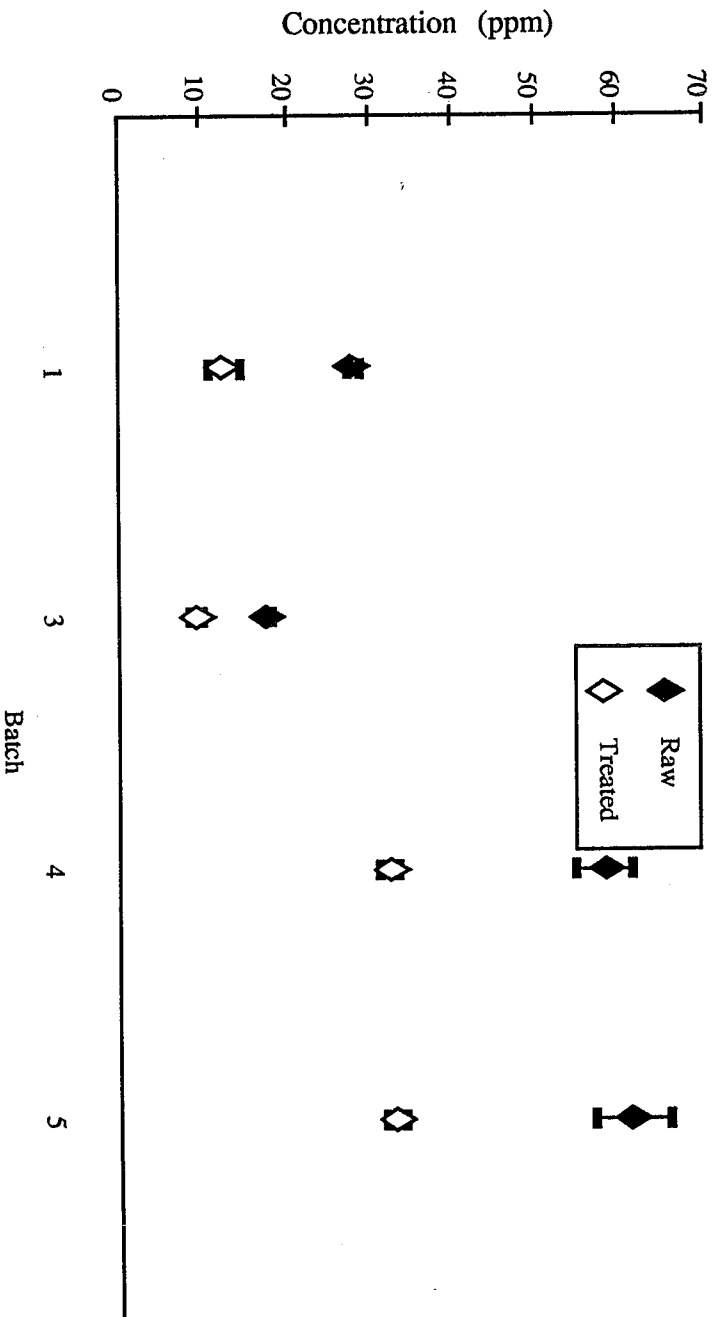


Figure 6-16. CALWET -- Copper

(The initial leach periods were lengthened because previous experience indicated that solidified matrices were barely wetted during the standard ANS 16.1 leach periods.) Samples were leached without agitation using demineralized water with an electrical conductivity of less than 5 μ mhos/cm at 25°C and a total organic carbon concentration of less than 3 milligrams per liter. The samples were submerged in the water for specified time intervals, after which the leachate was collected and set aside for analysis. The sample was then re-extracted in another aliquot of demineralized water. The cycle was repeated to obtain five leachates for each sample over the 10-week period. Because the material was not ground as part of the test, the ANS 16.1 leach test provides information on the ability of the treated solid waste material to contain contaminants.

Several factors should be considered in reviewing the results of this test. First, the test is normally conducted on monolith-like solids generated by solidification/stabilization processes in the nuclear industry. Such solids may differ in both chemical and physical characteristics from those generated by the STC contaminated soil process. In addition, the test was not performed on the raw wastes because of the friable nature of the contaminated soil.

PCP results were only slightly higher than minimum reporting limits, and only negligible amounts of each of the three selected metals -- arsenic, chromium, and copper -- were detected in the leachate extractions after each test period (Table 6-9). Except for chromium after the second test period, all metal values were at or near the minimum reporting limits.

As part of the ANS 16.1 leachability test, the leachability index (LI) is recommended as a standard method for evaluating solidified waste forms:

$$LI = \log \left(\frac{B}{D_e} \right)$$

where D_e is the effective diffusion coefficient (cm^2/sec), and B is constant ($1 \text{ cm}^2/\text{sec}$). The diffusive strength for a given analyte within the solidified matrix is regulated by its chemical activity (that is, effective concentration) as well as the permeability of the waste form. The determination of a diffusion coefficient based on Fick's Law has been documented in the ANS 16.1 leachability test for solidified low-level, radioactive wastes. The equation for a semi-infinite medium diffusion model with uniform initial concentration and zero surface concentration is:

Table 6-9. ANS 16.1 Leachate Analyses for STC-Treated Waste (Batch 3)

Constituent	Concentrations (mg/L)				
	Day 14	Day 28	Day 42	Day 56	Day 70
PCP	0.37	0.078	0.13	0.18	0.080
	0.21	0.15	0.17	0.088	0.094
	0.15	0.16	0.090	0.13	0.12
	0.21	0.11	0.11	0.13	0.12
Average	0.235 ± 0.094	0.125 ± 0.038	0.125 ± 0.034	0.13 ± 0.038	0.10 ± 0.020
Arsenic	<0.004	0.0052 ^q	<0.004	<0.004	<0.004
	<0.004	<0.004	<0.004	<0.004	<0.004
	<0.004	<0.004	<0.004	<0.004	<0.004
	<0.004	<0.004	<0.004	<0.004	<0.004
Average	<0.004	0.004 ± 0.0006	<0.004	<0.004	<0.004
Chromium	<0.01	0.16 ^q	0.036 ^q	0.011	<0.01
	<0.01	0.040	0.013	<0.01	<0.01
	<0.01	0.010	<0.01	<0.01	<0.01
	<0.01	<0.01	<0.01	<0.01	<0.01
Average	<0.01	0.055 ± 0.071	0.017 ± 0.0125	0.010 ± 0.0005	<0.01
Copper	0.027	<0.02	0.035 ^q	<0.02	0.02
	0.022	<0.02	<0.02	<0.02	<0.02
	<0.02	0.02	<0.02	<0.02	<0.02
	<0.02	<0.02	<0.02	<0.02	<0.02
Average	0.022 ± 0.0033	<0.02	0.024 ± 0.0075	<0.02	<0.02
pH	11.6	11.6	11.0	11.2	11.0

q = Fails Q test (Shoemaker, 1974), value may be disregarded with 90% confidence (although it has been used to calculate the average value).

$$D_e = \frac{\pi}{4} \left(\frac{V}{S} \right)^2 \left(\frac{\sum A_n}{A_o} \right)^2 \frac{1}{t_n^{1/2}}$$

where:

- A_n = Contaminant loss during leaching period, n (mg)
- A_o = Initial amount of contaminant in specimen (mg)
- V = Volume of specimen (cm³)
- S = Surface area of specimen (cm²)
- t_n = Time (sec)
- D_e = Effective diffusion coefficient (cm²/sec)

The linearity of a plot of $\left(\frac{\sum A_n}{A_o} \right)$ versus $t_n^{1/2}$ for a given analyte indicates that the release of an analyte from the solid to the aqueous solution is limited by diffusion. The leachability index is thus used to compare the relative mobility of contaminants on a uniform scale. This scale varies from very mobile, for a leachability index of 5 ($D_e = 10^{-5}$ cm²/sec) or less, to immobile, for values of 15 ($D_e = 10^{-15}$ cm²/sec) or greater. The leachability index was calculated from the results for PCP, arsenic, chromium, and copper. The values of the leachability indices for the STC-treated wastes are as follows:

PCP	-	LI	=	10.8
Arsenic	-	LI	=	12.2
Chromium	-	LI	=	11.0
Copper	-	LI	=	10.9

These values are well above the Nuclear Regulatory Commission's minimum leachability index standard of 6. However, the standard for this index has no specific basis in terms of human or environmental risk or toxicity.

6.3.6 Summary of Leach Tests

PCP showed considerably greater mobility under neutral leaching conditions in the raw waste than under acid TCLP or CALWET conditions. The treated wastes, however, showed very similar leachability of PCP under both acid and neutral TCLP conditions. Arsenic appeared to be well stabilized by the STC treatment under neutral leach conditions. Acid leaching, however, as

under TCLP and CALWET conditions, resulted in increased arsenic mobility in the treated waste relative to neutral leaching conditions. Chromium was generally rendered immobile by STC's treatment process under neutral leaching conditions. In all acid leachates, chromium concentrations for the treated wastes were significantly higher than for the raw wastes. The raw waste, however, showed no difference in leachability of chromium between acid and neutral TCLP conditions. Copper in the raw waste was slightly less mobile under neutral conditions than under acid leaching conditions. Treated waste leachate concentrations showed copper to be essentially immobile under both acid and neutral TCLP conditions.

6.4 CHEMICAL ANALYSES

The raw and treated wastes were analyzed for organic and inorganic constituents using TWA. In addition, chemical characterization of the raw and treated waste included pH, Eh, loss on ignition, and neutralization potential. Results are also presented for a STC solidified reagent mixture blank, as well as the sand used for the blank, and the process water used to mix each treatment batch. Oil and grease extracts were also analyzed for both the raw and treated wastes.

6.4.1 Organics

Pentachlorophenol (PCP) was the main organic contaminant of concern at the SPT site. Based on information from the site treatability study, replicate samples were analyzed for other constituents, including semivolatile organics such as tetrachlorophenol (TCP), phenanthrene, naphthalene, and phenol. Concentrations of these constituents were found to be negligible, and therefore are not included in this report. TCP was, however, detected in an auxiliary study where a modified TCLP method (using a borate buffer solution at pH 12) was performed followed by analysis for PCP and TCP.

Table 6-10 presents TWA (SW 846 Method 8270) data for PCP in the raw and treated wastes. Multiple test results are shown for both the raw and treated wastes, since multiple samples were obtained of raw and treated waste for each batch. The variance in concentrations for each batch represents differences existing between discrete samples within that batch. For comparison purposes, average values have been used for each batch of raw and treated waste because averages may provide a more reliable indicator of overall treatment effectiveness, as there is no justification for matching any single raw waste value to any single treated waste value. Figure 6-17 graphically shows the average concentrations of PCP for each batch, including standard deviation values shown as error bars.

Table 6-10. Total Waste Analyses (TWA) for PCP

Batch	Concentrations (mg/kg)						
	Raw Waste	Treated Waste Data Options					
		1	2	3	4	5	6
1	2,600	200 ^c	200 ^c	200 ^c	200 ^c	24 ^d	24 ^d
	1,500	82 ^c	82 ^c	82 ^c	82 ^c	24 ^d	24 ^d
	2,000	140 ^c	140 ^c	140 ^c	140 ^c	24 ^d	24 ^d
	2,600	480 ^d	NA	106 ^c	0	NA	200 ^c
	3,400	480 ^d	NA	106 ^c	0	NA	82 ^c
	2,000	480 ^d	NA	106 ^c	0	NA	140 ^c
Average	2,350 ± 660	310 ± 190	140 ± 59	120 ± 42	70 ± 86	24 ± 0	82 ± 74
% Reduction ^{a,b}	---	77	89	91	95	98	94
3	1,700 ^f	34 ^c	34 ^c	34 ^c	34 ^c	14	14
	2,200 ^c	480 ^{d,g}	NA	106 ^c	0	8 ^d	8 ^d
	2,200 ^f	480 ^{d,g}	NA	106 ^c	0	20	20
	2,000	480 ^{d,g}	NA	106 ^c	0	NA	34 ^c
	2,200	54 ^{c,g}	54 ^c	54 ^c	54 ^c	NA	54 ^c
	1,600	480 ^{d,g}	NA	106 ^c	0	NA	NA
Average	2,000 ± 270	330 ± 225	44 ± 10	85 ± 33	15 ± 24	14 ± 6	26 ± 18
% Reduction ^{a,b}	---	64	96	92	99	99	98
4	6,400 ^f	480 ^{d,g}	NA	106 ^c	0	58	58
	6,800	480 ^{d,g}	NA	106 ^c	0	85	85
	8,900	480 ^{d,g}	NA	106 ^c	0	330 ^f	330 ^f
	7,300	480 ^{d,g}	NA	106 ^c	0	NA	220 ^c
	9,000	480 ^{d,g}	NA	106 ^c	0	NA	NA
	7,800	200 ^c	200 ^c	200 ^c	200 ^c	NA	NA
Average	7,700 ± 1,100	430 ± 110	200 ± 0	120 ± 38	33 ± 82	160 ± 150	170 ± 130
% Reduction ^{a,b}	---	90	95	97	99	96	96

Table 6-10. Total Waste Analyses (TWA) for PCP (continued)

Batch	Concentrations (mg/kg)						
	Raw Waste	Treated Waste Data Options					
		1	2	3	4	5	6
5	6,300	480 ^{d,g}	NA	106 ^e	0	47 ^e	47 ^e
	10,000	480 ^{d,g}	NA	106 ^e	0	49 ^e	49 ^e
	9,600	400 ^e	400 ^e	400 ^e	400 ^e	230 ^f	203 ^f
	8,300	370 ^e	370 ^e	370 ^e	370 ^e	NA	400 ^e
	7,000	480 ^{d,g}	NA	106 ^e	0	NA	370 ^e
	8,700	NA	NA	NA	NA	NA	NA
Average	8,300 ± 1,400	440 ± 53	385 ± 15	220 ± 150	150 ± 210	110 ± 105	210 ± 170
% Reduction ^{a,b}	---	91	92	95	97	98	96

NA = Not Analyzed

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$$a = \text{Percent Reduction} = \left[1 - (1 + \text{Additives Ratio}) \times \frac{\text{Concentration of Treated Waste}}{\text{Concentration of Raw Waste}} \right] \times 100.$$

b = The additives ratio is the weight of additives, including water of hydration, divided by the weight of raw wastes. Values are 0.761, 0.764, 0.776, and 0.746 for Batches 1, 3, 4, and 5, respectively.

c = Value reported is an estimated concentration; compound was detected but at a concentration less than the reporting limit.

d = Laboratory reporting limit; compound was not detected.

e = Estimated concentration using twice the method detection limit.

f = Reported concentration was calculated using a greater dilution than the primary analysis.

g = Sample failed surrogate recovery criteria.

Option 1 = Percent reduction calculation based on using laboratory reporting limits for samples for which PCP was not detected.

Option 2 = Percent reduction calculation based on using actual measured or laboratory estimated concentrations only; samples for which PCP was not detected were not included in calculation.

Option 3 = Percent reduction calculation based on using twice the method detection limit for samples for which PCP was not detected.

Option 4 = Percent reduction calculation based on using values of zero for samples for which PCP was not detected.

Option 5 = Percent reduction calculation based on using PCP concentrations from modified EPA method 8270.

Option 6 = Percent reduction calculation based on using PCP concentrations from modified EPA method 8270 and laboratory estimated concentrations from original analysis.

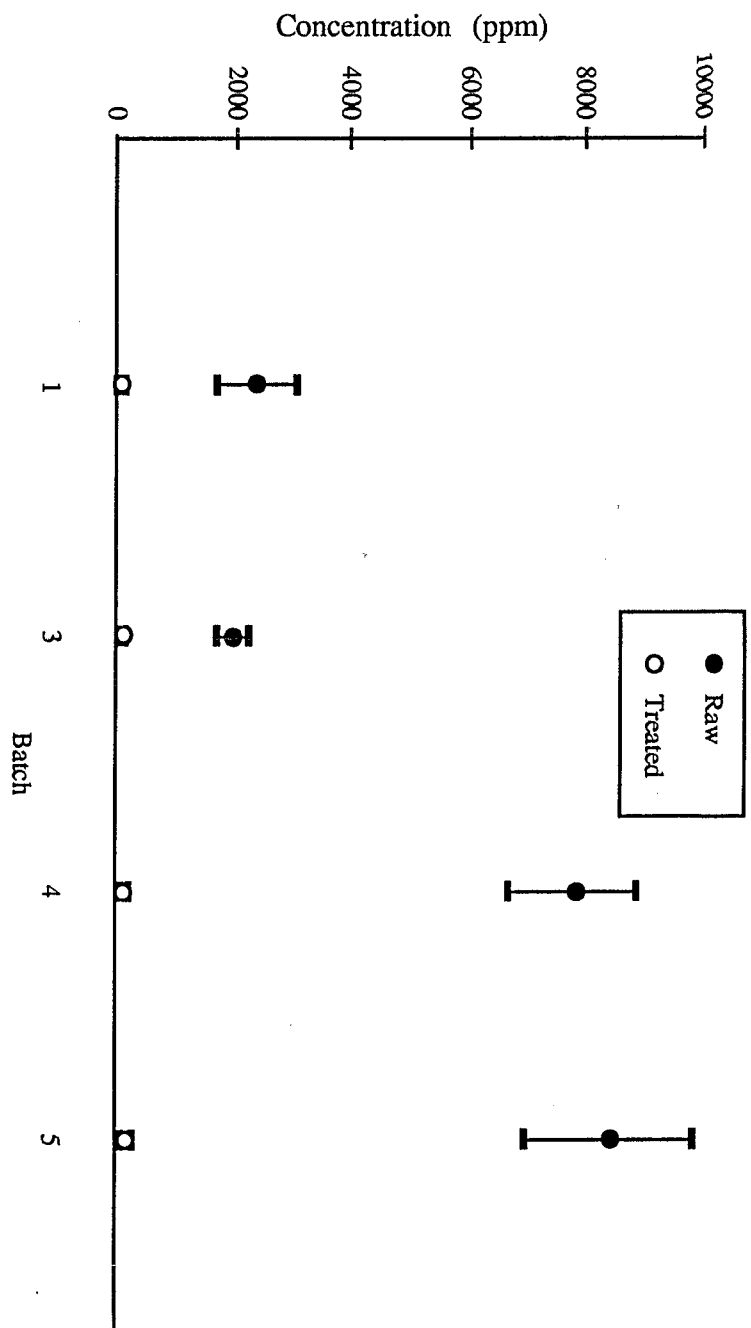


Figure 6-17. TWA -- PCP

As is evident from the treated waste data, PCP was not detected in many samples, although observable (estimated) concentrations were detected at levels significantly below the reporting limit. Reporting limits were determined by multiplying the method detection limit by a dilution factor specific for each analyte. Establishing reporting limits for PCP by TWA of treated samples presented problems because of the high concentrations of PCP and the correspondingly large dilution factors necessary to reach the analytical method quantitation range for PCP (approximately 100 to 200 $\mu\text{g/kg}$).

Several options for calculating percent reductions of PCP for TWA are presented in Table 6-10. Percent reductions for Option 1 are calculated using the laboratory reporting limits for all samples for which PCP was not detected. Option 2 uses actual measured concentrations or laboratory estimated concentrations only for calculating percent reductions. Percent reductions for Option 3 were calculated using "assigned" concentrations of twice the method detection limit (that is, $2 \times 53 = 106 \text{ mg/kg}$) for the samples for which PCP was not detected. Option 4 uses "assigned" values of zero for such samples in calculating percent reductions.

To obtain PCP data with lower reporting limits, additional samples were analyzed by TWA, using different dilution steps than had been used when analyzing the initial raw waste and treated samples for PCP quantitation. The results of these analyses are presented in Option 5. Finally, Option 6 presents calculated percent reductions using the concentrations from the additional analysis, plus estimated concentrations from the original analysis. Assigning nondetects a value of twice the detection limit is conservative in light of the precision of the method for PCP (± 50 percent), and the observable concentrations detected near or below the method detection limit. (Note: Because the method detection limit is defined on an aggregate basis, it is not unusual for individual laboratories to achieve significantly lower instrument noise than is assumed in the method). In addition, the results of the additional analysis by TWA, presented in Option 5, show PCP concentrations in treated waste that are consistently well below twice the method detection limit.

The extractable concentrations of PCP, as measured by TWA, were reduced by 89 to 99 percent (Options 2 and 5), indicating that the STC treatment was effective in stabilizing the organic component of the SPT waste. The STC process, however, was not capable of reducing the PCP concentrations below the required total threshold limit concentration (TTLC) for the State of California, but the concentrations of PCP were below the federal regulatory threshold limit for PCP as measured by the TCLP.

Additional Studies: pH and Temperature

Additional studies were performed to evaluate PCP containment under various pH extremes. The first study involved PCP extraction and analysis after partially dissolving the treatment matrix. This alternate TWA procedure used a combination of SW 846 Methods 3510 and 3550 to prepare the sample for analysis. In order to dissolve some of the cementitious portion of the treatment matrix and disrupt the structure of the silicate or organophilic silicate portion of the matrix, the material was crushed to pass 1 mm, then acidified with HCl to pH<2. The acidified aqueous phase was then subjected to solvent extraction according to Method 3510. After removal of the aqueous phase, the residual solids were subject to solvent extraction according to Method 3550. The organic phases from each procedure were analyzed according to Method 8270.

Table 6-11 shows PCP analysis results obtained from this low-pH study for both raw and treated wastes. Raw waste concentrations are totals from both the acid and solid phases of the extraction procedure. The results were consistent with average raw waste concentrations obtained during the pilot-scale demonstration. Batches 1, 3, and 5 resulted in slightly lower concentrations, but still within 1 standard deviation of the mean obtained in original raw waste TWA. Batch 4 results of this low-pH study showed greater concentrations of PCP than the original Method 8270 analysis. Treated wastes show reductions in PCP concentrations from those in the raw wastes; however, PCP concentrations in the treated waste are much higher than PCP concentrations obtained from the conventional TWA method. Acid treatment disrupted the treatment matrix, as evidenced by color changes and turbidity in the extraction liquids. The conventional analysis indicated treatment-associated percent reductions of 91 to 99 percent. The modified analysis used in this study indicated substantially lower percent reductions. Acid disruption of the matrix allowed recovery of 42 to 67 percent of the extractable PCP from the raw waste. The environmental relevance of these results is not known, because the treated waste is unlikely to contact either organic solvents or strong acids in the disposal setting. The missing fraction of the PCP (33 to 58 percent) may still be bound to the treatment matrix, or may have decomposed.

Table 6-11. TWA for PCP Using Modified Acid Extraction

Batch	Concentrations (mg/kg)		Percent Reduction ^{a,c}
	Raw Waste	Treated Waste ^a	
1	2,240	830	35
3	1,310	500	33
4	8,730	2,060	58
5	6,290	1,990	45

a = Results reported as the mean of duplicate samples.

$$b = \text{Percent Reduction} = \left[1 - (1 + \text{Additives Ratio}) \times \frac{\text{Concentration of Treated Waste}}{\text{Concentration of Raw Waste}} \right] \times 100.$$

c = The additives ratio is the weight of additives, including water of hydration, divided by the weight of raw wastes. Values are 0.761, 0.764, 0.776, and 0.746 for Batches 1, 3, 4, and 5, respectively.

Table 6-12 shows analytical results for an additional study performed to determine whether organics might be leached from the raw and treated waste under alkaline leaching conditions. The analyses were conducted using a modified TCLP at pH 12 using a 0.1 M borate buffer solution. The waste amount, leachate volume, and leaching time remained the same as for the standard TCLP. PCP was considerably more mobile under alkaline leaching conditions than under neutral or standard TCLP conditions, and showed percent reductions that ranged from 90 to 96 percent. TCP was not detected in the majority of the raw and treated waste samples from the SPT site. The small amounts of TCP detected in this study appear to be stabilized with percent reductions ranging from 86 to 91 percent reduction.

An additional study was performed to investigate the possibility of PCP volatilizing or being steam stripped as a result of the heat of hydration caused by the stabilization/solidification treatment processes. The heat of hydration generated during solidification using Portland cement may be expected to reach as much as 60° to 70°C. (The STC treatment reagents contain neither Portland cement nor any other exothermic reagents.)

Table 6-12. Analytical Results for TCLP-pH 12 Leachates

Constituent	Batch	Concentrations (mg/L) ^c		Percent Reduction ^{a,b}
		Raw Waste	Treated Waste	
PCP	1	110 ^d	6.2	90
	3	88 ^d	1.9 ^d	96
	4	320 ^d	13 ^d	93
	5	320 ^d	17 ^d	91
TCP	1	1.8 ^e	ND	NC
	3	1.4 ^e	0.11 ^e	86
	4	5.4 ^e	0.29 ^e	91
	5	6.1 ^e	0.43 ^e	88

ND = Not detected

NC = Not calculable

$$a = \text{Percent Reduction} = \left[1 - (1 + \text{Additives Ratio}) \times \frac{\text{Concentration of Treated Waste}}{\text{Concentration of Raw Waste}} \right] \times 100.$$

b = The additives ratio is the weight of additives, including water of hydration, divided by the weight of wastes. Values are 0.761, 0.764, 0.776, and 0.746 for Batches 1, 3, 4, and 5, respectively.

c = 0.1 M borate buffer solution used in leaching.

d = Reported value was calculated using a greater dilution than the primary analysis.

e = Value reported is an estimated concentration; compound was detected but at a concentration less than the reporting limit.

Raw waste from each of the four batches was combined, homogenized into a single sample, and heated to increasingly higher temperatures within airtight glovebags for one hour intervals. Five temperature ranges, each of one hour duration, were investigated for the study: 70° to 110°C, 110° to 200°C, 200° to 230°C, 230° to 300°C, and 300° to 330°C. PCP was analyzed in the waste before and after the heating process. Air monitoring for PCP and volatile organics, via three specific air sampling media (carbon adsorption tubes, impinger tubes, and cellulose ester particulate filters) was conducted during each temperature interval to identify any particulate or volatile release of these contaminants. A duplicate sample of the homogenized waste was analyzed

at each of the five temperature intervals to monitor reproducibility of the results. A detailed description of the treatment procedure is presented in the "PCP Thermal Study Final Report" (Kiber Associates, Inc., 1992).

Results of PCP analysis of the waste before and after heating are presented in Table 6-13 and show an average percent reduction greater than 99 percent. Air sampling results of the glovebags showed no detectable PCP in the air sampling media during the temperature ranges which most closely represent heat of hydration temperatures (60° to 70°C). Negligible amounts of methylene chloride, chloroform, and tetrachloroethane were detected during the temperature range of 70° to 110°C. PCP was detected along with other volatile compounds in greater concentrations at higher temperature levels. The highest concentration of PCP detected in either of the glovebags was during the fourth interval (230° to 300°C). Although the duplicate samples showed different behavior upon heating, the results do show that nearly all the PCP was lost by heating the contaminated waste to 330°C. However, only an insignificant fraction of the total PCP was recovered in air sampling. The theoretical boiling point of PCP is 310°C with decomposition, suggesting that decomposition could account for most of the loss of PCP.

Dioxins and Furans

Analytical results for polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) are reported in Tables 6-14 and 6-15, respectively. In more than half of the raw waste samples, dioxin concentrations were less than the reporting limits, and percent reductions were not calculable. For the remaining dioxin analyses, percent reductions ranged from -1,011 to 10 percent. Almost one-third of the PCDF analyses did not show concentrations above the reporting limits. The remaining calculated percent reductions for PCDF ranged from -169 to 18 percent. Extremely low initial concentrations for both the PCDDs and the PCDFs in the raw wastes from the SPT site make evaluating the effectiveness of the STC treatment process for these compounds very difficult.

Table 6-13. PCP Concentrations for SPT Site Waste Upon Heating

Concentrations (mg/kg)		Average Percent Reduction
Raw Waste	Heated Raw Waste	
5,100	2.7	99
	11.0	
Glovebag 1 ^a	Glovebag 2 ^a	Temperature Interval (°C)
ND	ND	70 - 110
0.90	2.1	110 - 200
0.11	1.0	200 - 230
ND	26	230 - 300
ND	4.8	300 - 330

ND = Not detected

^a = Results were reported in total µg of PCP recovered. Concentrations presented represent PCP volatilized from 0.249 kg and 0.248 kg of waste heated in glovebags 1 and 2 respectively.

Table 6-14. Analytical Results for Dioxins in STC-Treated Wastes

Constituent	Batch	Concentrations (mg/kg)		Percent Reduction ^{b,c}
		Raw Waste ^a	Treated Waste ^a	
2,3,7,8-TCDD	1	<0.0001	<0.0025	NC
	3	<0.0001	<0.0002	NC
	4	<0.0020	<0.0020	NC
	5	<0.0020	<0.0020	NC
TCDD	1	<0.0001	<0.0025	NC
	3	<0.0001	0.0048	NC
	4	<0.0020	<0.0020	NC
	5	<0.0020	<0.0020	NC
PeCDD	1	<0.0002	0.013	NC
	3	<0.0001	0.057	NC
	4	<0.0020	0.027	NC
	5	0.0022	0.014	-1,011
HxCDD	1	0.099	0.092	-63
	3	<0.0001	0.190	NC
	4	0.110	0.200	-223
	5	0.405	0.210	10
HpCDD	1	1.80	1.85	-81
	3	<0.0002	4.20	NC
	4	4.80	4.30	-59
	5	6.75	5.10	-32
OCDD	1	10.0	18.5	-226
	3	<0.0005	26.0	NC
	4	30.0	51.0	-202
	5	37.5	69.0	-221

NC = Not calculable

a = Results reported as the mean of duplicate samples.

b = Percent Reduction = $\left[1 - (1 + \text{Additives Ratio}) \times \frac{\text{Concentration of Treated Waste}}{\text{Concentration of Raw Waste}} \right] \times 100$.

c = The additives ratio is the weight of additives, including water of hydration, divided by the weight of wastes. Values are 0.761, 0.764, 0.776, and 0.746 for Batches 1, 3, 4, and 5, respectively.

Table 6-15. Analytical Results for Dibenzofurans in STC-Treated Wastes

Constituent	Batch	Concentrations (mg/kg)		Percent Reduction ^{b,c}
		Raw Waste ^a	Treated Waste ^a	
TCDF	1	<0.001	<0.001	NC
	3	<0.0001	0.0005	NC
	4	<0.001	<0.001	NC
	5	0.0013	<0.002	-169
PeCDF	1	0.020	0.009	18
	3	<0.001	0.011	NC
	4	0.033	0.023	-23
	5	0.0435	0.023	8
HxCDF	1	0.20	0.180	-59
	3	<0.0001	0.125	NC
	4	0.55	0.37	-20
	5	0.730	0.41	2
HpCDF	1	0.93	0.805	-52
	3	<0.0001	0.365	NC
	4	2.60	1.70	-16
	5	3.50	2.00	0.2
OCDF	1	0.78	0.775	-75
	3	<0.0002	0.320	NC
	4	2.30	1.60	-24
	5	2.85	1.70	-4

NC = Not calculable

a = Results reported as the mean of duplicate samples.

b = Percent Reduction = $\left[1 - (1 + \text{Additives Ratio}) \times \frac{\text{Concentration of Treated Waste}}{\text{Concentration of Raw Waste}} \right] \times 100$.

c = The additives ratio is the weight of additives, including water of hydration, divided by the weight of wastes. Values are 0.761, 0.764, 0.776, and 0.746 for Batches 1, 3, 4, and 5, respectively.

6.4.2

Inorganics

Although arsenic, chromium, and copper were contaminants of regulatory concern at the SPT site, routine total waste analyses (TWA) were performed for the 23 standard Hazardous Substance List (HSL) metals, plus molybdenum. These analyses typically did not identify additional anomalously high quantities of metals other than elements commonly found in soils, such as iron and aluminum. Tables 6-16 and 6-17 show analytical results for inorganic constituents analyzed for in the sand, water, and reagent mixture additives. Leachate analyses are also included for the sand and reagent mixture additives.

TWA results for arsenic, chromium, and copper for raw and treated wastes were included in the evaluation despite the expectation that concentrations of *total* metals would not be reduced by the STC process except by dilution due to the addition of treatment reagents (Table 6-18). Figures 6-18 through 6-20 graphically show average concentrations of TWA for arsenic, chromium, and copper, respectively, including standard deviation values shown as error bars. Treated waste TWA results show that concentrations of arsenic, chromium, and copper were reduced in absolute terms after treatment; however, after the dilution from added reagents is accounted for, mostly negative percent reductions are obtained.

The skewness of TWA percent reduction data most likely indicates that the treatment reagents affect the oxidation-reduction potential of the metal analytes, resulting in an increased quantity of metals available for extraction and detection. Indeed, the STC treatment reagents are strongly reducing, lowering the aggregate Eh of the raw waste approximately 60 percent after treatment (see Section 6.4.4). Alternatively, the treatment process may degrade or otherwise affect the soil matrix so that the acid digestion/extraction of the analytical method recovers a greater proportion of the theoretical available contamination.

6.4.3

pH

The pH of the raw and treated wastes and the reagent mixture was determined by EPA Method 9045 (Table 6-19). Raw waste samples were slightly acidic to neutral with pH values ranging from 6.3 to 7.1. After treatment the pH was much higher with values of 12.5 and 12.6. The sand and water additives had slight basic characteristics with a pH of 8.6 and 8.0 respectively, and the STC reagent blank mixture was very basic with a pH of 12.5 (Table 6-20).

Table 6-16. Metal Analyses of Water and Sand Additives

Constituent	Water - TWA ^a (mg/L)	Sand - TWA (mg/kg)	Sand - TCLP (mg/L)
Aluminum	<0.2	780	0.30
Arsenic	<0.01	<2	<0.01
Calcium	20	310	11
Chromium	<0.05	<10	<0.05
Copper	<0.03	<6	<0.03
Iron	0.05	1200	<0.1
Magnesium	4.1	<200	<1
Manganese	<0.02	9.3	0.072
Potassium	2.15	<200	1.1
Sodium	17	<200	1,300
Zinc	0.037	<4	0.053

a = Results reported as mean of duplicate samples.

Table 6-17. Metal Analyses of Reagent Mixture (Sand Plus Reagents)

Constituent	TWA (mg/kg)	TCLP (mg/L)	TCLP Distilled Water (mg/L)	TCLP-Cage (mg/L)	CALWET (mg/L)
Aluminum	4,300	<0.2	0.56	<0.2	15
Arsenic	2.5	<0.01	<0.01	<0.07	<0.1
Barium	27	0.21	0.40	0.21	<1
Calcium	61,000	1,900	660	2,000	1,200
Chromium	12	0.07	<0.05	0.053	<0.5
Copper	<6	<0.03	<0.03	<0.03	<0.3
Iron	3,100	<0.05	<0.05	<0.05	24
Magnesium	930	5.5	<1	11	<10
Manganese	27	<0.02	<0.02	<0.02	0.32
Potassium	1,400	19	19	15	51
Selenium	<1	---	<0.005	0.006	<0.05
Sodium	1,100	16	14	23	8,600
Zinc	8.1	<0.02	<0.02	<0.02	0.39

Table 6-18. Total Waste Analyses (TWA) for Inorganic Target Analytes

Constituent: Arsenic			
Batch	Concentrations (mg/kg)		Average Percent Reduction ^{a,b}
	Raw Waste	Treated Waste	
1	380 ^c	290	-17
	450 ^c	340	
	360 ^c	290	
	390 ^c	380	
	300 ^c	280	
	910 ^e	270	
	Average	465 ± 220	
3	260 ^c	160 ^c	-29
	220 ^c	320 ^{c,q}	
	240 ^c	160 ^c	
	260 ^c	150 ^c	
	380 ^{c,q}	190 ^c	
	260	210	
	Average	270 ± 56	
4	1,700 ^c	850 ^c	-4
	1,600 ^c	1,000 ^c	
	1,400 ^c	1,000 ^c	
	1,700 ^c	1,200 ^{c,q}	
	2,000 ^c	1,000 ^c	
	1,800	920 ^c	
	Average	1,700 ± 200	
5	2,200 ^c	1,200 ^c	-24
	2,200 ^c	1,100 ^c	
	2,300 ^c	1,800 ^c	
	1,900 ^c	1,200 ^c	
	1,800 ^c	2,600 ^c	
	2,700	1,400	
	Average	2,200 ± 320	
		1,550 ± 570	

Table 6-18. Total Waste Analyses (TWA) for Inorganic Target Analytes (continued)

Constituent: Chromium			
Batch	Concentrations (mg/kg)		Average Percent Reduction ^{a,b}
	Raw Waste	Treated Waste	
1	340	330	-48
	450	360	
	340	340	
	320	330	
	480	340	
	510	350	
Average	410 ± 83	340 ± 12	
3	420	260 ^c	-36
	280	260 ^c	
	290	210 ^c	
	290	280 ^c	
	480	350 ^c	
	300	230	
Average	340 ± 85	265 ± 48	
4	1,900	860 ^c	4
	1,700	1,000 ^c	
	1,700	1,000 ^c	
	1,700	990 ^c	
	1,700	960 ^c	
	1,800	890 ^c	
Average	1,750 ± 84	950 ± 61	
5	2,100	1,500 ^c	-5
	2,000	1,400 ^c	
	2,200	1,300 ^c	
	1,900	1,100 ^c	
	2,000	1,200 ^c	
	2,500	1,100	
Average	2,100 ± 210	1,300 ± 160	

Table 6-18. Total Waste Analyses (TWA) for Inorganic Target Analytes (continued)

Constituent: Copper			
Batch	Concentrations (mg/kg)		Average Percent Reduction ^{a,b}
	Raw Waste	Treated Waste	
1	340	260	-32
	390	290	
	330	280	
	330	280	
	380	280	
	450	270	
Average	370 ± 47	280 ± 10	
3	360	220	-15
	310	220	
	290	200	
	290	200	
	410	240	
	310	200	
Average	330 ± 48	210 ± 16	
4	1,200	570	4
	1,200	670	
	1,100	650	
	1,200	640	
	1,200	640	
	1,100	620	
Average	1,200 ± 52	630 ± 34	
5	1,200	930 ^c	-8
	1,300	870 ^c	
	1,300	760 ^c	
	1,200	680 ^c	
	1,300	720 ^c	
	1,300	730	
Average	1,300 ± 52	780 ± 97	

Table 6-18. Total Waste Analyses (TWA) for Inorganic Target Analytes (continued)

- a* = Percent Reduction = $\left[1 - (1 + \text{Additives Ratio}) \times \frac{\text{Concentration of Treated Waste}}{\text{Concentration of Raw Waste}} \right] \times 100$.
- b* = The additives ratio is the mass of additives, including water of hydration, divided by the mass of raw wastes. Values are 0.761, 0.764, 0.776, and 0.746 for Batches 1, 3, 4, and 5, respectively.
- c* = Duplicated analysis not within control limits.
- q* = Fails *Q* test (Shoemaker, 1974); value may be disregarded with 90% confidence (although it has been used to calculate the average value).
- s* = Value may be disregarded with 95% confidence (although it has been used to calculate the average value).

Table 6-19. Analytical Results for pH, Eh, Loss on Ignition, and Neutralization Potential for Raw and Treated Wastes

Analysis	Batch	Raw Waste	Treated Waste
pH (pH units)	1	7.2	12.4
		7.2	12.5
		6.9 ^a	12.5
	Average	7.1 ± 0.2	12.5 ± 0.1
	3	6.9	12.6
		7.0	12.6
		6.9	12.6
	Average	6.9 ± 0.1	12.6 ± 0.0
	4	6.2	12.5
		6.3	12.5
		6.3	12.5
	Average	6.3 ± 0.1	12.5 ± 0.0
	5	6.9	12.5
		6.9	12.6
		6.8	12.5
	Average	6.9 ± 0.1	12.5 ± 0.1
Eh (mV)	1	385	166
		387	157
		394	165
	Average	389 ± 5	163 ± 5
	3	393	164
		392	165
		395	165
	Average	393 ± 2	165 ± 1
	4	421	156
		420	167
		422	174
	Average	421 ± 1	166 ± 9
	5	399	173
		400	172
		399	178
	Average	399 ± 1	174 ± 3

Table 6-19. Analytical Results for pH, Eh, Loss on Ignition, and Neutralization Potential for Raw and Treated Wastes (continued)

Analysis	Batch	Raw Waste	Treated Waste
Neutralization Potential (meq/gram)	1	0.11 ^q	3.8
		0.14	3.7
		0.13	3.7
		0.13	NA
		0.14	NA
	Average	0.13 ± 0.01	3.7 ± 0.06
	3	0.13	3.6
		0.15	3.6
		0.11	3.9 ^q
		0.12	NA
	Average	0.13 ± 0.02	3.7 ± 0.17
	4	0.11	3.7 ^q
		0.12	3.5
		0.14	3.5
		NA	3.5
	Average	0.12 ± 0.02	3.6 ± 0.10
	5	0.16	3.7
		0.15	3.7
		0.14	3.6
		NA	3.7
	Average	0.15 ± 0.01	3.7 ± 0.05
Loss on Ignition (%)	1	6.4	18.1
		6.5	27.9
		7.7	28.2
	Average	6.9 ± 0.7	24.7 ± 5.7
	3	6.8	24.4
		6.8	19.6
		5.2 ^q	28.8
	Average	6.3 ± 0.9	24.3 ± 4.6
	4	7.9	24.3
		7.9	26.4
		7.3 ^q	27.6
	Average	7.7 ± 0.3	26.1 ± 1.7
	5	8.4	27.0
		9.1	27.5
		7.5	24.1
	Average	8.3 ± 0.8	26.2 ± 1.8

NA = Not analyzed

q = Fails Q test (Shoemaker, 1974); value may be disregarded with 90% confidence (although it has been used to calculate the average value).

Table 6-20. pH, Eh, Loss on Ignition and Neutralization Potential for Sand, Water, and STC Reagent Mixture

Analysis	Sand	Water	Reagent Mixture
pH (pH units)	8.6	8.0 7.9	12.5
Average	---	7.95	---
Eh (mV)	366	365 371	144
Average	---	368	---
Neutralization Potential (meq/gram)	NA	NA	3.5 3.7 3.9
Average	---	---	3.7 ± 0.2
Loss on Ignition (%)	12.2	NA	17.6
Average	---	---	---

NA = Not analyzed

6.4.4

Eh

The Eh is a measure of the oxidation-reduction potential of the material. Modified EPA Method 9045 was used to determine Eh for both the raw and treated wastes, as well as for the sand, water, and reagent mixture (Tables 6-19 and 6-20). Measurements were made with an Eh electrode and the waste (or sand/reagent blank) and water slurries prepared for the pH test. Oxidation-reduction potential ranged from 389 to 421 mV for the raw waste, with slightly lower values of 366 and 368 mV for the sand and water additives, respectively. The STC reagent blank mixture and treated wastes reveal much lower Eh values of 144 and 163 to 174, respectively. The decrease in the oxidation-reduction potential as a result of treatment indicates a reduction of metal valence/oxidation states and a less oxidizing environment.

6.4.5

Acid Neutralization Capacity

The acid neutralization capacity determines the buffering capacity of the material being tested. This test was not completed because of the acidic nature of the raw waste samples. Instead, neutralization potential measurements were conducted on both the raw and treated waste samples.

6.4.6

Neutralization Potential

The neutralization potential of cementitious reagents and treated wastes, reported in terms of milliequivalents (meq) per gram as shown in Tables 6-19 and 6-20, measures the amount of neutralizers present in the material. Samples of the cured treated waste and the STC reagent mixture weighing 100 grams each were dried to a constant weight at 60°C. The samples were ground to pass a 60-mesh sieve. Two-gram aliquots of each of the samples were placed into flasks and standardized hydrochloric acid (HCl) added. The mixtures were heated to near boiling until no gas evolution was visible. The samples were then brought to volume with deionized water, boiled for 1 minute, and allowed to cool. The sample mixtures were titrated with standardized sodium hydroxide (NaOH), and the neutralization potential calculated from the milliliters of acid consumed by the sample.

The result is expressed in calcium carbonate equivalents and represents tons of calcium carbonate available to neutralize 1,000 tons of material, based on the assumption that an acre plow-layer contains 2 million pounds of soil. Average neutralization potential values ranged from

0.12 to 0.15 meq/gram for the raw waste material. The treated waste and reagent mixture both had higher average values of 3.6 to 3.7 meq/gram.

6.4.7 Loss On Ignition

The American Society for Testing and Materials (ASTM) Method C114 for loss on ignition is a technique for determining the total moisture (including water of hydration) and carbon content of a cementitious solid. Samples were ignited at a temperature of 950°C for 15 minutes. The percent of weight loss was calculated based on the weight of the original sample. Percent loss on ignition values range from 6.3 to 8.3 for the raw waste, with the sand value at 12.2 percent loss. The STC reagent mixture lost 17.6 percent upon ignition, while the treated wastes had values ranging from 24.3 to 26.2 percent loss (see Tables 6-19 and 6-20).

6.4.8 Oil and Grease Analysis

Oil and grease in samples of the raw and treated wastes were extracted by sonication according to a modified EPA SW-846 Method 3550. Five grams of the solid were added to 25 milliliters (mL) of freon and sonicated. The leachates were acidified with HCl to a pH of less than 2 and extracted with multiple aliquots of freon in a separatory funnel. Freon was then added to the extract to bring it to 100 mL in volume. The oil and grease in the extracts were determined by infrared spectroscopy and are shown in Table 6-21. Calculated percent reductions in the treated waste range from 32 to 52 percent. Although the STC treatment process was not entirely effective in reducing the amount of extractable oil and grease in the SPT site waste, the presence of small quantities (< 2 percent) of oil and grease did not appear to adversely affect solidification of the waste as determined by petrographic observations (discussed in Section 6.6).

6.5 PHYSICAL TESTS

Physical tests performed on the raw and/or treated wastes include particle-size analysis, moisture content, bulk density, permeability, unconfined compressive strength, wet/dry weathering, and freeze/thaw weathering. These waste characteristics are summarized in Tables 6-22 through 6-23 and are discussed in the following sections.

Table 6-21. Oil and Grease Analyses of STC-Treated Waste

Constituent: Oil and Grease			
Batch	Concentrations (mg/kg)		Average Percent Reduction ^{a,b}
	Raw Waste	Treated Waste	
1	11,000	4,000	38
	10,000	3,700	
	11,000	3,500	
Average	10,670 ± 580	3,730 ± 250	
3	12,000	3,200	52
	12,000	3,000	
	11,000	3,400	
Average	11,670 ± 560	3,200 ± 200	
4	18,000	7,000	32
	20,000	8,000	
	19,000	6,900	
Average	19,000 ± 1,000	7,300 ± 600	
5	19,000	7,300	34
	20,000	7,800	
	20,000	7,100	
Average	19,670 ± 580	7,400 ± 360	

$a = \text{Percent Reduction} = \left[1 - (1 + \text{Additives Ratio}) \times \frac{\text{Concentration of Treated Waste}}{\text{Concentration of Raw Waste}} \right] \times 100.$

$b =$ The additives ratio is the weight of additives, including water of hydration, divided by the weight of wastes. Values are 0.761, 0.764, 0.776, and 0.746 for Batches 1, 3, 4, and 5, respectively.

Table 6-22. Physical Characteristics of the STC Raw Waste

Analysis	Batch				
	1	2	3	4	5
Mean Particle Size (mm)	0.065	0.085 ^b	0.060	0.060	0.075
	0.070	0.085 ^{b,q}	0.065	0.075	0.070 ^q
	0.060	0.080 ^b	0.065	0.085	0.075
	0.060	NA	0.060	0.075	NA
Average	0.064 ± 0.005	0.083 ± 0.003	0.063 ± 0.003	0.074 ± 0.40	0.073 ± 0.003
Moisture Content (%)	4.3	3.4	4.9	3.0	5.5 ^q
	5.9	3.2	4.7 ^q	2.8	3.1
	4.4	3.6	4.9	3.4	2.6
	4.9	NA	NA	NA	3.1
	NA	NA	NA	NA	3.1
Average	4.9 ± 0.7	3.4 ± 0.2	4.8 ± 0.1	3.1 ± 0.3	3.5 ± 1.1
Bulk Density (g/cm ³)	1.57	1.73	1.57	1.57	1.57
	1.34	1.72	1.34	1.34	1.34
	1.36	1.53 ^q	1.36	1.36	1.36
	NA	NA	1.73	1.73	1.73
	NA	NA	1.72	1.72	1.72
	NA	NA	1.53	1.53	1.53
Average	1.42 ± 0.13	1.66 ± 0.11	1.54 ± 0.17	1.54 ± 0.17	1.54 ± 0.17

NA = Not analyzed

a = Calculated from weight loss at 105°C; moisture content = $\frac{(\text{wet weight} - \text{dry weight})}{\text{dry weight}} \times 100$.

b = Numerous Batch 2 soil aggregates (PCP-encrusted "hardpan") of up to 10 mm in diameter remained through the waste homogenization and treatment processes. Batch 2 treated waste was therefore excluded from analytical evaluation.

q = Fails *Q* test (Shoemaker, 1974); value may be disregarded with 90% confidence (although it has been used to calculate the average value).

Table 6-23. Physical Characteristics of STC-Treated Wastes and Reagent Mixture

Analysis	Batch				RM
	1	3	4	5	
Moisture Content (%)	2.4	2.2	16.1	6.2	1.5
	3.0	1.7	16.4	4.8	3.7
	2.3	1.9	2.3	11.6	7.1
	NA	NA	3.8	12.4	NA
Average	2.6 ± 0.38	1.9 ± 0.25	9.7 ± 7.65	8.8 ± 3.81	4.1 ± 2.82
Bulk Density (g/cm ³)	1.57	1.57	1.59	1.60	1.94 ^a
	1.60	1.53	1.57	1.66	1.91
	1.55	1.56	1.58	1.59	1.91
Average	1.57 ± 0.03	1.55 ± 0.02	1.58 ± 0.01	1.62 ± 0.04	1.92 ± 0.02
Permeability (cm/sec x 10 ⁻⁷)	1.3	2.1	0.48	2.9 ^a	1.7
	1.6	0.41 ^a	1.3	1.1	1.6
	2.1	2.1	0.96	1.1	1.2
Average (x 10 ⁻⁷)	1.7 ± 0.40	1.5 ± 0.98	0.9 ± 0.41	0.8 ± 0.47	1.5 ± 0.27
Unconfined Compressive Strength (psi)	190	290	310	400	710
	300	250	240	420	860
	210	320 ^s	230	370	420 ^a
	260	260	190	350	480
	250	270	300	310	710
	720 ^a	270	280	340	680
	320	280	170	430	790
	240	280	380	260	780
	220	280	230	250	710
Average	300 ± 160	280 ± 20	260 ± 65	350 ± 65	680 ± 140

RM = Solidified reagent mixture

NA = Not analyzed

a = Calculated from weight loss at 60°C; moisture content = $\frac{(\text{wet weight} - \text{dry weight})}{\text{dry weight}} \times 100$.

q = Fails Q test (Shoemaker, 1974); value may be disregarded with 90% confidence (although it has been used to calculate the average value).

s = Value may be disregarded with 95% confidence (although it has been used to calculate the average value).

6.5.1 Particle-Size Analysis

The particle-size distribution of the raw waste was determined by ASTM Method D422-63, a combined sieve and hydrometer analysis. The sieve analyses were performed on that fraction of each sample larger than 0.074 mm (retained on the No. 200 sieve). This analysis consisted of passing a sample through a set of sieves and weighing the portion of material retained on each sieve. Hydrometer analyses were performed on the finer fractions (less than 0.074 mm). The hydrometer method is based on Stoke's Law and involved preparing a dilute suspension of fine particles in water, measuring the specific gravity of the suspension at specified time intervals, and correlating settling velocity, particle diameter, and time to determine the particle-size distribution. Particle size was not determined for the treated wastes because they were monolithic solids.

The mean particle size for the raw waste ranged from approximately 0.06 to 0.08 mm. This particle-size range is representative of very-fine sand material. Uniformity coefficients of 10 to 20 determined from the particle-size distribution curves indicate that the contaminated soil wastes were well-graded, consisting of several different particle sizes. The sand used in the reagent blank mix had a mean particle size of 0.65 mm.

6.5.2 Moisture Content

Moisture content is defined as the ratio of the weight of water retained by a solid to the dry weight of solids, expressed as a percent. The moisture content of the raw waste and untreated reagent mixture was determined by ASTM Method D2216-80. Moisture was determined on a dry-weight basis by measuring the mass of water removed when the sample was dried to a constant mass at $105^{\circ} \pm 5^{\circ}\text{C}$. The moisture content of the treated waste and reagent mixture was determined by TMSWC-4 (Test Methods for Solidified Waste Characterization; Stegeman and Côté, 1991). This method required that the sample be ground and pass through an ASTM No. 10 sieve. The sample weight was measured before and after it was dried in an oven maintained at $60^{\circ} \pm 3^{\circ}\text{C}$. The dry weight must be constant (weight change of less than 0.03 gram in 4 hours).

The moisture content for the raw waste ranged from 3.1 to 4.9 percent. Moisture content of the treated waste was more variable, ranging from 1.9 to 9.7 percent, with up to 7.7 percent standard deviation. The solidified reagent blank mixture had an average moisture content of 4.1 percent. Batches 1 and 3 showed a decrease in moisture content, whereas Batches 4 and 5

increased in moisture content following treatment. (There was considerable sample-to-sample variability for treated Batches 4 and 5 and for the solidified reagent blank mixture.)

6.5.3 Bulk Density

Bulk densities of the raw and treated wastes are important in the materials balance analysis to determine the expansion in volume of the material that may be attributed to the treatment process. Bulk density of raw waste samples was determined using the American Society of Agronomy (ASA) 13-2 Core Method. With this method, a cylindrical metal sampler was driven into the soil to the desired depth and removed to preserve a known volume of sample as it existed in-situ. Bulk density was calculated based on a soil sample of known volume and its mass. Bulk densities of the treated waste were determined using TMSWC-2, which was performed after the sample had sufficiently cured for 28 days. The bulk density was determined by weighing a cylinder of the treated waste and measuring the dimensions of the core, then dividing the volume into the mass.

Batch 1 represents the soil horizon at depths of 12-24 inches, directly below the surface hardpan, consisting predominantly of sandy material with an average bulk density of 1.42 g/cm³. Batch 2 represents the surface hardpan material (0-12 inches) with a higher average bulk density of 1.66 g/cm³. (Because of the mixing problems with Batch 2, treated samples were not further analyzed.) Bulk densities for raw wastes from Batches 3, 4, and 5 represent a calculated average bulk density (1.54 g/cm³) because these batches were composites of soils from the two differing soil horizons. Bulk densities of the treated, mixed batches (3, 4, and 5) were slightly higher (1.55 to 1.62 g/cm³) than the raw waste average bulk density for those batches. Batch 1 showed the greatest increase in bulk density upon treatment (1.57 g/cm³). The solidified STC reagent blank mixture had a bulk density of 1.92 g/cm³.

6.5.4 Permeability

Falling-head permeability was determined for the treated wastes using TMSWC-13. This method required solidified core samples of the treated waste and the solidified reagent mixture measuring 3 inches in diameter and 3 inches high. Permeability was determined with a triaxial cell by measuring changes of water volume over time under controlled temperature and pressure.

Average permeabilities of the treated wastes ranged from 0.8×10^{-7} cm/sec to 1.7×10^{-7} cm/sec. The solidified reagent mixture showed a permeability of 1.5×10^{-7} cm/sec. These values are of the same order of magnitude as the permeability requirements for hazardous waste landfill soil barrier liners of 10^{-7} cm/sec (40 CFR Part 264 Subpart N).

6.5.5 Unconfined Compressive Strength (UCS)

UCS is defined as the load per unit area, pounds per square inch (psi), at which an unconfined cylindrical sample fails a compression test. UCS determinations were performed on molded samples of solidified waste according to ASTM Method D1633-84, which uses strain-controlled application of an axial load to measure the UCS.

Average values for the treated waste samples after 28 days of curing ranged from 259 psi to 349 psi, with the solidified reagent blank mixture having the greatest measured strength of 682 psi. The results of this test indicate that the UCS of the products of the STC solidification/stabilization process meet the EPA guidance of 50 psi for placement in a landfill (U.S. EPA, 1986a). These values are, however, significantly below the American Concrete Institute (ACI) minimum recommended unconfined compressive strength of 3,000 psi for the construction of sidewalks (ACI, 1992).

6.5.6 Wet/Dry Weathering Test

Wet/dry weathering tests were performed on molded samples of the treated wastes and the solidified reagent blank mixture according to TMSWC-12. This test was performed using two specimens of treated waste 4.5 cm in diameter and 7.4 cm high. One of the specimens was the test sample; the other was the control. Each sample was removed from its mold, weighed, and placed in a beaker. The control was placed in a humidity chamber maintained at $22^{\circ} \pm 3^{\circ}\text{C}$, while the test specimen was dried in a vacuum oven at $60^{\circ} \pm 3^{\circ}\text{C}$ for 24 hours. The test specimen was cooled to room temperature in a desiccator. Upon cooling, 230 mL of distilled water at room temperature were added to each of the sample beakers. Both samples were placed in the humidity chamber for 24 hours, then sprayed with distilled water to remove loosely attached particles from the specimens. The specimens were transferred to two newly tared beakers. The original beakers were placed into an oven at $60^{\circ} \pm 3^{\circ}\text{C}$ to evaporate the water and dry them to a constant weight. This cycle was repeated 11 more times, with the weight loss recorded each time. The corrected relative weight loss of the test specimen was obtained by subtracting the relative weight loss of the

control from the relative weight loss of the sample. Thus, the results are expressed as the cumulative corrected relative weight loss percentage incurred through 12 wet/dry cycles, normalized to a control that was not subjected to the wet/dry cycles. These results are shown in Table 6-24.

Table 6-24. Wet/Dry Weathering Data of STC-Treated Wastes

Batch	Cumulative Corrected Relative Weight Loss (%)											
	Cycle 1	Cycle 2	Cycle 3	Cycle 4	Cycle 5	Cycle 6	Cycle 7	Cycle 8	Cycle 9	Cycle 10	Cycle 11	Cycle 12
1	0.03	0.04	0.02	0.01	0.01	0.00	-0.01	-0.02	-0.01	-0.03	-0.04	-0.04
3	0.00	-0.01	-0.04	-0.05	-0.06	-0.08	-0.08	-0.10	-0.14	-0.16	-0.18	-0.21
4	0.02	0.01	-0.01	-0.01	-0.05	-0.06	-0.09	-0.13	-0.14	-0.17	-0.19	-0.20
5	0.03	0.02	0.02	0.01	-0.02	-0.03	-0.07	-0.08	-0.11	-0.13	-0.16	-0.17
RM	0.02	0.02	0.04	0.04	0.02	0.02	0.00	0.001	-0.01	-0.01	-0.02	-0.02

RM = Solidified reagent mixture

The results of the test for all of the batches and the solidified reagent blank mixture show that less than 1 percent of the core weight was lost from the 12 wet/dry cycles. Negative values show that the weight loss of the control specimens was greater than the weight loss of the test specimens. These results suggest that the treated material and the reagent blank mixture withstood the stresses of the wet/dry weathering test with little weight loss, indicating durability in an exposed environment.

6.5.7 Freeze/Thaw Weathering Test

Freeze/thaw weathering tests were performed on molded samples of the treated wastes and the solidified reagent blank mixture according to method TMSWC-11. The test is similar to the wet/dry weathering test except that the test specimens are placed in a freezer at $-20^{\circ} \pm 3^{\circ}\text{C}$ for 24 hours. The test was performed using two specimens of treated waste 4.5 cm in diameter and 7.4 cm high. One of the specimens was the test sample; the other was the control. Each sample was removed from its mold, weighed, and placed in a beaker. The control was placed in a humidity chamber maintained at $22^{\circ} \pm 3^{\circ}\text{C}$, while the test specimen was placed in a freezer at $-20^{\circ} \pm 3^{\circ}\text{C}$ for 24 hours. Both beakers were removed from their respective chambers. 230 mL of distilled water at $4^{\circ} \pm 3^{\circ}\text{C}$ were added to the beaker containing the frozen specimen, and 230 mL of distilled

water at room temperature were added to the beaker containing the control specimen. Both samples were placed in the humidity chamber for 24 hours, then sprayed with distilled water to remove loosely attached particles from the specimens. The specimens were transferred to two newly tared beakers. The original beakers were placed into an oven at $60^{\circ} \pm 3^{\circ}\text{C}$ to evaporate the water and dry them to constant weight. This cycle was repeated 11 more times, with the weight loss recorded each time. The corrected relative weight loss of the test specimen was obtained by subtracting the relative weight loss of the control from the relative weight loss of the sample. Results of this test shown in Table 6-25 were expressed as the cumulative corrected relative weight loss percentage incurred through 12 freeze/thaw cycles, normalized to a control that was not subjected to the freeze/thaw cycles. Results for all wastes and the solidified reagent blank mixture indicate that 1 percent or less of the molded cylinder weights were lost over the 12 freeze/ thaw cycles. The results suggest that the treated wastes and the solidified reagent blank mixture successfully withstood the stresses of freeze/thaw weathering and will be durable in an exposed environment.

Table 6-25. Freeze/Thaw Weathering Data of STC-Treated Wastes

Batch	Cumulative Corrected Relative Weight Loss (%)											
	Cycle 1	Cycle 2	Cycle 3	Cycle 4	Cycle 5	Cycle 6	Cycle 7	Cycle 8	Cycle 9	Cycle 10	Cycle 11	Cycle 12
1	0.00	0.01	0.01	0.03	0.02	0.04	0.05	0.04	0.04	0.04	0.06	0.09
3	-0.02	-0.05	-0.04	-0.05	-0.05	-0.04	-0.04	-0.01	-0.03	-0.02	-0.01	0.02
4	-0.02	-0.03	-0.04	-0.03	-0.04	-0.05	-0.07	-0.10	-0.09	-0.05	-0.04	-0.01
5	0.00	-0.02	-0.03	-0.03	-0.05	-0.05	-0.07	-0.08	-0.08	-0.07	-0.08	-0.09
RM	-0.03	-0.05	-0.04	-0.04	-0.01	0.03	0.04	0.07	0.09	0.15	0.18	0.22

RM = Solidified reagent mixture

6.6 PETROGRAPHIC ANALYSES

Solidified samples from the STC SITE demonstration were examined using optical microscopy, scanning electron microscopy (SEM), X-ray diffractometry (XRD), and Fourier transform infrared spectroscopy (FTIR) techniques. The samples consisted of small cylindrical cores that had been taken from waste from Batches 1, 3, 4, and 5. In addition, a solidified blank

sample made from clean sand mixed with the STC proprietary reagent mixture was also analyzed. The cores were cut into appropriate sample sizes for the microscopic examination, and an adjacent piece was cut for the SEM examination. A portion of the remainder of the cores was broken off and about 50 grams reduced to small pieces. A fraction passing a 3/8-inch sieve but retained on a No. 8 sieve was submitted for FTIR examination. The rest of the sample was ground in a ring and puck mill until the whole sample was finer than a No. 150 sieve. These fine powder samples were submitted for additional FTIR and XRD analyses. Further preparation of samples is described in the following subsections.

Five samples of the raw waste were also submitted for optical microscopy, FTIR, and XRD analyses. Samples from Batch 1 were obtained from the deeper soil horizon (approximately 1 to 2 feet) while those from Batch 2 were from the shallower hardpan horizon (down to 1 foot). The remaining samples consisting of Batches 3, 4, and 5 were composite blends of the first two soil horizons. Batch 1 was the only batch analyzed that was not sieved as part of the pretreatment process. These samples are described in greater detail in the following subsection.

6.6.1 Optical Microscopy

Qualitative petrographic examination of twelve cylindrical cores was performed in accordance with ASTM Method C856-83, "Standard Practice for Petrographic Examination of Hardened Concrete." The cores were cut longitudinally and examined using a stereomicroscope at magnifications up to 45 X. Freshly broken surfaces were also studied using the stereomicroscope. Preparation of petrographic thin sections consisted of cutting a rectangular block 1 inch wide and 0.5 inch thick from the top portion of each core to a depth of approximately 1.8 inches. These rectangular blocks of solidified material were placed on glass microscope slides with epoxy resin, and reduced to a thickness of approximately 20 micrometers (0.00079 inch). A polarized-light microscope at magnifications up to 400 X was used to determine aggregate and binder mineralogy and microstructure. In addition, five raw waste samples were also examined using a stereomicroscope.

The unconsolidated, raw waste samples consisted mainly of very-fine grained (<0.2 mm) quartz, feldspars (potassium feldspars and plagioclase), hornblende, clay, and mica. Granitic lithic fragments up to 10 mm diameter were also observed. The soils were grayish-brown to dark brown and had a strong odor of oil. Wood fragments and other organic debris were present in small

amounts in all of the samples. Many of the larger particles had shiny coatings, presumably oil. Clumps of clay-size material appeared to be held together by an oily substance.

Results of the thin-section analyses of the solidified waste cores are detailed in Table 6-26. In addition, Figures 6-21 through 6-27 show examples of photographs of the cores and photomicrographs of the thin sections. Two cores for each of Batches 1, 3, 4, 5, and the STC reagent mixture were qualitatively analyzed. Evaluation criteria for this examination consisted of consolidation, hardness, microcracking, estimated air content, binder distribution, bonding, phases present in the binder material, and soil constituents.

In general, the cores of the solidified waste and reagent material were well consolidated (Figures 6-21 and 6-22). The outer portion of one of the QC samples was very poorly consolidated (Figure 6-23). This sample was somewhat soft, but cohesive, and resistant to fracturing from moderate impacts. Some of the cores displayed faint layering, and hardness ranged from soft to moderately hard with only minor microcracking observed. The estimated air content was three to five percent for Batches 3, 4, and 5, and five to seven percent for Batch 1 and the solidified reagent mixture. The air-void system was nonuniform and the voids contained no secondary deposits (Figure 6-24). The binder was moderately soft, generally evenly distributed, and the binder-aggregate bond was moderately tight. The binder microstructure was difficult to interpret because of the presence of large amounts of black opaque material that acted as a pigment (Figure 6-25). In areas where this material was not abundant, the binder contained patches of calcium hydroxide (portlandite), small amounts of green-brown stained glassy phase, and traces of residue portland cement clinker. Carbonation of the binder material around small air voids was observed and is the result of a reaction between calcium hydroxide and air. The soil-binder system appears to be well mixed based on distribution and size of the soil clumps (Figure 6-26). The average size of soil fragments is 1.5 mm. Clumps with diameters up to 1 cm occur and are typically surrounded by a black, tar-like rim (Figure 6-27).

Table 6-26. Petrographic Analysis of STC-Treated Wastes

Characteristics	Treated Wastes (Batches)					
	RM	1	1 - QC	3	4	5
Binder Distribution	Uniform	Nonuniform	Nonuniform	Uniform	Uniform	Uniform
Consolidation	Well consolidated	Under consolidated	Well consolidated	Well consolidated	Well consolidated	Well consolidated
Hardness	Moderately hard	Moderately soft	Soft	Moderately soft	Moderately soft	Moderately soft
Bond	Moderately tight	Moderately tight	Moderately tight	Moderately tight	Moderately tight	Moderately tight
Air Content (estimated)	5% to 7%	No estimate made	5% to 7%	3% to 5%	3% to 5%	3% to 5%
Microcracking	None	Minor	None	Minor	None	None
Phases present in binder	Calcium hydroxide Black pigment Isotropic glass Portland cement	Calcium hydroxide Black pigment Isotropic glass Portland cement	Calcium hydroxide Black pigment Isotropic glass Portland cement	Calcium hydroxide Black pigment Isotropic glass Portland cement	Calcium hydroxide Black pigment Isotropic glass Portland cement	Calcium hydroxide Black pigment Isotropic glass Portland cement
Soil constituents	Quartz, microcline; No oily clumps	Quartz, plagioclase feldspar, potassium feldspar, horn- blende, clay, biotite, wood fragments, misc. organic debris, paper	Quartz, plagioclase feldspar, potassium feldspar, horn- blende, clay, biotite, wood fragments, misc. organic debris, paper	Quartz, plagioclase feldspar, potassium feldspar, horn- blende, clay, biotite, wood fragments, misc. organic debris, paper	Quartz, plagioclase feldspar, potassium feldspar, horn- blende, clay, biotite, wood fragments, misc. organic debris, paper	Quartz, plagioclase feldspar, potassium feldspar, horn- blende, clay, biotite, wood fragments, misc. organic debris, paper

RM = Solidified reagent mixture

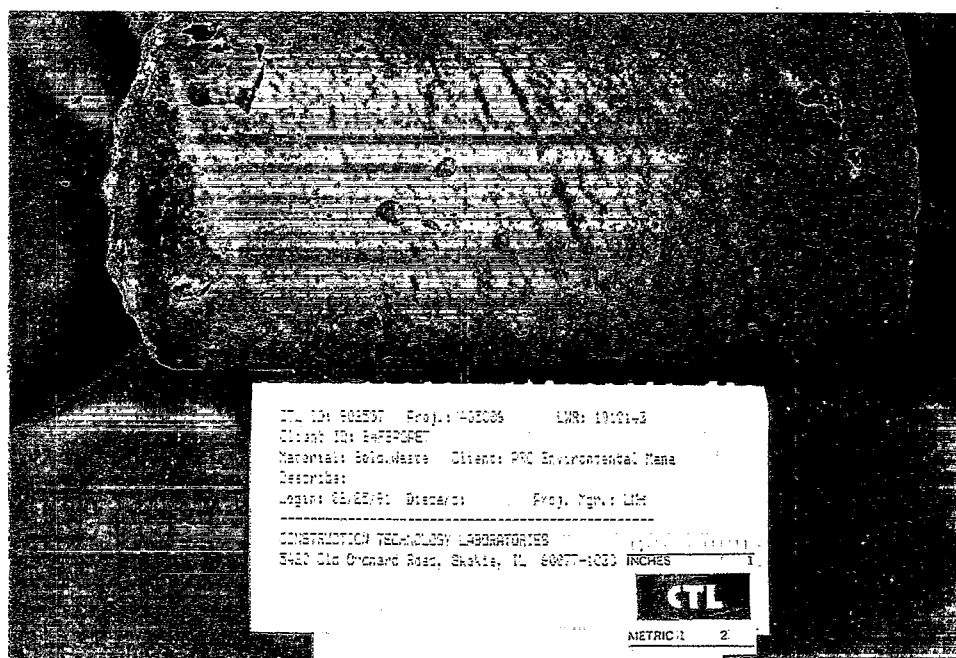


Figure 6-21. STC solidified waste core (Batch 4).

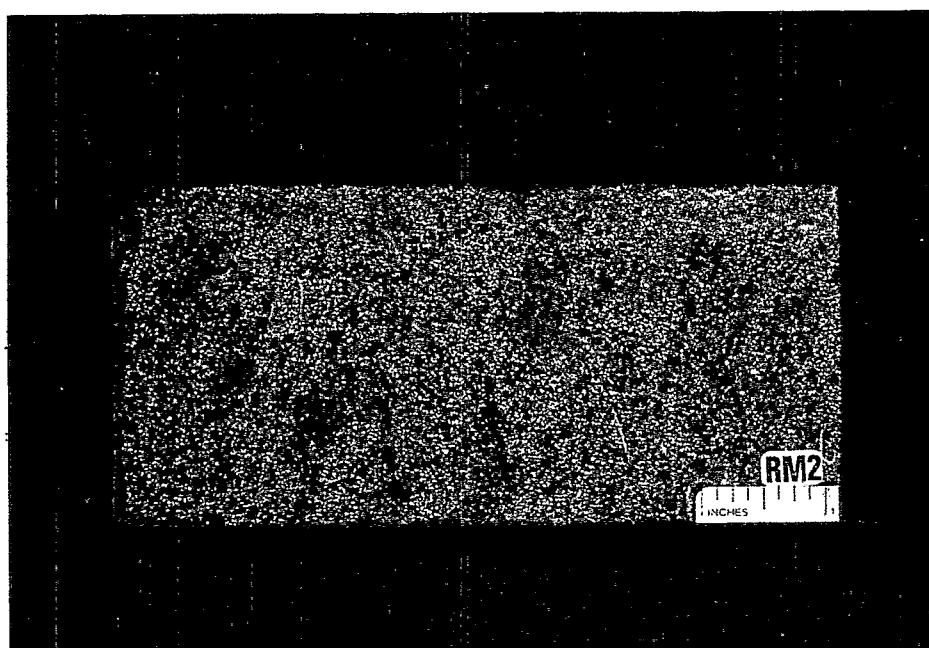


Figure 6-22. Longitudinal cut of solidified reagent mixture and sand.

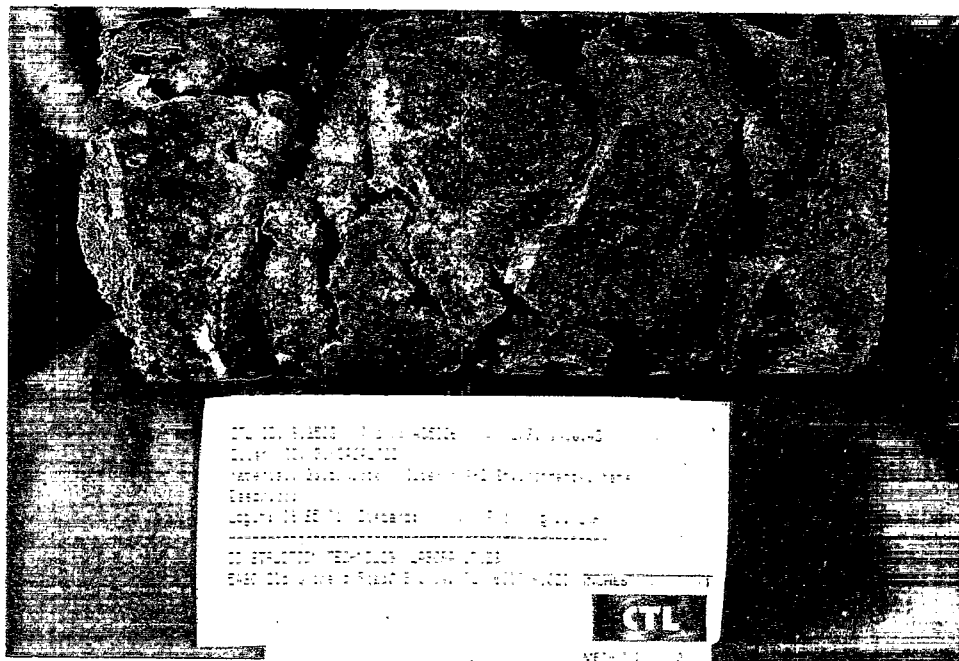


Figure 6-23. Poorly consolidated solidified waste core (Batch 1-QC).

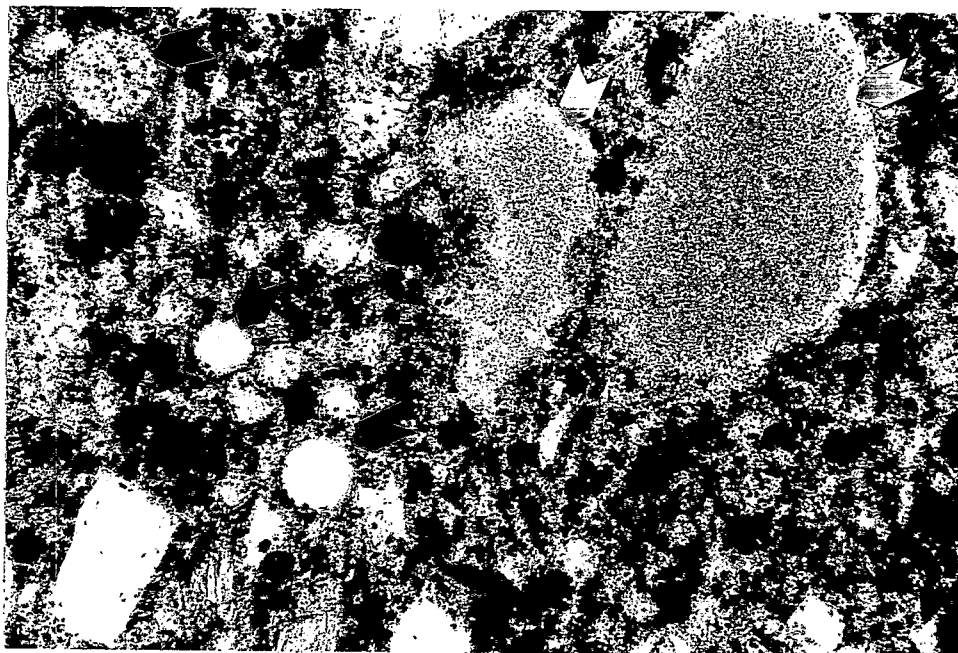


Figure 6-24. Photomicrograph showing less uniform binder distribution. Green arrows show clay lumps. Red arrows show spherical air voids. Binder contains wood fragments (brown). 200x magnification (Batch 1).

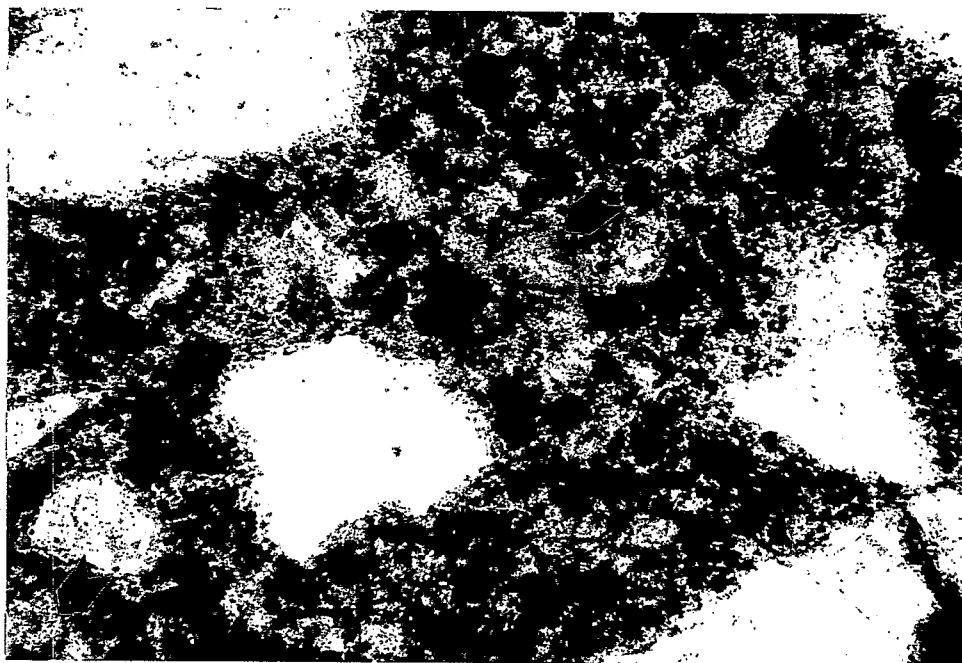


Figure 6-25. Photomicrograph showing soil particles (white) surrounded by densely pigmented binder. Binder contains wood fragment (arrows). 200x magnification (Batch 5).

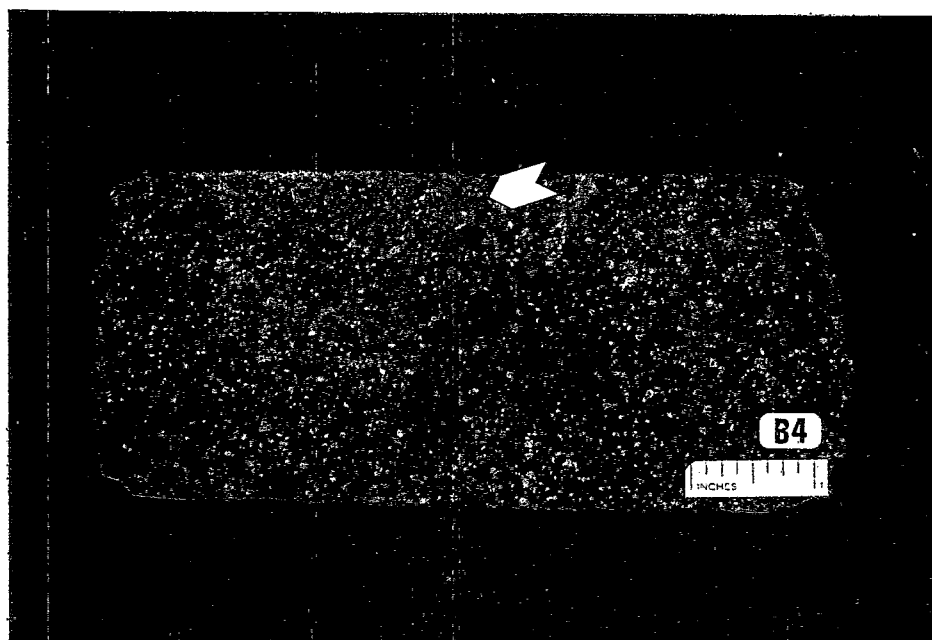


Figure 6-26. Longitudinal cut of solidified waste core (Batch 4). Note abundance of soil particles. Arrow shows coated clumps of soil.

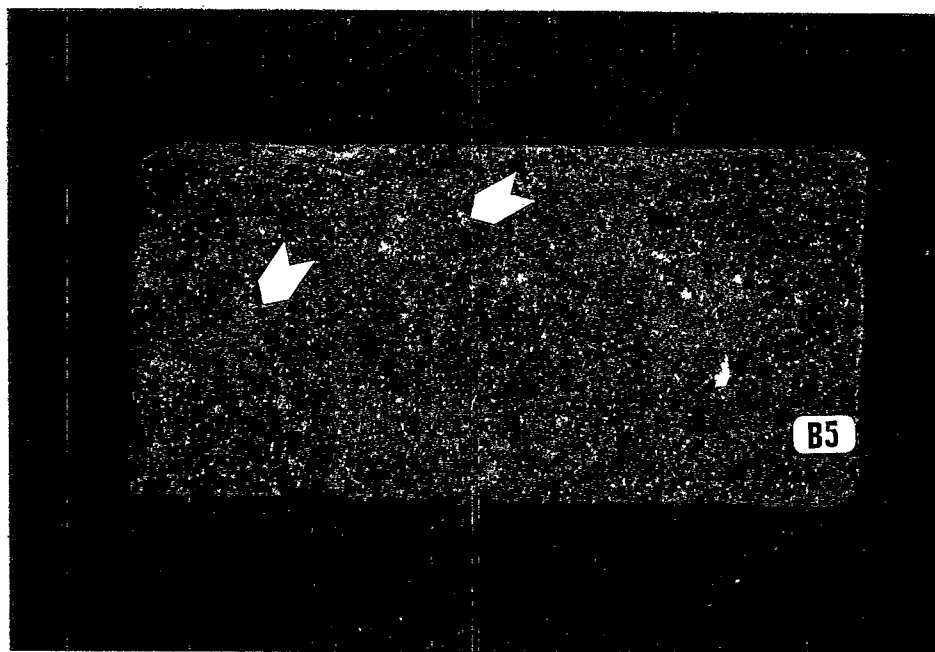


Figure 6-27. Longitudinal cut of solidified waste core (Batch 5). Arrows show coated clumps of soil particles.

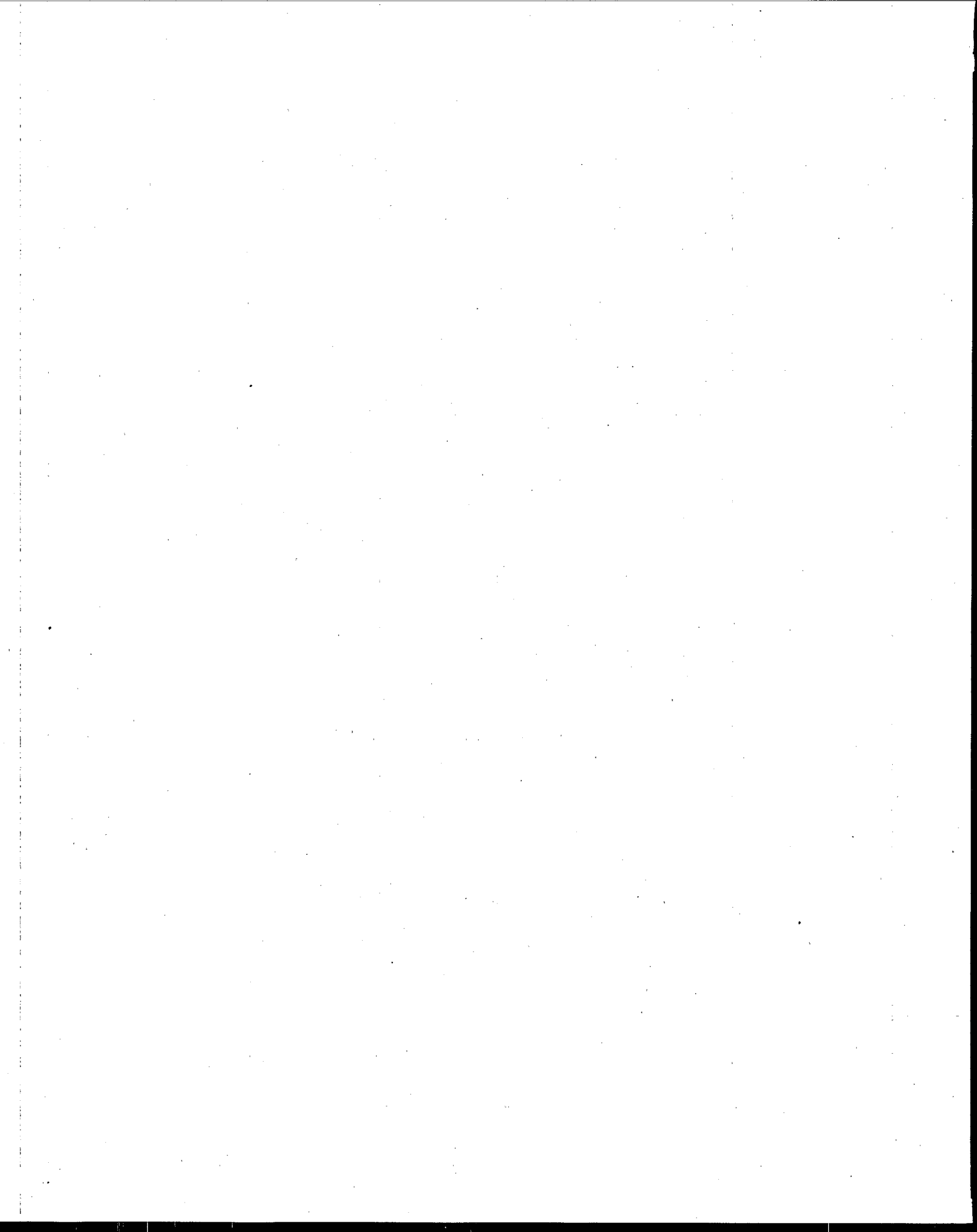
Two untreated and six solidified waste samples were examined using XRD techniques. The contaminated soil samples represented both the deep (Batch 1) and shallow (Batch 2) soil horizons. Four of the solidified samples were treated waste (Batches 1, 3, 4, and 5), one was the solidified waste QC sample, and one was the solidified STC reagent mixture. All samples were ground and passed through an ASTM No. 15 mesh sieve, then ground to a fine powder. The powders were then packed from the backside into an aluminum XRD sample holder. A Phillips X-ray diffractometer equipped with a theta-compensating slit, a graphite monochromator, and a scintillation detector was used. Copper K-alpha radiation was generated at 40 kV and 35 mA. The signal was processed through a ratemeter set at a scale of 1,000 with a time constant of 2 seconds and a background suppression setting of 1.2.

The patterns for each of the cores were examined, and phases that could be identified were roughly classified as major, minor, or trace to indicate their relative abundance. Results of this qualitative XRD analysis are summarized in Table 6-27. Batches 1 and 2 of the raw waste consisted of similar mineralogic phases and are listed together. Similarly Batch 1 and 1-QC are listed together.

Components of the untreated waste were also present in the solidified waste samples, as expected. In addition to quartz, the raw waste samples contained a considerable amount of clay -- weathered from the two mineral groups, plagioclase feldspar and potassium alkali feldspar -- and minor amounts of hornblende and trace mica. The micas consist of biotite and/or illite; peak weakness prevented more positive identification.

The solidified STC reagent mixture contained predominantly quartz with minor amounts of potassium feldspars. Minor portlandite (calcium hydroxide) and traces of gypsum and ettringite were also detected.

The treated waste samples were all characterized as containing major amounts of quartz and oligoclase with slightly lesser amounts of the potassium feldspars microcline and orthoclase. Minor amounts of portlandite were detected in all solidified samples as well as traces of ettringite and either illite and/or biotite. Some samples had traces of hornblende. An elevated background from roughly 25 to 30 degrees 2-theta indicated that the binder material was primarily amorphous. One or two very small additional peaks were observed in the solidified materials, but



they were not sufficiently distinct for identification. These peaks may be the result of aluminosilicate hydrates formed by the binder. No peaks were observed for unhydrated portlandite cement materials, indicating the cement content was fairly low. These analyses indicate that the STC process used predominantly silica and potassium-aluminum silicates in addition to calcium hydroxide and sulfates to form the binding agent.

6.6.3 Scanning Electron Microscopy (SEM)

Only solidified samples were submitted for SEM analysis. These included one from each of Batches 1, 3, 4, and 5; a QC sample from Batch 1; and a sample from the solidified STC reagent blank mixture. Sections of cores that had been cut to approximately 2.5 cm by 4.5 cm by 1 cm were used. An attempt was made to impregnate these sections with epoxy prior to grinding and polishing, but not all samples were receptive to the epoxy. Polished samples were mounted on aluminum stubs and coated with carbon. After elemental analysis was complete, samples were coated with gold to provide better imaging at high magnifications. Secondary electron (SE) emissions provided the normal mode of imaging in the SEM. Backscattered emissions (BSE) allowed imaging with elemental contrast -- light elements (e.g., silicon and calcium) result in dark areas and heavy elements (e.g., copper and iron) show up as light areas. The SEM was also equipped with energy dispersive X-ray (EDX) fluorescence analysis, providing an elemental analysis. Once EDX identified the elements present, elemental mapping capabilities were used to determine the location of elements by dot mapped photographs. SEM photomicrographs include the following information as read from left to right: beam energy in keV; magnification factor divided by 1,000; a scale bar and the number of microns the bar represents; and an identification number.

The SEM analyses indicated relatively good binder-to-aggregate bonding except in rare cases where oily particle coatings prevented good bonding. The oil has a dual negative effect of preventing wet cement paste from penetrating and binding to the aggregate and also may partially inhibit hydration of the cement. An example of paste absence resulting from an oily coated aggregate is shown in Figure 6-28. The oily coated aggregate with a very poor bond to the paste is in the lower right, while other aggregates in the same view demonstrate good bonding. Lack of paste surrounding the aggregates may also be the result of paste removal during sample preparation. In general, however, the quality of the surrounding binder sufficiently microencapsulated such particles.

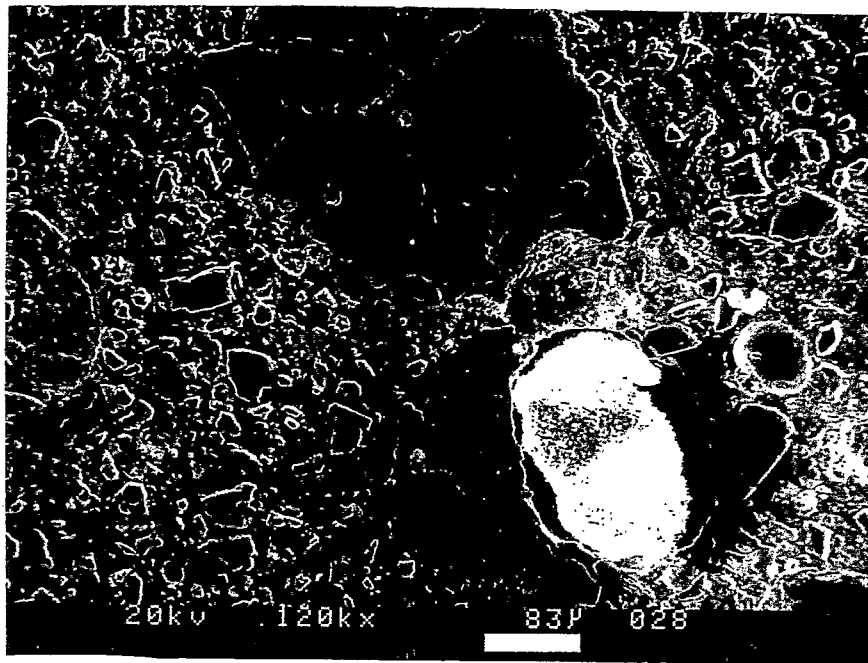


Figure 6-28. SE image of poor and well bonded aggregate (Batch 1).

The following elements were commonly detected using the EDX probe: calcium, silicon, iron, aluminum, potassium, and chlorine, with minor amounts of sulfur, arsenic, chromium, titanium, and copper. An EDX probe of the bright area in Figure 6-29 detected the presence of chromium, copper, and arsenic. An elemental spectrum of the entire area of Figure 6-29 is shown in Figure 6-30. Elemental maps of the heavy metal contamination suggest good containment of the metals. Figure 6-31 shows an embedded particle in which a substantial amount of titanium was detected. The elemental dot map for titanium (Figure 6-32) reveals that although small amounts of this element are detected in the surrounding area, titanium from the particle is primarily contained in the particle itself. Another example shows a BSE image of the bright area in Figure 6-33 where copper, chromium, and arsenic were detected. Maps depicting the location of these elements are provided in Figures 6-34, 6-35, and 6-36, respectively. There appear to be boundaries on the maps that correlate well with the boundary of the bright image. Mixing of binder and waste followed by consolidation did not appear to cause migration of metals from contaminated particles into the surrounding material.

Air voids are generally rounded in shape, vary in size, and appear relatively open in all of the treated samples. A typical area of distribution for air voids is shown in Figure 6-37. Voids, paste, aggregate, and areas of contamination are evident by their shades of gray as related to their elemental content. The dark areas in this image show rounded voids indicating good consolidation; irregular shaped voids would indicate poor consolidation. Figure 6-38 shows an open air void in paste.

SEM analyses are also useful for observing paste morphology of the solidified samples. An area showing typical aggregate/paste bond is Figure 6-39. There is some removal and/or absence of paste around aggregate edges and also signs of aggregate pull-out indicating less-than-optimum bonding. A higher magnification of such an aggregate/paste bond is shown in Figure 6-40.

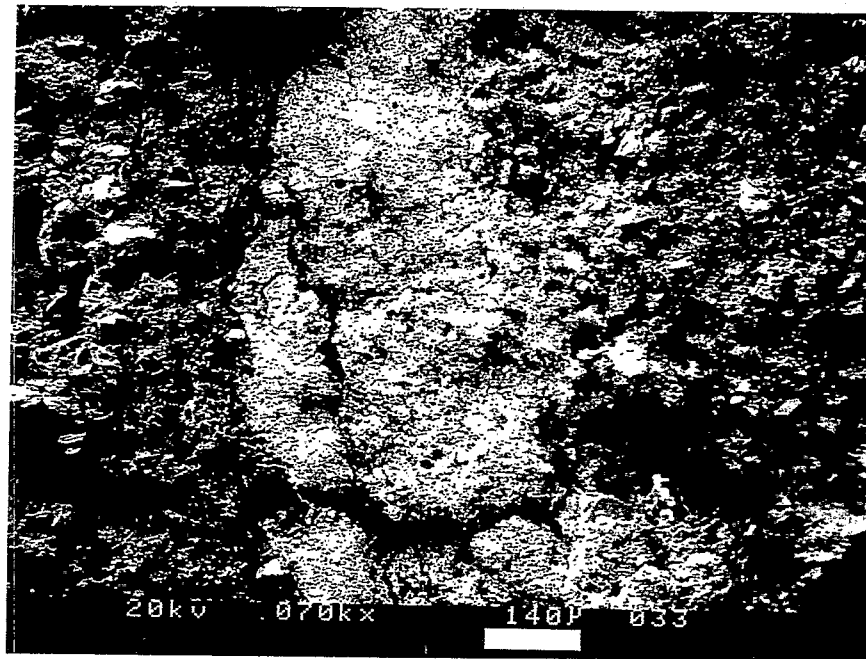


Figure 6-29. BSE image. Cu, Cr, and As were detected in the bright area (Batch 3).

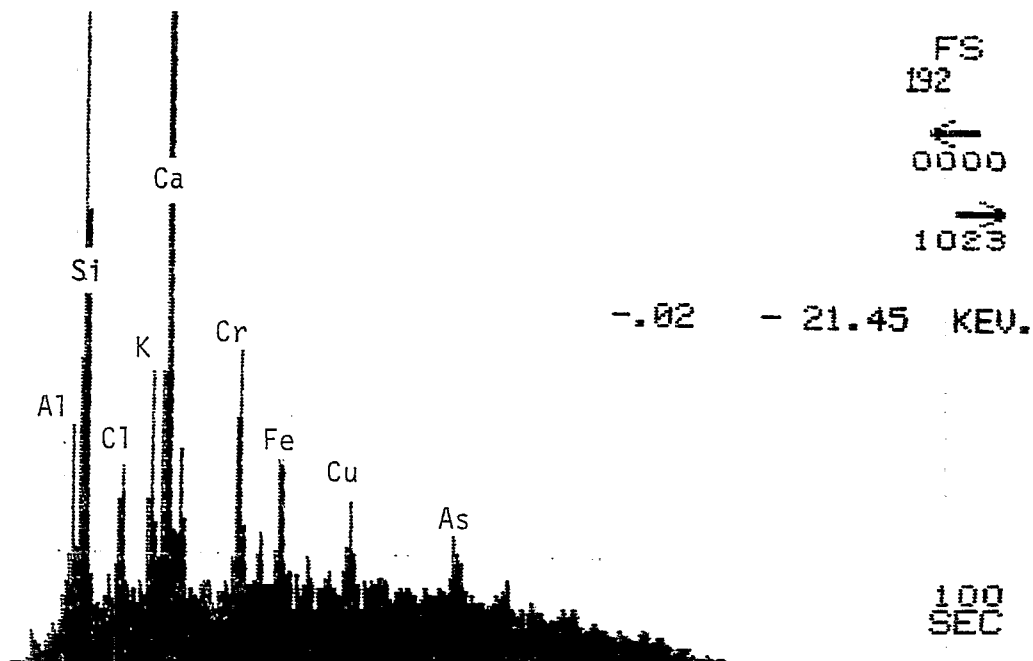


Figure 6-30. Elemental spectrum of area in Figure 6-29 (Batch 3).

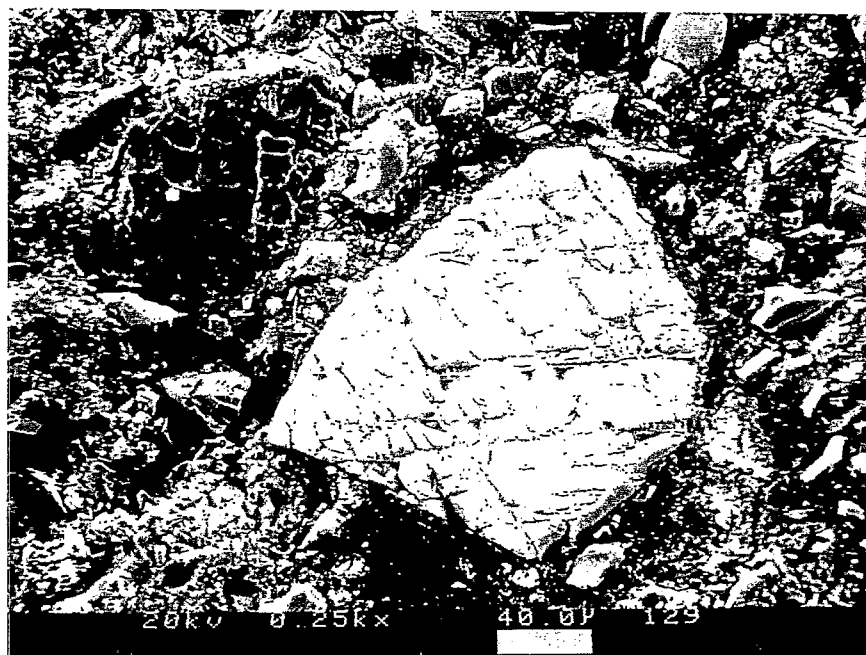


Figure 6-31. BSE image. Ti was detected in the particle (Batch 5).

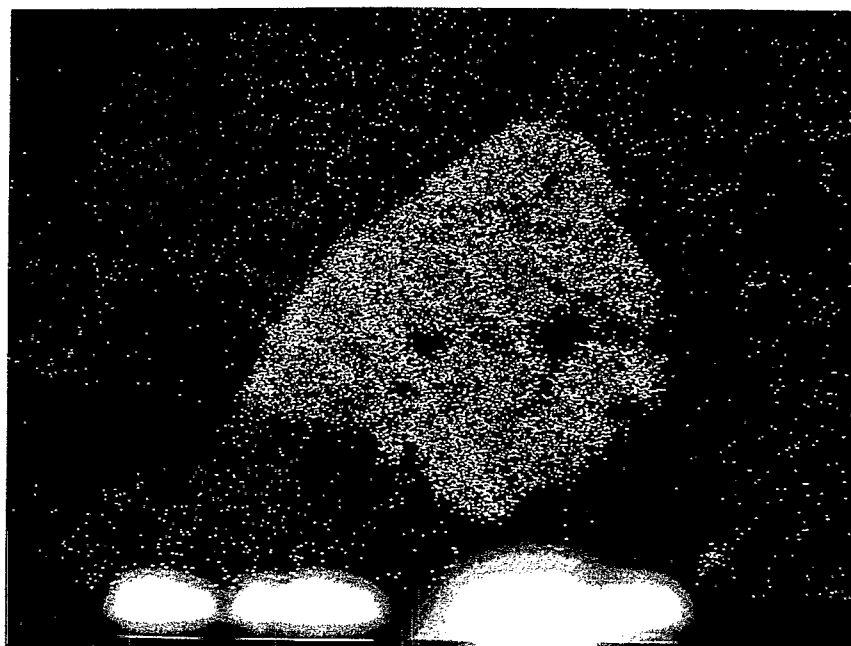


Figure 6-32. Ti map of Figure 6-31 (Batch 5)

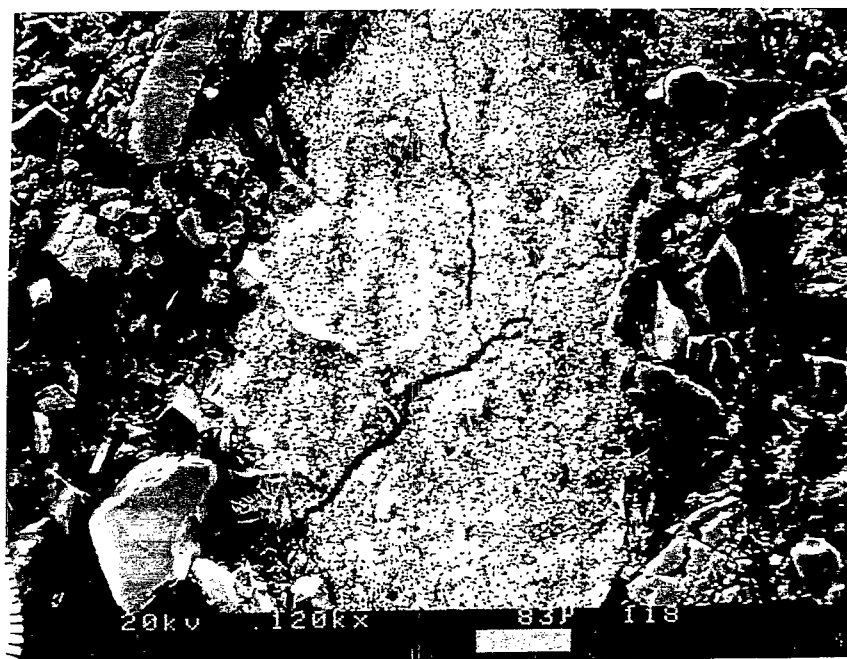


Figure 6-33. BSE image. Cu, Cr, and As were detected in the bright area (Batch 5).

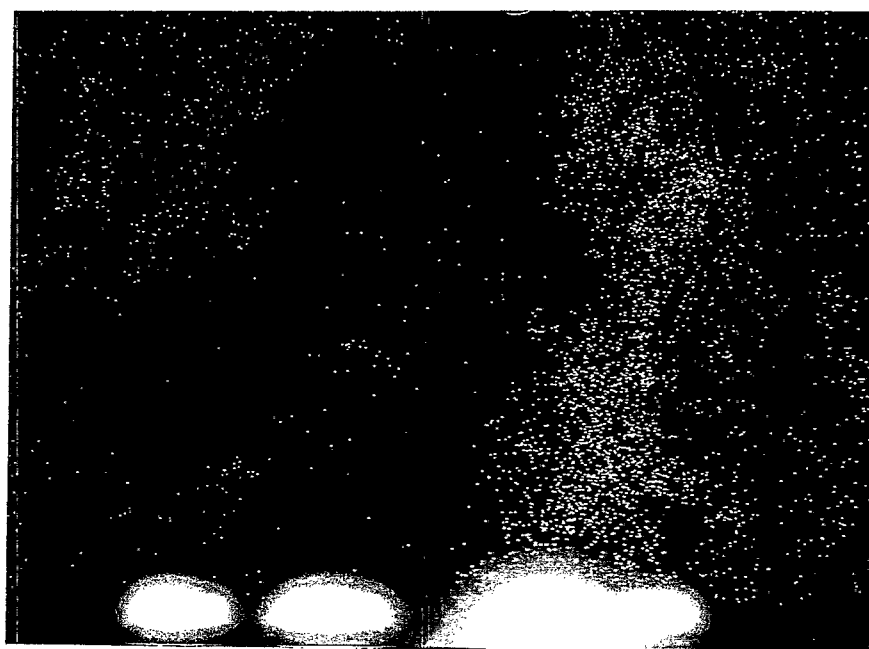


Figure 6-34. Cr Map of Figure 6-33 (Batch 5).

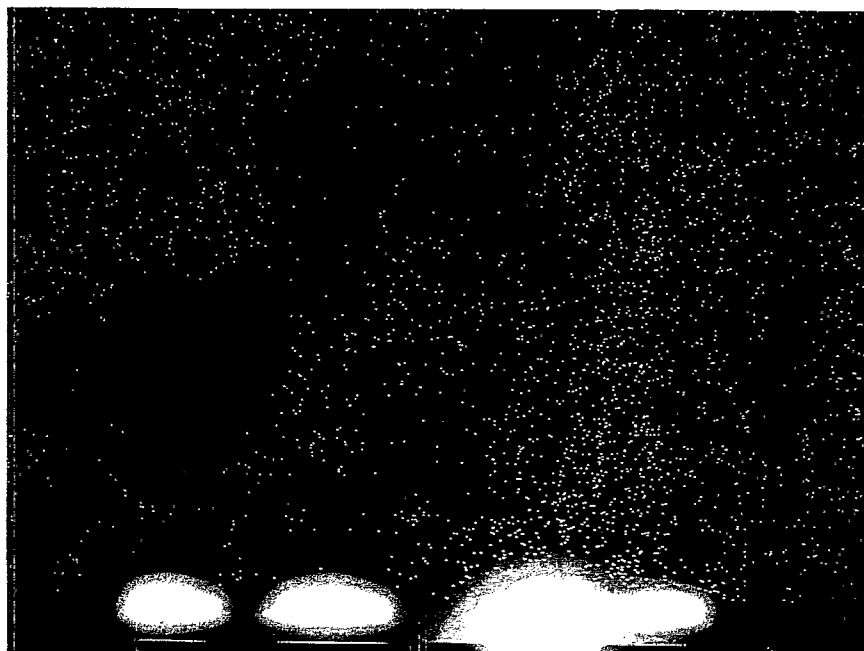


Figure 6-35. Cu map of Figure 6-33 (Batch 5).

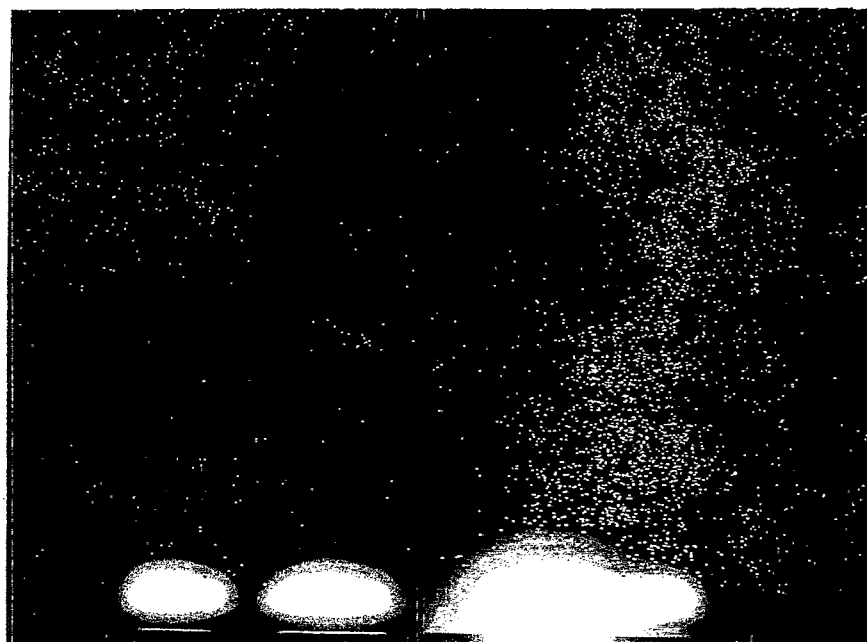


Figure 6-36. As map of Figure 6-33 (Batch 5).

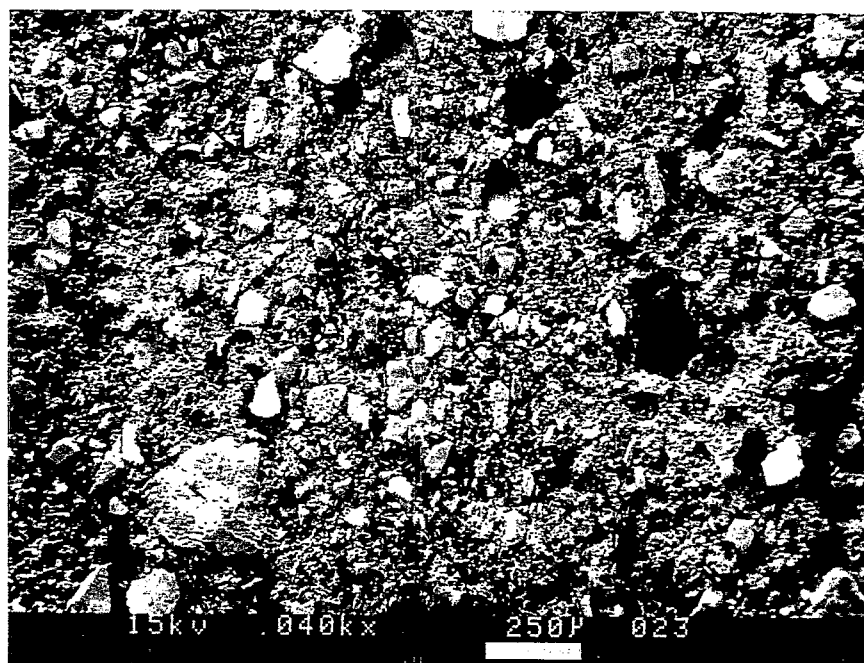


Figure 6-37. BSE image of typical distribution (Batch 1).

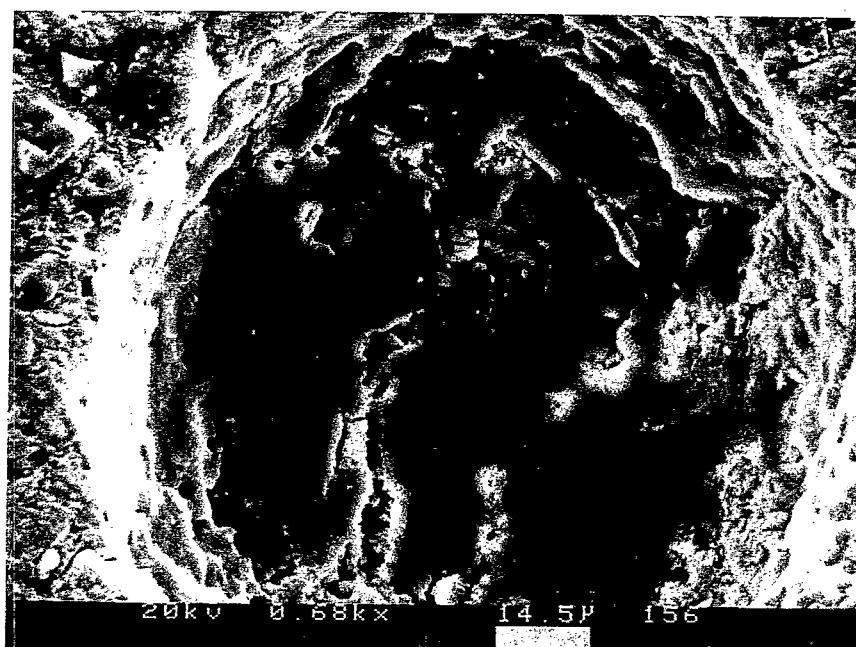


Figure 6-38. Air void in paste (Batch 1).

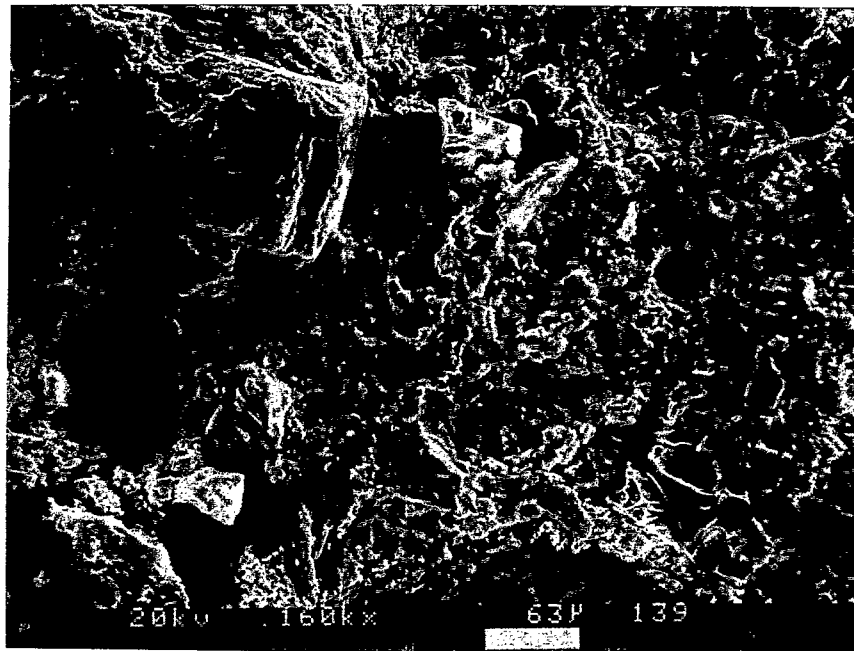


Figure 6-39. SE image of paste and aggregate (Batch 3).

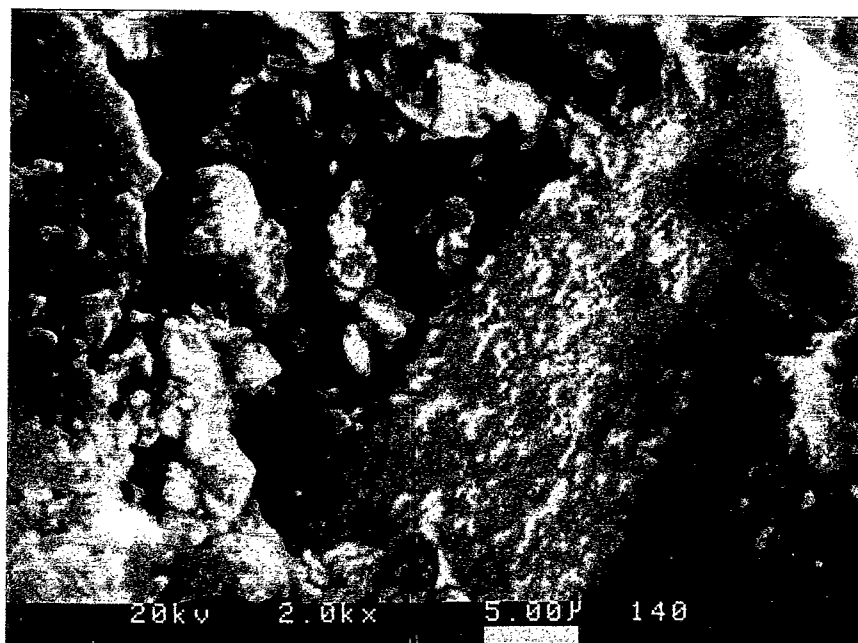


Figure 6-40. Aggregate paste bond (Batch 3).

Four raw waste samples and six solidified waste samples were analyzed -- using FTIR techniques -- to evaluate the thermal stability of the treated waste samples relative to raw waste samples. Each of the solidified waste samples were crushed to a diameter of 3/8 inch or less, but were large enough to be retained on a No. 8 sieve. Solidified waste processed to pass a No. 150 sieve was also evaluated for each batch.

Approximately 50-250 milligrams (mg) were taken from each sample and heated sequentially at 100°, 200°, 300°, 400°, and 500°C in a Wilks Pyro-chem pyrolysis unit. Infrared spectra of the condensed organics volatilized from the sample and weight loss data were recorded after each pyrolysis run. A few of the solidified waste samples were pyrolyzed at 600°C after all of the data gathered at lower temperatures showed no evidence of organic materials released on heating. The infrared data were collected using a Bio-Rad FTS-7 Fourier Transform infrared spectrophotometer. To assure an adequate signal-to-noise ratio for the infrared data, 250 scans were coded at four wavenumber resolution. All spectra were obtained using attenuated total reflectance (ATR) techniques. It was determined from the analyses of the raw waste samples that less than 1 percent (by weight) of organic volatiles could be detected by this technique.

These analyses were directed toward the condensed volatile species because it was believed that higher-boiling species make up the bulk of the organic contaminants in the soils. It should be noted that water and lower-boiling aromatic species (such as toluene and xylenes), which might be present in the soils, were not detected by the method of analysis used in this investigation.

Tables 6-28 and 6-29 depict the pyrolysis weight loss data for both the raw and treated waste samples. Based on this data, the total amount of organic materials volatilized from the raw soil samples was approximately 1 to 2 percent (by weight) of the samples. Since some weight loss was recorded after each pyrolysis run, it is believed that a substantial amount of adsorbed water was also released from the waste samples upon heating. The weight loss after each pyrolysis run of the solidified waste material is believed to be due to the presence of water both as an adsorbed species and as water of hydration of the silicate matrix.

Pyrolysis of the raw waste samples showed that the organic compounds were volatilized between 200°C and 300°C. There is no evidence of the presence of condensed organic compounds below 200°C or above 300°C. The organic materials volatilized from the four raw waste samples

were all similar in composition. Infrared analysis identified these materials as a mixture of aliphatic and aliphatic-substituted aromatic hydrocarbon compounds. The spectra also showed evidence for the presence of carboxylic acid groups (possibly from the oxidation of unsaturated hydrocarbon species), and nitrogen-hydrogen bonds present as amine or amide groups. This composition is consistent with the residue from a heavy oil, such as diesel oil. PCP was not specifically detected in the infrared spectra of the organic pyrolyzates.

Table 6-28. Pyrolysis Percentage Weight Loss Data for Raw Wastes

Temperature (°C)	Batch			
	1	3	4	5
100	2.49	1.92	0.99	0.18
200	0.78	0.70	1.16	2.18
300	0.93	1.05	1.65	1.45
400	0.62	0.35	0.83	0.91
500	0.62	0.00	0.99	---

Table 6-29. Pyrolysis Percentage Weight Loss Data for Treated Wastes

Temperature (°C)	Batch					
	RM	1	1 - QC	3	4	5
100	2.87	2.88	2.74	3.23	5.87	12.07
200	3.06	9.05	4.25	7.32	7.45	3.37
300	1.72	1.59	7.40	7.04	2.42	0.90
400	0.13	1.69	1.77	0.72	0.09	0.41
500	1.02	1.59	0.53	0.50	0.84	0.49
600	0.00	---	---	---	1.30	0.66

Pyrolysis of the solidified waste sample showed almost no evidence for the release of organic contaminants. No organic compounds were detected after pyrolysis of the solidified waste, either in the form of large chunks (3/8-inch diameter), or as a processed powder (<150 mesh). One exception yielded a small amount of primarily aliphatic hydrocarbon species, after pyrolysis of the sample in chunk form, at 400°C. Although these results are only qualitative, they indicate that any volatilization of organic components in the STC-treated waste available for volatilization were substantially retarded by the STC silicate matrix.

Samples of both the treated wastes and the reagent blank mixture remained on site for 32 months following the demonstration to evaluate the long-term stability of the treated waste. Samples in uncovered containers were stored in ventilated lockers at ambient temperatures. Bulk treated waste was cast by batch into cardboard cylindrical forms (which were removed after a 28-day curing period). The resulting monoliths were stored in a lined, uncovered, open-air pad.

Long-term samples were analyzed at 6, 18, and 32 months after the STC demonstration using TWA for PCP and TCLP for arsenic, chromium, and copper. The 32-month cured samples were also analyzed using TCLP-Distilled Water for arsenic, chromium, and copper. Physical testing included unconfined compressive strength tests at 6, 18, and 32 months and permeability testing at 32 months. Results for the long-term TWA and TCLP tests are presented in Table 6-30. Averages of six samples for the analyses of 6-month and 32-month cured samples and four samples for the analyses of 18-month cured samples are compared to both the raw waste samples analyses and the treated 28-day cured sample analyses. Calculated percent reductions are also presented.

In general, the chemical stability of PCP treated by the STC process as measured by TWA was maintained over the 32-month period. TWA of PCP after the 6-month period showed somewhat greater extractable concentrations for Batches 4 and 5 than after the initial 28-day period. Extractable concentrations of PCP generally remained consistent for Batches 1 and 3 over this time period. The 18-month analyses showed decreased concentrations of PCP in the treated waste; however, Batch 5 continued to show considerable analytical variability. Percent reductions following the 18-month period averaged 96 percent reduction. For Batches 3, 4, and 5, the 32-month analyses were consistent with the 18-month analyses and did not indicate any significant changes in concentrations of PCP in the treated waste. However, Batch 1 did show an apparent increase in the concentration of PCP in the treated waste (mainly due to an outlier concentration more than 4 times greater than the average of the other 5 samples). As a result, the percent reduction for Batch 1 decreased from 98 percent at 18 months to 86 percent at 32 months. (Discarding the outlier concentration results in a Batch 1 percent reduction of 91 percent at 32 months.) Percent reductions following the 32-month period averaged 93 percent reduction.

Table 6-30. Long-Term Test Results

PCP-TWA	Concentrations (mg/kg)				
Batch	Raw Waste	Treated Waste			
		28-day	6-month	18-month	32-month
1	2,600	200 ^c	54 ^c	44 ^{c,q}	170
	1,500	82 ^c	106 ^c	24 ^c	74
	2,000	140 ^c	106 ^c	20 ^c	110
	2,600	106 ^c	140	19 ^c	180
	3,400	106 ^c	100	NA	88
	2,000	106 ^c	120	NA	530 ^s
Average	2,350 ± 660	120 ± 42	100 ± 29	27 ± 12	190 ± 170
% Reduction ^{a,b}	---	91	92	98	86
3	1,700 ^f	34 ^c	120	26 ^c	110 ^c
	2,200 ^c	106 ^c	97	9 ^c	44
	2,200 ^f	106 ^c	69	42 ^c	28
	2,000	106 ^c	110	43 ^c	30
	2,200	54 ^c	100	NA	13
	1,600	106 ^c	100	NA	71
Average	2,000 ± 270	85 ± 33	99 ± 17	30 ± 16	49 ± 36
% Reduction ^{a,b}	---	92	91	97	96

Table 6-30. Long-Term Test Results (continued)

PCP-TWA	Concentrations (mg/kg)				
Batch	Raw Waste	Treated Waste			
		28-day	6-month	18-month	32-month
4	6,400 ^f	106 ^e	520	100 ^e	110
	6,800	106 ^e	340	100 ^e	160
	8,900	106 ^e	320	100 ^e	160
	7,300	106 ^e	420	94 ^g	70
	9,000	106 ^e	280	NA	46
	7,800	200 ^{cs}	360	NA	85
Average	7,700 ± 1,100	120 ± 38	370 ± 85	99 ± 3	105 ± 47
% Reduction ^{ab}	---	97	91	98	98
5	6,300	106 ^e	900	1,200 ^g	500
	10,000	106 ^e	640	58 ^c	480
	9,600	400 ^e	550	220 ^c	280
	8,300	370 ^e	560	160 ^c	280
	7,000	106 ^e	1,660 ^g	NA	510
	8,700	NA	510	NA	440
Average	8,300 ± 1,400	220 ± 150	800 ± 440	410 ± 530	415 ± 110
% Reduction ^{ab}	---	95	83	91	91

Table 6-30. Long-Term Test Results (continued)

Arsenic-TCLP		Concentrations (mg/L)			
Batch	Raw Waste	Treated Waste			
		28-day	6-month	18-month	32-month
1	1.3	0.048 ^g	0.19	0.062 ^d	0.56
	2.2	0.096 ^g	0.32	0.071 ^d	0.57
	1.3	0.051 ^g	0.18	0.063 ^d	0.57
	2.2	0.044 ^g	0.17	0.087 ^d	0.16
	2.3	0.19 ^{g,q}	0.24	NA	0.19
	1.6	0.084 ^g	0.33	NA	0.36
Average	1.8 ± 0.47	0.086 ± 0.055	0.24 ± 0.071	0.071 ± 0.011	0.40 ± 0.19
% Reduction ^{a,b}	---	92	77	93	61
3	1.1	0.091 ^g	0.30	0.073	0.12
	0.99	0.081 ^g	0.29	0.060	0.17
	0.97	0.10 ^g	0.28	0.056	0.16
	0.88	0.16 ^{g,s}	0.25	0.24 ^q	0.16
	0.93	0.097 ^g	0.22	NA	0.19
	1.5 ^q	0.078	0.23	NA	0.22
Average	1.1 ± 0.23	0.10 ± 0.030	0.26 ± 0.033	0.11 ± 0.089	0.17 ± 0.033
% Reduction ^{a,b}	---	83	56	82	72

Table 6-30. Long-Term Test Results (continued)

Arsenic-TCLP		Concentrations (mg/L)			
Batch	Raw Waste	Treated Waste			
		28-day	6-month	18-month	32-month
4	2.0	0.91 ^s	0.37	0.21	0.37
	2.3	0.76 ^s	0.79	0.18	0.86
	2.3	0.67 ^s	0.67	0.13	0.70
	2.2	1.1 ^s	0.69	0.22	0.60
	3.6 ^s	0.84 ^s	0.43	NA	0.15
	2.0	0.97 ^s	0.73	NA	0.13
Average	2.4 ± 0.60	0.875 ± 0.15	0.61 ± 0.17	0.185 ± 0.040	0.47 ± 0.30
% Reduction ^{ab}	---	35	55	86	65
5	3.1	0.56	0.37	0.19	0.54
	3.6	0.47	0.24	0.15	0.30
	3.3	0.61	0.62	0.19	0.58
	3.3	0.65	0.58	0.165	0.70
	3.8	0.60	0.53	NA	0.27
	2.9	0.40	0.45	NA	0.72
Average	3.3 ± 0.33	0.55 ± 0.10	0.465 ± 0.14	0.17 ± 0.020	0.52 ± 0.19
% Reduction ^{ab}	---	71	75	91	73

Table 6-30. Long-Term Test Results (continued)

Chromium-TCLP		Concentrations (mg/L)			
Batch	Raw Waste	Treated Waste			
		28-day	6-month	18-month	32-month
1	0.051	0.24	0.27	0.34	0.65
	0.21	0.25	0.29	0.405	1.1
	0.053	0.24	0.29	0.42	0.70
	0.20	0.25	0.28	0.255	0.50
	0.22	0.24	0.28	NA	1.2
	0.052	0.25	0.28	NA	0.46
Average	0.13 ± 0.087	0.245 ± 0.005	0.28 ± 0.008	0.355 ± 0.075	0.77 ± 0.31
% Reduction ^{a,b}	---	-230	-280	-380	-940
3	0.05	0.19	0.27	0.27	0.48
	<0.05	0.20	0.24	0.26	0.50
	<0.05	0.18	0.25	0.30	0.53
	<0.05	0.17	0.24	0.35	0.51
	<0.05	0.18	0.25	NA	0.40
	<0.05	0.20	0.24	NA	0.42
Average	<0.05	0.19 ± 0.012	0.25 ± 0.012	0.295 ± 0.040	0.47 ± 0.052
% Reduction ^{a,b}	---	NC	NC	NC	NC

Table 6-30. Long-Term Test Results (continued)

Chromium-TCLP	Concentrations (mg/L)				
Batch	Raw Waste	Treated Waste			
		28-day	6-month	18-month	32-month
4	0.071	0.29	0.52	1.09	1.7
	0.073	0.28	0.55	1.23	1.6
	0.077	0.29	0.36 ^a	1.29	1.3
	0.083	0.27	0.55	1.13	1.5
	0.23 ^s	0.27	0.58	NA	1.3
	0.084	0.27	0.52	NA	1.3
Average	0.10 ± 0.062	0.28 ± 0.010	0.51 ± 0.078	1.2 ± 0.091	1.45 ± 0.18
% Reduction ^{a,b}	---	-390	-810	-2,000	-2,500
5	0.24	0.32	0.67	0.82	1.2
	0.25	0.31	0.58	1.1	1.3
	0.24	0.33	0.68	1.2	1.2
	0.28	0.29	0.61	1.1	1.1
	0.37 ^a	0.29	0.65	NA	1.1
	0.23	0.38	0.60	NA	1.1
Average	0.27 ± 0.053	0.32 ± 0.033	0.63 ± 0.04	1.1 ± 0.16	1.2 ± 0.082
% Reduction ^{a,b}	---	-110	-310	-610	-680

Table 6-30. Long-Term Test Results (continued)

Copper-TCLP	Concentrations (mg/L)				
Batch	Raw Waste	Treated Waste			
		28-day	6-month	18-month	32-month
1	2.2	0.087	0.12	0.29	0.10
	4.5	0.085	0.11	0.39	0.41 ^a
	2.2	0.090	0.11	0.59	0.11
	4.4	0.089	0.11	0.18	0.07
	4.5	0.089	0.13	NA	0.11
	2.5	0.099 ^a	0.12	NA	0.08
Average	3.4 ± 1.2	0.090 ± 0.005	0.12 ± 0.008	0.36 ± 0.17	0.15 ± 0.13
% Reduction ^{ab}	---	95	94	81	92
3	1.5	0.074	0.16	0.13	0.08
	1.5	0.077	0.13	0.17	0.08
	1.4	0.070	0.13	0.175	0.09
	1.2	0.067	0.12	0.099	0.07
	1.2	0.071	0.16	NA	0.14
	1.5	0.088	0.14	NA	0.13
Average	1.4 ± 0.15	0.075 ± 0.007	0.14 ± 0.017	0.14 ± 0.036	0.098 ± 0.029
% Reduction ^{ab}	---	91	82	82	88

Table 6-30. Long-Term Test Results (continued)

Copper-TCLP	Concentrations (mg/L)				
Batch	Raw Waste	Treated Waste			
		28-day	6-month	18-month	32-month
4	6.2	0.10	0.25	0.56	0.14
	6.1	0.10	0.25	0.87	0.17
	6.2	0.11	0.25	1.26	0.22
	6.5	0.10	0.27 ^a	0.92	0.17
	8.7 ^a	0.10	0.25	NA	0.10
	5.5	0.11	0.25	NA	0.10
Average	6.5 ± 1.1	0.10 ± 0.005	0.25 ± 0.008	0.90 ± 0.29	0.15 ± 0.046
% Reduction ^{a,b}	---	97	93	75	96
5	9.2	0.055	0.17	0.96	0.10
	9.3	0.058	0.27 ^a	1.4	0.11
	8.8	0.085 ^a	0.16	1.8	0.11
	9.5	0.054	0.13	1.6	0.10
	12 ^a	0.057	0.14	NA	0.10
	7.8	0.064	0.14	NA	0.09
Average	9.4 ± 1.4	0.062 ± 0.012	0.17 ± 0.052	1.44 ± 0.36	0.10 ± 0.008
% Reduction ^{a,b}	---	99	97	74	98

Table 6-30. Long-Term Test Results (continued)

NA = Not analyzed

a = Percent Reduction = $\left[1 - (1 + \text{Additives Ratio}) \times \frac{\text{Concentration of Treated Waste}}{\text{Concentration of Raw Waste}} \right] \times 100.$

b = The additives ratio is the weight of additives, including water of hydration, divided by the weight of raw wastes. Values are 0.761, 0.764, 0.776, and 0.746 for Batches 1, 3, 4, and 5, respectively.

c = Value reported is an estimated concentration; compound was detected at an amount less than the reporting limit.

d = The analyte was found in the associated blank as well as in the sample.

e = Estimated concentration using twice the method detection limit.

f = Reported concentration is calculated using a greater dilution than the primary analysis.

g = Spiked sample recovery not within control limits.

q = Fails Q test (Shoemaker, 1974); value may be disregarded with 90% confidence (although it has been used to calculate the average value).

s = Value may be disregarded with 95% confidence (although it has been used to calculate the average value).

No consistent trends emerged from TCLP testing for arsenic in the long-term treated wastes. Average 6-month TCLP leachate concentrations of arsenic for Batches 1 and 3 increased from the 28-day concentrations. Average arsenic concentrations in Batches 4 and 5 were slightly lower in the 6-month tests; however, high analytical variability for these two batches indicates that the arsenic content in the leachate of the 6-month cured samples was similar to that of the 28-day cured sample leachate. Percent reduction for the 6-month period ranged from 77 to 55 percent. The TCLP leachate concentrations of the 18-month cured samples for arsenic showed significant decreases from the 28-day and 6-month leachate concentrations. Percent reductions improved over the 18-month period, initially ranging from 35 to 92 percent reduction after 28 days to 82 to 93 percent after 18 months. The TCLP leachate of the 32-month cured samples showed increases in concentration of arsenic from the concentrations observed at the 18-month period. The average percent reduction for arsenic across all batches decreased from 88 percent at 18-months to 68 percent at 32 months. Comparing the 32-month cure data to the 28-day cure data for arsenic presents mixed results. For Batches 1 and 3 the leachate concentration of arsenic increased over the long term. Consequently, the percent reductions for Batches 1 and 3 decreased from the 28-day cure to the 32-month period. However, for Batches 4 and 5 the leachate concentration of arsenic decreased over the long term. Percent reductions for Batches 4 and 5 increased from the 28-day cure to the 32-month period.

Although chromium was not a critical contaminant for treatment based on its low leachable concentrations in the raw waste, leachable concentrations in the treated waste steadily increased over time to approximately three times the concentrations obtained following the 28-day cure. Average percent reductions for chromium dropped from -243 percent reduction after the initial 28-day curing period to -467 percent reduction after 6 months. Chromium concentrations in the leachate of the 18-month cured samples showed increases of between 20 to 135 percent with time, resulting in greater negative percent reductions. The chromium concentration in the leachate of the 32-month cured samples continued to show increases with time, resulting in even greater negative percent reductions. The average increase in chromium concentration in the leachate from the 18-month cure to the 32-month cure was 52 percent. The average increase in chromium concentration in the leachate from the 28-day cure to the 32-month cure was 271 percent. Average percent reduction for chromium across all batches decreased from -243 percent at 28 days to -987 percent at 18 months to -1,358 percent at 32 months.

With the exception of the 18-month cured samples, leachable concentrations of copper remained consistent over the long-term study. Copper showed no significant change in TCLP-

extract concentrations for Batches 1, 4, and 5 between the 28-day cure and 6 months. Batch 3 showed a slight increase in TCLP-extract concentration after 6 months. This resulted in a decrease in the percent reduction for copper from 90 to 82 percent for Batch 3. Average percent reduction for copper after 6 months was 92 percent. After 18 months, average percent reductions for copper dropped to 78 percent reduction. The copper concentrations in the leachate at 32 months showed slight to moderate decreases from the leachate concentrations for the 18-month cure samples. Average percent reduction for copper across all batches increased to 93 percent for the 32-month cure. With the exception of Batch 5, the copper concentration in the leachate for the 32-month samples statistically did not increase from the concentrations in the leachate for the 28-day samples.

Table 6-31 presents the TCLP-Distilled Water results for arsenic, chromium, and copper. The TCLP-Distilled Water leachate concentrations of arsenic were essentially unchanged from the 28-day to the 32-month samples. Percent reductions were 98 percent for all batches except Batch 4 which decreased from 98 percent at 28 days to 93 percent at 32 months. The chromium concentration in the TCLP-Distilled Water leachate increased from the 28-day to the 32-month samples. Percent reductions for chromium decreased significantly from the 28-day to the 32-month samples. The percent reductions for copper after 32 months decreased moderately from the 28-day samples. Average percent reduction for copper decreased from 89 percent at 28 days to 82 percent at 32 months.

Table 6-32 presents additional long-term (18-month) results for samples collected from the weathered monoliths that remained exposed on the demonstration site. TWA for PCP and TCLP for arsenic, chromium, and copper were used to evaluate the level of leaching from both the surface and interior cores of the monoliths. These results are compared to average raw waste concentrations for each of the batches. Chromium is the only contaminant that showed increased concentrations in the weathered monolith cores, as well as slight increased concentrations in the weathered surface over the 18-month time period. In general, the weathered monolith surfaces had higher concentrations of contaminants than the weathered monolith cores. One exception is chromium in Batch 3 for which the weathered core contained greater leachable quantities than the weathered surface. Table 6-33 shows additional long-term ion-speciation analyses for chromium (VI) relative to total chromium in both raw and treated waste TCLP-Distilled Water extracts for Batch 5. The wastes were analyzed 8 months after the demonstration, revealing greater leachable quantities for both chromium (VI) and total chromium in the treated waste. The 8-month leachate

Table 6-31. Analytical Results for TCLP-Distilled Water Leachates

Constituent: Arsenic			
Batch	Concentrations (mg/L)		
	Raw Waste	Treated Waste	
		28-day	32-month
1	0.95 ^c	<0.01 ^f	0.02 ^g
	1.0 ^c	<0.01	<0.003 ^h
	0.71 ^c	<0.01	<0.003 ^h
	0.82 ^c	<0.01	NA
	0.91 ^c	<0.01	NA
	0.42	<0.01	NA
Average	0.80 ± 0.21	<0.01	<0.009
Percent Reduction	--	>98	>98
3	0.66 ^c	<0.01	<0.003 ^h
	0.72 ^c	<0.01	<0.003 ^h
	0.75 ^c	<0.01	0.06 ^g
	0.80 ^c	<0.01	NA
	0.77 ^{cf}	<0.01	NA
	0.65	<0.01	NA
Average	0.725 ± 0.060	<0.01	<0.02
Percent Reduction	--	>98	>95
4	1.3	<0.010	0.06
	1.3	0.010 ^f	0.03
	1.3	0.013 ^g	0.07
	1.1	<0.010 ^f	NA
	1.4	<0.010 ^f	NA
	1.1	<0.010	NA
Average	1.25 ± 0.12	<0.011 ± 0.001	0.05 ± 0.02
Percent Reduction	--	>98	93
5	1.2	0.013	0.04 ^g
	1.1	0.013	<0.003 ^h
	0.97	0.011	<0.003 ^h
	1.1	0.011	NA
	0.96	0.013	NA
	1.1	<0.010	NA
Average	1.1 ± 0.09	<0.012 ± 0.001	<0.015
Percent Reduction	--	>98	>98

Table 6-31. Analytical Results for TCLP-Distilled Water Leachates (continued)

Constituent: Chromium			
Batch	Concentrations (mg/L)		
	Raw Waste	Treated Waste	
		28-Day	32-month
1	0.21	<0.05	0.29
	0.31	<0.05	0.28
	0.091	<0.05	0.34
	0.16	<0.05	NA
	0.20	<0.05	NA
	0.17	<0.05	NA
Average	0.19 ± 0.072	<0.05	0.30 ± 0.032
Percent Reduction	--	>54	-178
3	0.20	<0.05	0.36
	0.18	<0.05	0.28
	0.18	<0.05	0.22
	0.066 ^a	<0.05	NA
	0.22	<0.05	NA
	0.19	<0.05	NA
Average	0.17 ± 0.054	<0.05	0.29 ± 0.07
Percent Reduction	--	>48	-201
4	0.079	0.066 ^a	0.84
	0.060	0.055	0.90
	0.10	0.055	0.74
	0.067	0.052	NA
	0.066	0.050	NA
	0.067	0.060	NA
Average	0.073 ± 0.015	0.056 ± 0.006	0.83 ± 0.081
Percent Reduction	--	-36	-1900
5	0.15	0.078	0.73
	0.11	0.077	0.66
	0.063	0.079	0.67
	0.057	0.076	NA
	0.10	0.084 ^a	NA
	0.15	0.079	NA
Average	0.105 ± 0.040	0.079 ± 0.003	0.69 ± 0.037
Percent Reduction	--	-31	-1000

Table 6-31. Analytical Results for TCLP-Distilled Water Leachates (continued)

Constituent: Copper			
Batch	Concentrations (mg/L)		
	Raw Waste	Treated Waste	
		28-day	32-month
1	0.49	0.030	0.04
	0.73	0.030	0.04
	0.25	0.031	0.06 ^a
	0.37	<0.030	NA
	0.45	<0.030	NA
	0.43	0.032	NA
Average	0.45 ± 0.16	<0.0305 ± 0.001	0.05 ± 0.01
Percent Reduction	--	>88	80
3	0.41	0.030	0.05
	0.37	0.030	0.04
	0.36	<0.030	0.03
	0.21 ^a	<0.030	NA
	0.45	<0.030	NA
	0.41	<0.030	NA
Average	0.37 ± 0.084	<0.030	0.04 ± 0.01
Percent Reduction	--	>86	81
4	0.98	0.056	0.08
	1.10	0.053	0.10 ^a
	0.97	0.055	0.08
	0.97	0.055	NA
	1.00	0.055	NA
	0.92	0.051	NA
Average	0.99 ± 0.060	0.054 ± 0.002	0.09 ± 0.01
Percent Reduction	--	90	84
5	0.71	0.032	0.06
	0.54	0.031	0.05
	0.45	0.032	0.06
	0.50	0.031	NA
	0.50	0.034	NA
	0.63	<0.030	NA
Average	0.555 ± 0.097	<0.032 ± 0.001	0.06 ± 0.006
Percent Reduction	--	>90	81

Table 6-31. Analytical Results for TCLP-Distilled Water Leachates (continued)

- a* = Percent Reduction = $\left[1 - (1 + \text{Additives Ratio}) \times \frac{\text{Concentration of Treated Waste}}{\text{Concentration of Raw Waste}} \right] \times 100$.
- b* = The additives ratio is the weight of additives, including water of hydration, divided by the weight of raw wastes. Values are 0.761, 0.764, 0.776, and 0.746 for Batches 1, 3, 4, and 5, respectively.
- c* = Duplicate analysis not within control limits.
- d* = Estimated concentration because compound was detected at an amount less than the reporting limit.
- e* = Reported value was calculated from a greater dilution than the primary analysis.
- f* = Post digestion spike for Furnace AA analysis of control limits (85-115%), while sample absorbance was less than 50% of spike absorbance.
- g* = Reported value is less than the method detection limit. For this data point, the method detection limit value was used to calculate averages and standard deviation.
- q* = Fails *Q* test (Shoemaker, 1974); value may be disregarded with 90% confidence (although it has been used to calculate the average value).

Table 6-32. 18-Month Weathered Monolith Test Results

Constituent	Batch	Raw Waste (ppm) ^a	Treated Waste (ppm) Weathered Monoliths	
			Cores ^b	Surface
PCP-TWA	1	2,350	20	50
	3	1,980	17	NA
	4	7,700	41	NA
	5	8,320	97	260
Arsenic-TCLP	1	1.82	0.088	NA
	3	1.06	0.053	0.170
	4	2.40	0.247	NA
	5	3.33	0.502	0.650
Chromium-TCLP	1	0.13	0.326	NA
	3	<0.05	0.207	0.076
	4	0.10	1.160	NA
	5	0.27	1.325	0.391
Copper-TCLP	1	3.42	0.239	NA
	3	1.38	0.141	0.243
	4	6.53	1.122	NA
	5	9.43	0.767	1.000

NA = Not analyzed

a = Results reported as the mean of six samples.

b = Results reported as the mean of two samples.

contained approximately four times the quantity of total chromium compared to the 28-day TCLP-Distilled Water leachate, and almost three times as much total chromium as the initial raw waste sample, again indicating that STC's stabilization treatment process does not reduce the leaching of total chromium over the long term. Increased quantities of the ion-species chromium (VI) in the 8-month leachate compared to the raw waste values indicate that STC's treatment process results in the oxidation of chromium, thereby rendering it more mobile.

The physical strength on the STC-treated waste was evaluated after 6, 18, and 32 months using the unconfined compressive strength (UCS) test. Results reported in Table 6-34 show an average increase in strength of 43 percent over the 6-month period and 347 percent over the 18-month period. The average strength decreased over the 32-month period, resulting in an average increase of only 243 percent from the 28-day results, compared to the 347 percent increase in strength observed after 18 months. Permeability for all batches decreased over time. Permeability results are reported in Table 6-35. For the 28-day samples the permeability ranged from 0.9×10^{-7} cm/sec to 1.7×10^{-7} cm/sec. For the 32-month samples the permeability ranged from 1.2×10^{-7} cm/sec to 4.2×10^{-7} cm/sec.

6.8 QUALITY ASSURANCE RESULTS

This section of the report presents a summary of quality assurance (QA) validation results for data obtained during the STC demonstration. Of primary interest are the analyses of raw and treated wastes, water, and various leachates for pentachlorophenol (PCP) and arsenic as the critical parameters. Selected non-critical parameters were also included to provide both a characterization of the contaminated soil and a comprehensive evaluation of the proposed technology. Non-critical parameters included volatile organic compounds (VOCs), semivolatile organic compounds other than PCP, metals other than arsenic, oil and grease, Eh, pH, and loss on ignition. All analyses discussed in this section were performed by the Engineering-Science, Inc. Berkeley Laboratory (ESBL). A more detailed discussion of the validation process and its results and findings is presented in the "Quality Assurance Report for the STC Site Demonstration" (Engineering-Science Inc., 1992). In general, the usability of the data generated by ESBL to meet the objectives of the demonstration is not affected by the QA outliers found during validation of the data.

Table 6-33. Long-Term Chromium Analysis -- TCLP-Distilled Water (Batch 5)

Constituent	Raw Waste (ppm)	28-day Treated Waste (ppm)	8-month Treated Waste (ppm)
Chromium (VI)	<0.01 <0.01	NA	0.15 0.18
Total Chromium	0.13 0.12	0.08	0.31 0.32

NA = Not analyzed

Table 6-34. Long-Term Unconfined Compressive Strength

Batch	Unconfined Compressive Strength (psi)			
	28-day ^a	6-month ^a	18-month ^a	32-month
1	301 ± 162	480 ± 41	958 ± 63	688 ± 114 ^b
3	278 ± 20	450 ± 85	763 ± 19	720 ± 48 ^c
4	259 ± 65	350 ± 230	1,017 ± 73	679 ± 109 ^d
5	347 ± 65	420 ± 190	1,375 ± 26	795 ± 136 ^d

a = Results reported as mean and standard deviation of three samples.

b = Results reported as mean and standard deviation of eight samples.

c = Results reported as mean and standard deviation of two samples.

d = Results reported as mean and standard deviation of nine samples.

Table 6-35. Long-Term Permeability Testing

Batch	Permeability (10 ⁻⁷ cm/sec)	
	28-day ^a	32-month ^b
1	1.7 ± 0.40	4.2
3	1.5 ± 0.98	2.8
4	0.9 ± 0.41	1.2
5	1.5 ± 0.27	2.0

a = Results reported as mean and standard deviation of three samples.

b = Results reported are from single samples.

6.8.1 Quality Assurance Review of Critical Parameters

Critical parameters that were identified to evaluate the immobilization technology include PCP and arsenic, both formerly common wood-treating agents. Since QA criteria that apply to the PCP analyses also apply to all other semivolatile analytes, semivolatile analyses in general are discussed here. Similarly, QA issues concerning all metals are discussed here, even though arsenic is the only critical parameter within this fraction.

6.8.1.1 Detection Limits

The detection limits for semivolatile organic compounds, determined by gas chromatography/mass spectroscopy (GC/MS) in accordance with the procedures of a specific method, are the method detection limits (MDL). The MDL is the smallest concentration of an analyte that is distinguishable from background by the instrument used for the analysis. The MDL values presented in the quality assurance project plan (QAPjP) are based upon the values listed in EPA Method 8270. The actual values obtained are dependent upon several test-specific factors: the specific instrument used, the matrix type, dilution factor, and other conditions under which the analysis was performed. Due to excessive concentrations of PCP in some samples, occasionally in the percent range, dilution factors can be in the range of 1,000 to 2,500. Therefore, it was possible that contaminants present at low concentrations relative to PCP may not have been detected. This was not considered a critical issue, however, as PCP contamination was the primary organic contaminant of concern. Detection limits for metals, determined by various methods, are specific to their analysis methods.

6.8.1.2 Holding Times

Different holding times criteria exist for the quantitation of semivolatile organic compounds, depending on whether or not the sample is a leachate. Non-leachate samples were prepared within 14 days of sample collection. A maximum holding time of 40 days was specified between preparative extraction and determinative analysis, so that a maximum 5-day holding time was allowed between field collection and determinative analysis for non-leachate samples. Leachate samples included TCLP, TCLP-Distilled Water, and CALWET samples. A maximum holding time of 14 days was specified between field collection and the beginning of leachate extraction, and a 7-day holding time was specified between the end of leachate extraction and preparative extraction. During this evaluation, the leaching period was considered to end at

leachate filtration. Forty days were specified between preparative extraction and determinative analysis; therefore, a maximum 61-day holding time was considered acceptable from field collection to determinative analysis.

Some semivolatile samples violated holding time criteria. Three specific problems were identified. First, the holding time criteria of 40 days between preparative extraction and determinative analysis was violated in pretreatment and reagent mix total waste analysis. Second, CALWET samples from pretreatment soils often exceeded the maximum allowable holding time from sample collection to leachate extraction. Third, some TCLP-Distilled Water and CALWET samples from pretreatment soils were not filtered immediately after leachate extraction.

In the first holding time issue mentioned above, several samples violated the 40-day maximum holding time between preparative extraction and determinative analysis. Violations ranged between 1 to 12 days over the allowable 40-day holding time. The method blanks associated with these samples met all holding time criteria.

A study was performed by ESBL to determine if the accuracy of the PCP analyses was significantly compromised by violating the 40-day holding time between preparative extraction and determinative analysis. The results of this study were inconclusive due to imprecision, as indicated by high relative percent differences (RPD) between analyses, and a lack of QC information. The study consisted of two experiments. In the first experiment, four samples that violated the 40-day holding time were reanalyzed 34 days beyond the allowable holding time. The RPDs between the original analyses and the re-analyses were 17, 18, 16, and 100 percent. The low RPDs in three of the four samples are evidence that PCP concentrations in the samples remain constant beyond the 40-day holding time. In the fourth sample, the PCP concentration in the re-analysis was three times higher than that of the original analysis, possibly indicating operational error.

The purpose of the second experiment was to determine if PCP concentrations in samples that met all holding time requirements change significantly after holding times have expired. If the PCP concentrations in these samples could be shown to be independent of holding time, then the relevance of the 40-day holding time would be lessened and results from samples analyzed beyond holding time could be allowed to stand unflagged. Four treated waste samples analyzed within the allowable 40-day holding time were reanalyzed 2, 5, 15, and 49 days beyond holding time. PCP concentrations remained relatively constant with time, but precision was poor. RPDs

between the original analyses and their re-analyses varied from 9 to 149 percent. Only three of sixteen RPDs met the usual RPD acceptance limit of 50 percent for PCP. Since the results were too imprecise to be conclusive, samples that violated the holding time received qualifying flags. All positive detections were flagged as estimated values, and all non-detects were flagged with the sample reporting limit as estimated. Since exceeding the allowable holding time could cause a loss of target analytes from the sample, the results of these samples may be biased low.

The second holding time problem involved some CALWET analyses from raw waste samples held in excess of the maximum allowable 14-day holding time between field collection and leachate extraction. Several environmental samples exceeded the maximum allowable holding time by 8 to 10 days. Again, positive results were flagged as estimated, and non-detects were flagged with the reporting limit as estimated.

Third, several samples were shelved for an excessive length of time between the end of leachate extraction and leachate filtration. The leachates were left in light and at room temperature, possibly causing the concentrations of analytes in the samples to drop significantly due to processes such as photo-oxidation. All CALWET samples in one sample group were filtered 6 days after the end of leachate extraction. Also, several TCLP-Distilled Water samples sat for 5 to 6 days between extraction and filtration.

A study was performed by ESDL to determine if the accuracy of the PCP analyses was compromised by the holding times and the storage conditions of the leachate samples between leachate extraction and filtration. Three TCLP leachate samples were analyzed from each of three composite raw waste samples for a total of 9 samples. For each composite, the first leachate sample was filtered immediately after extraction, the second leachate sample was left at room temperature for five days and then filtered, and the third leachate was left at 4°C for 5 days and then filtered. In two composite samples, the PCP concentration was higher in the immediately filtered extract than in the extracts filtered after a 5-day waiting period. The other composite yielded a lower PCP concentration in the immediately filtered extract than in the extracts filtered after a 5-day waiting period. More importantly, results were precise and reproducible. RPDs between PCP concentrations in the immediately filtered samples and the samples left at room temperature for 5 days were found to be 16, 8, and 56 percent. RPDs between PCP concentrations in the immediately filtered leachates and the samples left in refrigeration for 5 days were 41, 6, and 48 percent. By comparison, the maximum allowable RPD for PCP in matrix spike/matrix spike duplicate (MS/MSD) samples is 50 percent. Therefore, any effects that the

holding time and storage conditions had on the PCP concentrations in the leachate were considered minimal in comparison to the precision limitations of the method. Hence, PCP results in leachate samples filtered several days after extraction were considered to be valid, and these data remained unflagged.

Finally, the re-analysis of one TCLP-Cage blank violated the 40-day holding time criteria between preparative extraction and determinative analysis. Positive results were flagged as estimated, and non-detects were flagged with the sample reporting limit as estimated. Concentrations estimated in this sample may be biased low.

Several different holding time criteria exist for the evaluation of metals analyses. For leachate samples, 180 days is allowed between sample collection and the start of the leachate extraction procedure, except for mercury, for which 28 days was allowed. Between preparative extraction and determinative analysis, 28 days was allowed for mercury and 180 days was allowed for the analysis of all other metals. Since preparative extraction dates were not provided, the data could not be validated for this holding time. For waste and water samples, 180 days was allowed between sample collection and determinative analysis for all metals except mercury. A maximum holding time between sample collection and analysis of 28 days was allowed for mercury.

The above holding time criteria for metals were met for all but the CALWET samples from one sample group. For these environmental samples and their associated extraction blank, leachate filtration followed leachate extraction by 13 days. No standard holding time was specified, since metals are not susceptible to photo-oxidation, volatilization, or other degradation processes as organic compounds. Therefore, the quality of the data was not expected to be adversely affected due to the lengthy holding time. Thus, no corrective action was taken for these data.

6.8.1.3 Accuracy and Precision

The accuracy of the analytical results was evaluated using the percent recoveries of surrogate compounds added to each individual sample prior to analysis and the percent recoveries of matrix spike compounds in MS/MSD samples. The precision of the analytical results was evaluated using the relative percent difference (RPD) for percent recoveries of matrix spike compounds in the MS/MSD samples. The recovery of spiking compounds is an indication of the

effect of the sample matrix upon the accuracy of the analysis results. Also, unusually high or low recoveries may indicate operational error.

Both surrogate and spike percent recovery samples were used to determine the precision and accuracy of semivolatile analyses. Based on results of these QA measures, the overall degree of accuracy and precision for semivolatile analyses was considered to be acceptable. Nevertheless, several different sources of matrix interference were identified. The most severe matrix interference problems were attributed to the reagent mixture. It appeared that the spiked compounds reacted with or remained tightly bound to the reagent matrix during sample preparation. Therefore, sample spiking was not considered to be a meaningful assessment of analytical accuracy and precision for samples containing the reagent mixture.

Six surrogate compounds were added to each individual semivolatile sample prior to analysis to obtain information about the accuracy of the analysis. The evaluation of surrogate recoveries served two basic purposes: 1) to evaluate the severity of matrix interference and 2) to determine if any fundamental problems exist with the analytical process. The percent recovery (PR) of surrogate compounds was calculated using the following equation:

$$\%R = \frac{(MS \text{ or } MSD) - SR}{SA} \times 100$$

where:

MS = Matrix spike concentration

MSD = Matrix spike duplicate concentration

SR = Sample result concentration

SA = Concentration of spike added

Matrix interference may reduce the accuracy of an analysis. For example, lower recovery might be expected from a waste sample than from a water sample since a portion of the surrogate compounds may remain adsorbed to the waste. During the STC demonstration, the high concentration of PCP in some of the environmental samples may have presented matrix interference.

Surrogate recoveries were also used to determine if any functional problems existed with the analytical process. Unusually high or low recoveries are often caused by errors in sample extraction, improperly calibrated equipment, or other instrument or operator errors. In such cases, the samples were re-extracted and re-analyzed in order to provide a more accurate result.

Acceptable surrogate recovery intervals were established for each of the six semivolatile surrogate compounds and for each physical state of the samples. Fundamental problems with the analytical procedure or excessive matrix interferences were suspected if the surrogate compounds were not recovered within the acceptable ranges. For samples with surrogate recoveries out of specification, samples were flagged and corrective action was taken. Surrogate recovery percentages were reported for every environmental sample and method blank sample that underwent analysis. If any two surrogates within a base/neutral or acid fraction were out of specification, or if any one surrogate compound was recovered at less than 10 percent, a re-analysis of that sample was performed. In cases where matrix spike/spike duplicate pairs were analyzed and the associated environmental sample was out of specification, the MS/MSD results were considered to be the re-analysis.

Although the majority of the analyses met acceptance criteria, some samples failed to meet surrogate recovery criteria, even after re-analysis. Two sources of possible matrix interference were identified: 1) the reagent mixture used in the demonstration and 2) the compounds spiked into samples as part of the MS/MSD analysis. In addition, surrogate compounds were not recovered from some pretreatment samples that required large dilutions. Finally, operational errors were encountered, requiring re-analysis for the samples involved.

The analysis of MS/MSD samples gives an indication of the accuracy and the precision of analytical results. For semivolatile analyses, eleven compounds were spiked into and recovered from MS/MSD samples. The recovery of spiked compounds is an indication of the effect of the sample matrix upon the accuracy of the analysis results. Also, fundamental problems with the analytical process may be suspected if unusually high or low recoveries were achieved. The recovery of spiked compounds is calculated using the same equation as for percent recovery of surrogate compounds discussed above.

The precision of analytical results is indicated by the RPD between the MS and MSD samples. The RPD gives an indication of the reproducibility of the analytical results. Low RPDs indicate a precise, reproducible analysis, while high RPDs may indicate a matrix effect or

operational error, causing precision problems between duplicate samples. The RPD is calculated using the following equation:

$$RPD = \frac{(MS - MSD)}{(MS + MSD)/2} \times 100$$

where:

MS = Matrix spike concentration

MSD = Matrix spike duplicate concentration

Semivolatile MS/MSD samples were analyzed in conformance with the STC SITE demonstration QAPjP. At least one MS/MSD pair was analyzed for every twenty environmental samples of the same matrix. Percent recoveries were not calculated for the matrix spike compound PCP in samples originally containing high concentrations of PCP. In these samples, spiked amounts of PCP were insignificant relative to the amount of PCP already in the sample, and recoveries were approximately an order of magnitude greater than spiked amounts. Several MS/MSD analyses yielded recoveries and RPDs that did not conform to acceptable limits. Matrix effects were suspected in samples from which spiking compounds were recovered at unacceptable percentages, but met RPD criteria. In these cases, reproducibility of results was demonstrated. More severe matrix effects or operational problems must be suspected if RPD values are also out of the specified control criteria. In all cases where QC criteria were not met, both a calibration check standard and a blank spike sample were analyzed to demonstrate that the laboratory analytical system was in control.

The accuracy of metals analyses was evaluated using spike recoveries for MS samples. Precision of metals analyses was evaluated using RPDs calculated for laboratory duplicate samples. Spike recovery limits range from 75 to 125 percent and do not apply when the sample concentration exceeds the spike concentration by a factor of four or more. Corrective action was taken in cases where percent recovery criteria were violated. In cases where the recovery of a metal spike exceeded 125 percent but the metal was not detected in relevant environmental samples, the data was considered acceptable for use. If the spike recovery of a metal was less than 75 percent or greater than 125 percent, the metal detected in associated environmental samples was qualified as estimated. If a percent recovery of a spiking metal was less than 30 percent and the metal was not detected in associated environmental samples, then the data was qualified as unusable.

Arsenic samples were both precise and accurate. Percent recoveries for arsenic were well within the QC range, except a few minor violations. The worst violation, in MS/MSD results for one sample, was a 131 percent recovery, 6 percent above the upper limit. Results for arsenic were also reproducible. Only two samples had an RPD that exceeded 20 percent. Both of these were waste samples, indicating that a matrix effect may potentially be attributed to the waste medium. Also, percent recovery criteria for metals in general were violated much more often in waste samples than in leachate samples, further indicating a matrix effect caused by the waste. Finally, selenium was often recovered below the minimum acceptable level in TCLP and CALWET leachates. Since selenium results were reproducible, another matrix effect was suspected rather than operational error.

6.8.1.4 Sample Contamination

The existence and magnitude of contamination problems were assessed by evaluating the results of blank analyses. Ideally, blank samples should contain no contaminants. In reality, however, sample contamination is a frequent occurrence, and it is important to determine if trace compounds detected in the environmental samples originate on the site or are introduced in transit or in the laboratory during analytical preparation. The extent of contamination in the method blanks associated with each environmental sample batch was assessed to determine the certainty of the reported contaminant quantities in the environmental samples for both metals and semivolatile organics.

In the semivolatile organic fraction, compounds detected in the method blanks included PCP, phthalate esters, and one detection of N-nitroso-dimethylamine. The detection of N-nitroso-dimethylamine appeared in the one TCLP extraction blank; however, no N-nitroso-dimethylamine was detected in any of the environmental samples. Therefore, it appears that the detection was due to laboratory contamination. The phthalate esters detected in blank samples included diethylphthalate, di-n-butylphthalate, bis(2-ethylhexyl)phthalate, and di-n-octylphthalate. Of these compounds, bis(2-ethylhexyl)phthalate was detected most frequently. All environmental samples associated with phthalate contaminated blanks either were free of phthalate detections or had detections on the same order of magnitude as those in the blanks, indicating that their presence was due to laboratory contamination. Phthalate esters are components of plastic, and are considered to be common laboratory contaminants.

PCP was detected in several method blanks, and is not considered to be a common laboratory contaminant. Since PCP was detected frequently at relatively high concentrations in the environmental samples, some degree of cross contamination was suspected. Standard evaluation procedures were used in each case to determine if the detected PCP was from the sample or resulted from contamination. Three CALWET environmental samples contained PCP concentrations ranging from 10,000 to 14,000 $\mu\text{g/L}$. The blank related to these samples was a CALWET extraction blank which contained 190 $\mu\text{g/L}$ of PCP. A portion of the PCP detected in each environmental sample likely appears as the result of sample contamination, therefore the PCP concentrations may be biased high.

Metals occurring in preparation blanks included sodium, potassium, zinc, aluminum, calcium, and iron. There were no detections of arsenic in any preparatory blanks throughout the analyses. The most frequently occurring blank contaminant for the metals fraction was sodium. Sodium was detected at concentrations ranging from 1 to 9,500 milligrams per liter (mg/L) in aqueous samples. Sodium was detected almost exclusively in TCLP and CALWET extraction blanks. This result was expected, since sodium is a component of the extraction solutions. Detections of these metals in associated environmental samples were qualified indicating that the detected analytes originated in the laboratory or during transit rather than on the job site. Instrument calibration blanks were not reported, and thus the metals data is unqualified with regard to these blanks.

6.8.2 Quality Assurance Review For Non-Critical Parameters

Non-critical parameters were identified in the test plan to provide further information for the characterization of the contaminated soil and for the evaluation of the solidification/stabilization treatment process. Volatile organic compounds (VOC), Eh, pH, loss on ignition, and oil and grease analyses were performed.

6.8.2.1 Detection Limits

The detection limits for volatile organic compounds, determined by GC/MS in accordance with the procedures of EPA Method 8240, are the MDLs. The MDL values presented in the QAPjP are based upon values listed in EPA Method 8240. However, the actual values obtained are dependent upon several sample-specific factors: 1) the specific instrument used; 2) the matrix type; 3) dilution factors; and 4) other conditions under which the analyses were performed. The

only dilutions used in volatile analyses during the course of this project were 1 and 5. Therefore, detections are as low as possible and the analyses were sensitive enough to detect trace levels of contamination.

The detection limits for oil and grease analyses in the QAPjP are based on those listed in EPA Method 413.2. Detection limits for oil and grease were 1 mg/L for aqueous samples and 10 mg/kg for waste matrices. The detection limit in the QAPjP for the loss on ignition analyses was based on that found in ASTM C114. The detection limit for these analyses was 0.2 percent. Since non-detects are not possible in Eh and pH analyses, detection limits are not applicable.

6.8.2.2 Holding Times

Two holding times criteria were specified for volatile analysis of leachate samples. A maximum of 14 days was allowed from sample collection to the start of leachate extraction. Also, a maximum holding time of 28 days was allowed between sample collection and determinative analysis. The holding time for volatile analysis for TWA samples was 14 days from sample collection. All volatile analyses performed in the course of this project were completed within allowable holding times. Holding times were considered to start at the end of the 28-day curing period for treated waste and reagent mixture samples.

A maximum holding time of 28 days between sample collection and determinative analysis was specified for oil and grease. All oil and grease analyses performed during the course of the project met this holding time criteria. Again, the holding time for the reagent mixture and post-treatment samples was considered to begin at the end of the 28-day curing period.

6.8.2.3 Accuracy and Precision

For volatile organic compounds, the accuracy of the analytical results was evaluated using the percent recovery of surrogate compounds spiked into each sample and the percent of matrix spike compounds in MS/MSD samples. Precision of the analytical results was evaluated using the RPD for percent recoveries of spike compounds in the MS/MSD samples. Three volatile surrogate compounds were added to every environmental sample and blank sample prior to volatile organic analysis to obtain information about the accuracy of the analytical system. The recovery of surrogate compounds served two basic purposes: 1) to evaluate the severity of matrix interference and 2) to determine if any operational problems exist with the analytical process. Since some

degree of error is expected, acceptable recovery were established for each of the three surrogate compounds and for each physical state of the samples.

Surrogate compounds were recovered from every environmental sample and method blank sample that underwent analysis. If one or more volatile surrogate compounds were out of specification for a single sample, a re-analysis was performed. In cases where MS/MSD pairs were analyzed and the associated environmental sample was out of specification, the MS/MSD results were considered to be the re-analysis. Ideally, the surrogate recoveries in the re-analysis should closely match those of the original analysis, demonstrating that the analysis is being performed properly and that matrix interference is the source of the problem. However, if the results of the re-analysis were within acceptance criteria, these results were reported and the problem with the original analysis was considered due to instrumentation or operator error.

Corrective action was taken in cases where volatile surrogate recoveries were out of specification. If one or more of the surrogate compounds were out of specification but had recoveries greater than 10 percent, positive results for the sample were flagged as estimates, and non-detects were estimated using sample reporting limits. If any surrogate compound in the volatile fraction was recovered at less than 10 percent, then positive results were flagged as estimates and non-detects were flagged as unusable.

The volatile analyses that did not conform to surrogate recovery specifications were considered out of compliance due to matrix effects and not operational error in the analytical system. Three major sources of matrix interference were identified: 1) the spiking procedure in the MS/MSD analysis, 2) the soil matrix, and 3) the reagent mixture that was being tested in the field demonstration.

MS/MSD sample analyses tended to result in excessive recovery of 1,2-dichloroethane-d4; however, in each case, the accompanying non-spiked sample met surrogate recovery criteria. Also, surrogate compounds in relevant extraction and method blanks were recovered at acceptable percentages, discounting operational error. A similar trend was observed in semivolatile analyses. These results strongly suggest that the compounds spiked into the samples for MS/MSD analysis were a source of matrix interference. Since the environmental samples did not undergo a spiking process, the accuracy of these results is not believed to have been adversely affected.

Another trend observed in the volatile surrogate recovery data was that in some samples, toluene-d8 was recovered slightly in excess of its maximum acceptable recovery limit. The high toluene-d8 was attributed to matrix effects in all cases. Surrogate recovery results were shown to be reproducible; relative percent differences between the waste samples and their re-analyses varied from 1 to 6 percent. Also, all extraction and method blanks for volatile analyses met surrogate recovery criteria, discounting operational error.

Two of the samples with high toluene-d8 recovery also failed surrogate recovery criteria for bromofluorobenzene (BFB). One sample had a BFB recovery of 206 percent, suggesting an operational error, while another yielded a BFB recovery of 73 percent, one percentage point below the minimum allowable percentage. Results from both of these samples were flagged unusable. By far, the environmental sample in most serious violation of surrogate recovery criteria was a volatile analysis of the reagent mixture, resulting in a 448 percent recovery of toluene-d8 and no recovery at all for the surrogate compound BFB. The analysis yielded only non-detects and the results were flagged as unusable. Matrix interference from the reagent mixture was suspected to be the cause of these unusual recoveries. The associated MS and MSD samples displayed similar results. All method blanks analyzed in association with the sample had surrogates which met the recovery criteria, ruling out operational error. Semivolatile analyses of samples containing the reagent mixture displayed similar problems with surrogate recoveries. It is postulated that the BFB remained sorbed to the reagent mixture during analysis.

The precision and accuracy of volatile analyses were further evaluated using MS/MSD samples. Five compounds were spiked into and recovered from MS/MSD samples and analyzed for volatile constituents. Percent recoveries and RPDs were calculated using the same equations as were used for the critical parameters. At least one MS/MSD sample pair was analyzed for each twenty samples of the same matrix and dilution factor. All MS/MSD sample pairs except for volatile analysis of the reagent mixture met the recovery criteria. Volatile MS/MSD analyses were further evidence of significant matrix interference caused by the reagent mixture. Percent recoveries were excessive for three of the five spiking compounds in both the MS and MSD samples associated with the reagent mixture. Also, RPDs for three volatile spiking compounds exceeded maximum allowable values. A method blank that underwent MS/MSD analysis on the same date as the reagent mixture samples, met recovery criteria, discounting operational error.

The accuracy and precision of oil and grease analyses was quantitated using MS/MSD analyses. All MS/MSD samples analyzed for oil and grease met acceptance criteria. Percent

recoveries ranged from 85 to 121 percent, which is within the acceptable range of 75 to 125 percent stated in the QAPjP. Although no maximum acceptable RPD has been set, the largest RPD obtained was only 3 percent, indicating that analyses were precise and reproducible.

MS/MSD samples were analyzed at acceptable frequencies. For aqueous matrices, one MS/MSD pair was analyzed from an environmental sample and one was analyzed from a method blank sample. A total of two aqueous samples were analyzed for oil and grease. For the waste, four MS/MSD pairs were analyzed from environmental samples and three were analyzed from method blank samples. A total of 29 waste samples were analyzed for oil and grease.

6.8.2.4 Sample Contamination

The existence and magnitude of contamination problems were assessed by evaluating the results of method blank analyses. Hence, method blanks were examined for contamination and the related environmental samples were evaluated with respect to the identified contamination to determine the certainty of reported values. For both the volatile organic fraction and the oil and grease fraction, method blanks were reported appropriately for each matrix and for each leachate extraction procedure. Method blanks were not analyzed for the other non-critical analyses.

Methylene chloride was detected frequently in volatile method blanks. Concentrations of methylene chloride in the blanks ranged from 8 to 28 $\mu\text{g/L}$ in blank aqueous samples and 11 to 21 $\mu\text{g/kg}$ in blank waste samples. Methylene chloride detections in the environmental samples were roughly of the same order of magnitude as the detections in the method blanks. In the environmental samples, concentrations of methylene chloride ranged from no detection to 21 $\mu\text{g/L}$ in aqueous samples and from no detection to 140 $\mu\text{g/kg}$ in waste samples. Methylene chloride is a common laboratory contaminant, and thus all methylene chloride detections in environmental samples were qualified as non-detects.

Oil and grease were also examined for contamination problems as determined by method blank association. At least one method blank was reported for each matrix. Oil and grease were not detected in any of the method blanks associated with this project.

6.8.3 Overall Completeness of the Analytical Data

The QAPjP defines completeness as an assessment of the amount of valid data obtained from a measurement system, compared to the amount of data that was planned to be obtained so that a particular statistical level of confidence in the data results from the measurement system. This measurement system is comprised of the various analytical methods used throughout the course of the project including GC/MS analysis for volatiles and semivolatiles, atomic absorption for metals, and EPA Method 413.2 for quantitation of oil and grease. The degree of completeness is the number of analyses with acceptable data divided by the total number of samples collected and tested, multiplied by 100.

There were very few data collected in this project that were determined unacceptable. Three volatile analyses, one semivolatile CALWET extraction blank, and four semivolatile environmental samples were flagged unusable due to surrogate recovery violations. The selenium analysis in one sample was flagged unusable due to inadequate recovery of the metal in the MS/MSD analysis. Results for all other metals in the same sample were acceptable. In another sample, silver was flagged unusable due to inconsistent recoveries in the MS/MSD analysis.

The QA objective for the degree of completeness for the STC SITE demonstration was 90 percent. Based on the data collected, the degree of completeness exceeds 99 percent. Therefore, the objective for degree of completeness was met.

6.8.4 Quality Assurance Review For Long-Term Data

This summary presents quality assurance (QA) validation for data obtained during analysis of six month long-term treated samples, including both semivolatile and metal analyses with PCP and arsenic identified as the critical parameters. Six environmental samples underwent total waste analysis by EPA Method 8270; three samples were analyzed for PCP only, and three samples were analyzed for semivolatiles plus tetrachlorophenol (TCP). Metals data was obtained from six environmental analyses of TCLP leachates from treated samples. For each batch, one leachate sample was analyzed for 24 project-specified metals and five leachate samples were analyzed for 5 project-specified metals.

6.8.4.1 Holding Times

Holding times criteria were examined using criteria set forth in the QAPjP. The holding times criteria related to field collection did not apply to the long-term samples. Evidence was provided that exceeding holding times did not severely affect the quality of semivolatile or metals analyses. The holding times between preparative extraction and determinative analysis were met in all cases.

6.8.4.2 Accuracy and Precision

The accuracy and precision of the semivolatile analyses were evaluated using surrogate recoveries and MS/MSD results. Percent recoveries of surrogate compounds were used to evaluate the accuracy of individual analyses; a set of surrogate recovery results was required for every environmental sample. MS/MSD results were used in evaluating both the accuracy and precision of the analyses. Percent recoveries indicated the accuracy, while RPDs between recoveries in the spike and spike duplicate were used as a measure of precision. One MS/MSD pair was required for every 20 samples of the same matrix and concentration level. Results of the MS/MSD analysis affect samples of the same matrix and concentration level.

Surrogate recoveries were reported as required for every environmental and blank sample analyzed for semivolatiles. Twenty-eight environmental samples were analyzed for semivolatiles. Twelve of those did not meet the surrogate recovery criteria. Normally, when surrogate recovery results are unacceptable (two surrogate compounds in the same fraction are outside the recovery range or any one surrogate compound is recovered at less than 10 percent) a re-extraction and re-analysis is performed. However, no re-extractions were performed, since surrogate recoveries were not considered to be a valid test for accuracy due to the sorptive nature of the media. Surrogate compounds were suspected to primarily adsorb to the reagent matrix rather than pass onto the GC column for analysis. Thus, low surrogate recoveries were considered to reflect adsorptive capacity of the reagent mixture rather than the accuracy of the analytical procedure.

The analytical procedure was considered to be acceptable based on surrogate recoveries from blank samples. Surrogate compounds were recovered from six blank samples. Every blank sample yielded recoveries within acceptable intervals, with one exception. One method blank showed a high recovery of 2-fluorobiphenyl. However, no corrective action was required, since 2-fluorobiphenyl is a base/neutral surrogate, and its recovery affects base/neutral analytes only.

PCP, the target analyte, is an acid fraction compound, and all acid fraction surrogates met recovery criteria.

Both accuracy and precision of semivolatile analyses were evaluated using MS/MSD results. MS/MSD analyses were performed for the twelve PCP analyses, and the twelve analyses for semivolatiles plus tetrachlorophenols. All environmental and MS/MSD analyses were of soil samples, evaluated at medium level. Acceptance criteria were met for one pair of MS/MSD samples with the following exceptions: 1) the percent recovery for pyrene was low at 8 percent in both the MS and MSD samples, although the RPD for pyrene was acceptable, and 2) 4-Nitrophenol was not recovered in either MS or MSD sample. These results reflect the sorptive nature of treated waste samples containing the reagent mixture. PCP was recovered high at 132 percent in the MSD sample, causing the RPD to be high. High recovery of PCP was an expected occurrence due to high concentrations of PCP in the environmental sample.

The accuracy and precision of metals analyses was evaluated on the basis of MS/MSD samples. Accuracy was evaluated by examining the percent recoveries, and precision was judged on the basis of relative percent differences in recoveries between the MS and MSD samples. One MS/MSD was required for every twenty similar environmental samples.

One MS/MSD analysis was used to evaluate the 24-metal analyses. Percent recoveries were not calculated for arsenic and calcium because the sample concentrations were greater than four times the matrix spike concentration. Selenium was recovered low at 28 percent in the MS analysis and 25 percent in the MSD analysis; however, the RPD between these figures was acceptable. Selenium was recovered low in previous spiked samples of TCLP and CALWET leachates and thus may be lost in the leaching process. Selenium results in four samples were flagged as unusable.

One MS/MSD analysis was adequate to evaluate the accuracy and precision of the 5-metal analyses, since there were twenty analyses for 5 metals. The results complied with QC criteria. Percent recoveries were not calculated for arsenic, because the sample concentration was greater than four times the matrix spike concentration. Two laboratory control samples were also analyzed to monitor overall performance. In both samples, the laboratory control sample recoveries were within the specified control limits, indicating acceptable results.

In general, the percent recovery for semivolatile surrogates, and acid surrogates in particular, were consistently extremely low. In many of the samples containing the reagent mix, no acid surrogates were recovered. These results suggest that a significant portion of the spiked compounds remained absorbed to the reagent mixture during the analysis. These results also indicate that the reagent mixture would have the same effect on target compounds similar to the surrogates. This trend supports the effectiveness of the reagent mixture on phenolic compounds since all three acid surrogate compounds were phenols.

6.8.4.3 Sample Contamination

Two method blanks were analyzed to determine if contamination problems existed for the semivolatile analyses. No detections occurred in analyses of either of the two method blanks. Thus the environmental analyses were considered to be free of contamination. For the metals analyses, contamination problems were assessed using six blank analyses. Four TCLP extraction blanks were analyzed for 24 metals, one preparation blank was analyzed for 24 metals, and one preparation blank was analyzed for 5 metals. Zinc was the only detected contaminant, appearing in the TCLP extraction blanks at 0.022 to 0.032 mg/L, slightly above the reporting limit of 0.02 mg/L. Two zinc detections in environmental samples were considered to have originated from contamination during laboratory preparation or analysis of the samples, and were thus qualified as non-detects. In the first case, zinc was detected at 0.028 mg/L, with the associated TCLP extraction blank at 0.030 mg/L zinc. The second environmental sample yielded a zinc detection of 0.022 mg/L, while the associated TCLP extraction blank detected zinc at 0.020 mg/L. Both detections of zinc in the two samples were qualified as non-detects.

6.8.4.4 Completeness of Long-Term Data

All semivolatile samples were analyzed with acceptable QC results. Completeness for the semivolatile fraction of the six month long-term samples is therefore 100 percent. In the metals fraction, results for selenium were determined unusable in four samples due to MS/MSD violations. Since there were 196 specific results, the degree of completeness for metals analyses is 98 percent. The degree of completeness for the entire set of data is estimated to be over 99 percent. Thus, the goal of 90 percent stated in the QAPjP has been met.

6.8.5 Quality Assurance Review for Additional Studies

In addition to the analyses described in the STC QAPjP, three studies were conducted to 1) obtain lower reporting limits for the target compound PCP, and TCP (see Section 6.4.1); 2) determine if PCP would be more easily leached from soils at high pH levels (pH = 12) (see Section 6.4.1); and 3) analyze total and hexavalent chromium from TCLP-Distilled Water leachates for one raw waste and two treated waste samples of Batch 5 (see Section 6.7).

6.8.5.1 Holding Times

Holding times were examined using the criteria set forth in the STC QAPjP. The holding times between leachate extraction and preparative extraction and between preparative extraction and analysis were acceptable for all samples of the first two studies involving PCP and TCP. A study conducted as part of this project provided evidence that holding time violations did not severely affect the quality of semivolatile analyses. For the chromium leachate samples, a 180-day holding time was allowed between sample collection and leachate extraction. This holding time was exceeded for all leachate samples, since the work was requested by the EPA Project Manager after the holding time had expired. It was agreed that holding time criteria related to sample collection would not be applied toward specially requested work orders. Additionally, the 180-day holding time requirement between preparative extraction and analysis was met for all samples in this study. Thus, no flags were issued on the basis of holding time requirements for any of the additional studies.

6.8.5.2 Accuracy and Precision

Surrogate recoveries were reported as required for every environmental sample. Violations of surrogate recovery QC criteria were noted for the first study involving PCP and TCP analysis. The surrogate compound, 2-fluorophenol (2-FP), was recovered below the minimum acceptable percentage (25 percent) in all fourteen environmental analyses in the study. 2-FP was recovered at less than 10 percent in ten analyses performed on environmental samples. The only samples with acceptable recovery of 2-FP were the method blanks. The recovery of the base-neutral compound 2-fluorobiphenyl (2-FBP) exceeded the upper recovery limit in three environmental samples. However, since 2-FBP is the only base-neutral compound out of compliance, the acceptance criteria were met and no corrective action was necessary. The acid fraction surrogate phenol-d5 was not recovered in four environmental samples. Also, 2,4,6-tribromophenol, another

acid fraction surrogate, was not recovered in one sample. Finally, in two diluted samples surrogate compounds were not recovered due to dilution.

The low surrogate recoveries from the first study involving PCP and TCP analyses may indicate sorption of the surrogate compounds to the matrix, since these samples contain the reagent mixture, which readily adsorbs some organic compounds. The blank analyses conformed to surrogate recovery criteria, indicating that the analytical system was in control. The above samples were flagged, indicating that they did not meet surrogate recovery criteria; however, surrogate recovery is not a highly valid test for accuracy due to the sorptive nature of the sample. No re-analyses were performed.

In the second study involving PCP leachability at pH 12, surrogate compounds were recovered within acceptable intervals for all environmental and blank samples. Some spike/spike duplicate samples were analyzed as a test for accuracy and precision. A matrix spike sample was analyzed as part of this study. Although this study was not anticipated in the approved QAPjP, all QAPjP and EPA Contract Laboratory Program (CLP) criteria were met, with the exception of one compound, 4-chloro-m-cresol, which was recovered just above its acceptance limit. Since a duplicate was not analyzed, no relative percent differences (RPD) were obtained. The critical matrix spike/spike duplicate (MS/MSD) compound, PCP, met both percent recovery and RPD criteria. For this reason, no flags were issued on the basis of MS/MSD criteria. All internal standard areas were within acceptable CLP acceptance criteria.

The accuracy and precision of the chromium analytical results were evaluated on the basis of MS/MSD samples. Accuracy was evaluated by examining the percent recoveries of spiked analytes, and precision was judged by the RPDs in recoveries between the MS and MSD samples. Percent recoveries and RPDs were in acceptable ranges for both total and hexavalent chromium. One MS/MSD pair was adequate, since only four samples were evaluated in this study. Since all MS/MSD requirements were met, no flags were issued.

6.8.5.3 Sample Contamination

Contamination problems were examined for all three studies. Blank samples were free of detectable contamination in the first study; however in the second study, PCP was detected at 43 µg/L in the extraction blank. PCP concentrations in associated environmental samples ranged from 88,000 µg/L to 320,000 µg/L. Based on the evaluation criteria set forth in CLP "Functional

Guidelines", the data from the environmental samples were valid and required no flagging. The method blank in the second study yielded no detections. An extraction blank and a preparation blank were analyzed in the third study involving chromium analysis in order to determine if any sample contamination had occurred. Neither blank yielded any detections. Therefore, the samples were considered to be free of contamination and no flags were issued.

6.8.5.4 Completeness

All of the data required for the three additional studies were analyzed in an acceptable fashion. No flags were issued for unusable data, indicating that all of the data was usable. Thus, the degree of completeness for the three additional studies is 100%.

6.9 SUMMARY OF RESULTS

The STC immobilization technology reduced the short-term mobility and leachability of arsenic and copper as measured by the TCLP and TCLP-Distilled Water methods. The solidification/stabilization treatment process was also successful in reducing the mobility and potential leachability of PCP as measured by the TCLP-Distilled Water test and TWA. However, leachability was not effectively reduced for chromium as measured by any of the leaching procedures, except possibly the ANS 16.1 test. However, chromium was not targeted for treatment in this demonstration and no specific additives were included to treat chromium. In addition, the CALWET leach test showed very inconsistent trends for all of the analytes.

Based on California state regulatory levels for legal disposal as nonhazardous waste in landfills, the STC treatment process did not consistently meet total (TTLC) or solubility (STLC) threshold limit concentration requirements for SPT waste. CALWET leach results were both below and above California's STLC levels for arsenic, copper, and PCP. TWA for chromium and copper were well below California's TTLC; however, TWA for arsenic and PCP were above California TTLC requirements for both the raw and treated wastes. Federal leach criteria could not be adequately evaluated since TCLP concentrations of arsenic, chromium, and PCP were below federal TCLP regulatory levels in both the raw and treated wastes.

Preliminary evidence based on freeze/thaw and wet/dry durability tests suggests that the physical and structural characteristics of the STC-treated wastes would resist the normal effects of weathering. Low unconfined compressive strengths of the treated waste, although above

minimum levels for disposal in landfills, were not sufficient for construction purposes. Additional tests would be needed to determine the appropriate reagent mixture necessary to meet construction requirements, if desired. Initial six-month TCLP-extract and TWA showed increased concentrations of contaminants released from the treated waste. Eighteen-month analyses showed improved percent reductions for arsenic, averaging 88 percent reduction, and PCP averaging 96 percent reduction. Chromium and copper concentrations showed slight to moderate increases in the TCLP-extracts over time. Unconfined compressive strengths increased an average of 71 percent.

7.0 DEMONSTRATION COSTS

The cost of demonstrating STC's solidification/stabilization treatment process at the Selma Pressure Treating site was approximately \$1.5 million. This cost includes site characterization and preparation, demonstration planning and field work, laboratory analyses, and report preparation. The developer's portion of this cost was approximately \$100,000. The balance of approximately \$1.4 million was allocated to the U.S. EPA SITE Program.

7.1 U.S. EPA SITE CONTRACTOR COSTS

Technical support to the U.S. EPA SITE program for the evaluation of the STC technology was provided by a contractor and subcontractors. Each SITE project is divided into two phases: planning (Phase I) and demonstration (Phase II). Phase I costs are actual costs; Phase II costs include actual costs plus labor estimates through the completion of report preparation. Specific activities under each phase and a cost breakdown for each phase are presented below.

7.1.1 Phase I: Planning Stage

Phase I activities included:

- Solidification/stabilization technology review
- Protocol evaluation
- Site characterization, sampling, and analysis
- Treatability testing
- Development of the demonstration plan
- Site subcontractor procurement

Costs for Phase I activities are summarized below:

• Labor	\$ 117,240
• Equipment and supplies	\$ 8,500
• Travel	\$ 16,980
• Laboratory analyses	\$ 62,860

7.1.2 Phase II: Demonstration Stage

Phase II activities included:

- Mobilization and site preparation
- Sample collection and field oversight
- Sample processing after the 28-day curing period
- Laboratory analyses
- Report preparation
- Long-term testing

Costs for Phase II activities are summarized below:

• Labor	\$494,940
• Equipment and supplies	\$ 85,000
• Travel/transportation	\$ 18,000
• Analytical	\$630,167

Labor costs include estimates through report preparation. Transportation costs include equipment mobilization and demobilization. Analytical costs include long-term testing.

7.2 DEVELOPER (STC) COSTS

The costs presented in this section were provided by STC. These costs were based upon all expenses incurred by STC in preparing for and conducting the SITE demonstration.

• Labor	\$ 25,500
• Travel	\$ 21,710
• Equipment	\$ 34,400
• Transportation	\$ 4,060
• Raw Materials	\$ 3,596
• Miscellaneous	\$ 12,600

Equipment and miscellaneous costs include such one-time costs as health and safety equipment and training. Raw materials costs include the STC proprietary reagents. Transportation costs include equipment mobilization and demobilization.

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