

**TECHNOLOGY EVALUATION REPORT  
RETECH, INC., PLASMA CENTRIFUGAL FURNACE**

**Volume I**

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## **NOTICE**

The information in this document has been funded by the U.S. Environmental Protection Agency under Contract No. 68-CO-0048 and the Superfund Innovative Technology Evaluation (SITE) Program. It has been subjected to the Agency's peer review and administrative review, and it has been approved for publication as a U.S. EPA document. Mention of trade names or commercial products does not constitute an endorsement or recommendation for use.





## 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 by EPA's Office of Solid Waste and Emergency Response (OSWER) and Office of Research and Development (ORD) to enhance the development of hazardous waste treatment technologies necessary for implementing new cleanup standards that require greater reliance on permanent remedies. This is accomplished by performing technical demonstrations that provide engineering and economic data on selected technologies.

This project consists of an analysis of the Retech, Inc. Plasma Centrifugal Furnace. The Demonstration Tests took place at the Department of Energy's Component Development and Integration Facility located in Butte, Montana. The demonstration effort was directed at obtaining information on the performance and cost of the process in order to assess the technology's potential applications at other hazardous waste sites. This Technology Evaluation Report describes the field activities and laboratory results from the Demonstration Tests. An interpretation of the available data, an economic analysis, and a discussion of the potential applicability of the technology is provided in the Applications Analysis Report.

Additional copies of this report may be obtained at no charge from the EPA's Center for Environmental Research Information, 26 West Martin Luther King Drive, Cincinnati, Ohio, 45268, using the EPA document number found on the report's front cover. Once this supply is exhausted, copies can be purchased from the National Technical

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## **ABSTRACT**

A demonstration of the Retech, Inc. Plasma Centrifugal Furnace (PCF) was conducted under the Superfund Innovative Technology Evaluation (SITE) Program at the Department of Energy's (DOE's) Component Development and Integration Facility in Butte, Montana. The furnace uses heat generated from a plasma arc to melt and vitrify solid feed material. The feed soil was a mixture of Silver Bow Creek soil and 10% by weight No. 2 diesel oil, spiked to provide 28,000 ppm zinc oxide and 1,000 ppm hexachlorobenzene in the soil/oil mixture.

Pre-treatment soil and scrubber liquor/makeup sampling was performed to characterize the material inputs to the process. Following treatment, the vitrified soil, scrubber liquor, and stack gas were sampled to determine the technology's suitability for use in destroying and immobilizing contaminants in the test soil. The results from this testing were used to draw conclusions on the technology.

The following conclusions were derived from the test results: (1) the treated soil did not leach any metals at levels above the regulatory limits; (2) the process achieved a Destruction and Removal Efficiency (DRE) of greater than 99.99% for the Principal Organic Hazardous Constituent (POHC); (3) the air pollution control system did not reduce the level of particulate emissions to below the RCRA limit; (4) a high percentage of the metals fed to the furnace are encapsulated in the treated soil; (5) the PCF is advantageous over other incinerator technologies in that it can successfully immobilize heavy metals in the slag, however, this treatment option can be more expensive than conventional incineration systems.

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## **ABBREVIATIONS AND SYMBOLS**

<b>A</b>	<b>amps</b>
<b>ACLs</b>	<b>Alternate Concentration Limits</b>
<b>BTEX</b>	<b>Benzene, Toluene, Ethyl benzene, and Xylene</b>
<b>CDIF</b>	<b>Component Development and Integration Facility</b>
<b>CEM</b>	<b>Continuous Emission Monitor</b>
<b>cf</b>	<b>cubic feet</b>
<b>CFR</b>	<b>Code of Federal Regulations</b>
<b>DAS</b>	<b>Data Acquisition System</b>
<b>DOE</b>	<b>Department of Energy</b>
<b>DOT</b>	<b>Department of Transportation</b>
<b>DRE</b>	<b>Destruction and Removal Efficiency</b>
<b>dscf</b>	<b>dry standard cubic feet</b>
<b>\$</b>	<b>U.S. Dollar</b>
<b>EPA</b>	<b>Environmental Protection Agency</b>
<b>°F</b>	<b>degree Fahrenheit</b>
<b>ft</b>	<b>feet</b>
<b>gal</b>	<b>gallons</b>
<b>gpm</b>	<b>gallons per minute</b>
<b>gr</b>	<b>grains</b>
<b>hr</b>	<b>hour</b>
<b>INEL</b>	<b>Idaho National Engineering Laboratory</b>
<b>kg</b>	<b>kilograms</b>

## ABBREVIATIONS AND SYMBOLS (CONTINUED)

<b>kW</b>	<b>kilowatts</b>
<b>lb</b>	<b>pounds</b>
<b>L</b>	<b>liters</b>
<b>m</b>	<b>meters</b>
<b>mg</b>	<b>milligrams</b>
<b>ORD</b>	<b>Office of Research and Development</b>
<b>OSWER</b>	<b>Office of Solid Waste and Emergency Response</b>
<b>PCDD</b>	<b>polychlorodibenzodioxin</b>
<b>PCDF</b>	<b>polychlorodibenzofuran</b>
<b>PCF</b>	<b>Plasma Centrifugal Furnace</b>
<b>PIC</b>	<b>Product of Incomplete Combustion</b>
<b>POHC</b>	<b>Principal Organic Hazardous Constituent</b>
<b>ppbv</b>	<b>parts per billion, by volume</b>
<b>ppm</b>	<b>parts per million</b>
<b>ppt</b>	<b>parts per trillion</b>
<b>psia</b>	<b>pounds per square inch, absolute</b>
<b>psig</b>	<b>pounds per square inch, gauge</b>
<b>%</b>	<b>percent</b>
<b>RCRA</b>	<b>Resource Conservation and Recovery Act</b>
<b>SAIC</b>	<b>Science Applications International Corporation</b>
<b>SARA</b>	<b>Superfund Amendment and Reauthorization Act</b>
<b>SBC</b>	<b>Silver Bow Creek</b>
<b>scfm</b>	<b>standard cubic feet per minute</b>
<b>SITE</b>	<b>Superfund Innovative Technology Evaluation</b>
<b>TCDD</b>	<b>2,3,7,8-tetrachlorodibenzodioxin</b>
<b>TCDF</b>	<b>2,3,7,8-tetrachlorodibenzofuran</b>
<b>TCLP</b>	<b>Toxicity Characteristic Leaching Procedure</b>
<b>THC</b>	<b>Total Hydrocarbons</b>

## ABBREVIATIONS AND SYMBOLS (CONTINUED)

TICs	Tentatively Identified Compounds
V	volts
VOST	Volatile Organic Sampling Train
wk	week

## **SECTION 1**

### **EXECUTIVE SUMMARY**

#### **INTRODUCTION**

This report summarizes the activities and results of Demonstration Testing of the Plasma Centrifugal Furnace (PCF) technology developed by Retech, Incorporated (Retech). The study was conducted at the U.S. Department of Energy's (DOE's) Component Development and Integration Facility (CDIF) in Butte, Montana under the Superfund Innovative Technology Evaluation (SITE) Program developed by EPA.

The Retech technology, a Plasma Centrifugal Furnace, is a thermal process which uses the heat generated from a plasma torch to decontaminate metal and organic contaminated waste. This is accomplished by melting metal bearing solids and, in the process, thermally destroying organic contaminants. The molten soil forms a hard, glass-like nonleachable mass on cooling. The waste feed used in the Demonstration Tests was comprised of heavy metal-bearing soil from the Silver Bow Creek Superfund Site mixed with 10% by weight No. 2 diesel oil. The mixture was spiked to provide 28,000 ppm of zinc oxide and 1,000 ppm of hexachlorobenzene. In addition to complete monitoring of the system, sampling of all input and output streams was performed during each of three Demonstration Tests.

The goal of the Demonstration Tests was to obtain specific operating, design, analytical, and cost information to evaluate the performance of the pilot-scale Plasma Centrifugal Furnace (PCF-6) under actual operating conditions. Under the SITE Program, the feasibility of utilizing the PCF technology as a viable hazardous waste treatment system at other sites throughout the country was also studied. To this end, the specific critical test objectives were:

- to characterize the residues produced at optimum operation including Destruction and Removal Efficiency (DRE), fate and transport of metals, and residue quality;
- to identify pre- and post-feed waste treatment requirements.
- to evaluate the ability of the Plasma Centrifugal Furnace to effectively vitrify inorganic and metal constituents within a soil into a monolithic nonleachable mass; and
- to determine if the furnace can meet 99.99% DRE for target analytes in a soil contaminated with up to 10% organics.

The SITE Demonstration Tests at the CDIF were conducted between July 22 and 26, 1991. For a one-week period during the Demonstration Tests, EPA SITE Program staff along with their evaluation contractor, Science Applications International Corporation (SAIC), were present to observe and record data on the operation of the technology and to perform sampling and analytical work. Quality assurance/quality control (QA/QC) audit teams from S-Cubed, an EPA Risk Reduction Engineering Laboratory (RREL) contractor, validated the test protocols in both the on-site tasks and in the main laboratory.

## CONCLUSIONS AND RESULTS

Presented below is a summary of the conclusions and results relating to the defined objectives of the test program.

- The Toxicity Characteristic Leaching Procedure (TCLP) was performed on both the feed soil and the treated slag. With regard to the metals, the feed soil only exhibited significant leachability characteristics for calcium (with an estimated mean leachate concentration of 175 mg/L and a 95% confidence limit of 162 to 188 mg/L ) and the spiked zinc (with an estimated mean of 982 mg/L and a 95% confidence limit of 948 to 1,017 mg/L). Sodium was also present in the leachate at 1,475 mg/L (95% confidence interval of 1,100 to 1,850 mg/L), but was not selected as a tracer compound since it is a weakly dissociable metal and, therefore, behaves differently from typical metals regulated by TCLP tests. The treated soil does not show strong leachability for any metals except sodium which leached at approximately the same level as in the feed soil. Both tracer metals, calcium and zinc, showed significant reductions in leaching properties as a result of treatment.
- The only organic compounds that were found to be leachable from the feed soil were 2-methylnaphthalene and naphthalene. Although the feed was spiked with high levels of hexachlorobenzene (1,000 ppm), it did not leach from the feed soil. No organic compounds were found to leach from the treated slag.
- The DRE is based on the concentration of the target analyte in the feed soil and the amount captured in the stack gas. For the Demonstration Tests, the 95% confidence interval for the estimated mean of the hexachlorobenzene spiked into the feed soil was 864 to 1,080 ppm. Hexachlorobenzene was not detected in the stack gas during any of the three tests. Therefore, all DREs determined were based on the detection limit from each of the tests. For the

Principal Organic Hazardous Constituent (POHC), hexachlorobenzene, the average DRE values ranged from >99.9968 to >99.9999% for all the Demonstration Tests.

- 2-Methylnaphthalene was found in the feed soil with an estimated mean concentration between 390 ppm to 526 ppm. Again, none of this compound was detected in the stack gas, so the DRE range for all three Demonstration Tests, based on detection limit from each of the tests was >99.9939 to >99.9996.
- A group of volatile compounds, total xylenes, was found in the feed soil at an estimated mean concentration between 128 and 139 ppm. Over the course of all the tests, this led to an average DRE range of >99.9929 to >99.9934%. Because the collection volume of gas analyzed for volatile compounds remained constant for each test, DREs calculated for xylenes are reported as an average for the entire demonstration.
- The components of the PCF can be broken down into two main categories: the thermal treatment section and the exhaust gas treatment system. The furnace unit demonstrated that it was entirely capable of processing the waste feed, however, the gas treatment system did not perform up to expectations. The air pollution control device allowed an average of 0.374 grains/dscf of particulates to be emitted to the atmosphere throughout the three tests. This exceeded the RCRA regulatory limit of 0.08 grains/dscf. Only very small amounts of particulate matter, organic compounds, or inorganic compounds were found in the scrubber sump at the conclusion of the tests, indicating poor gas treatment efficiency.



- HCl emissions were very low for all three tests, ranging from 0.007 to 0.0017 lbs/hr. Because of the low chlorine input, the regulatory requirement of less than 4 lbs/hr was met.
- NO<sub>x</sub> emissions were high, averaging approximately 5,000 ppm (uncorrected to 7% oxygen) during the Demonstration Tests. Because of the low flowrates of the stack gas, the emission rates averaged approximately 2.5 lbs/hr.
- Small quantities of volatile and semivolatile organic compounds were formed as products of incomplete combustion (PICs) in the plasma furnace. The volatile compound found most abundantly in the stack gas was benzene at approximately 19 ppbv. Benzene and substituted benzenes are prevalent in many forms throughout the feed diesel oil and hence benzene is a readily formed PIC. The most dominant semivolatile organic compound released in the stack gas was benzoic acid.
- The entire system is a high maintenance item. During the course of the Demonstration Tests, the exhaust gas blower failed twice (because of the high particulate loading in the flue gas), the torch developed a deionized water leak, and numerous preventive maintenance activities took place. The on-line factor for the process during the Demonstration Tests was 70%, but a more realistic on-line factor could be considered to be approximately 60%.
- The configuration of the furnace for the Demonstration Tests allowed the treatment of soils contaminated with heavy metals and hazardous organic compounds with no free liquid in the soil. Based on observations during the Demonstration Tests, it is anticipated that, with a different feeder, the Retech process could treat a wide variety of organic and inorganic wastes in either a solid or liquid matrix. However, judicious selection of an effective air pollution

control system downstream of the furnace is necessary before remediation can proceed.

- Successful operation of the PCF is limited by several logistical considerations. The furnace used during the Demonstration Tests must be erected in a climate-controlled environment with access to a 3-phase power supply of at least 480 volts and 1,600 amps. Plant cooling water with adequate heat rejection (cooling tower) is necessary to supply all cooling circuits on the furnace.
- Several cost scenarios can vary the unit cost of operation for the furnace. The cost of operation is strongly dependent on two factors: the on-line factor and the feed rate. The present configuration of the feeder, furnace, and slag collector allows an average feed rate of 120 lbs/hr. However, feed rates of 500 and 1,000 lbs/hr could be achieved with a few minor furnace modifications. For a feed rate of 500 lbs/hr with an on-line factor of 70% is considered then it is estimated that the cost per ton for this technology is \$1,816/ton of contaminated waste. For a feed rate of 2,200 lbs/hr with the same on-line factor the cost would be \$757/ton.

## **SECTION 2**

### **INTRODUCTION**

#### **SITE PROGRAM OBJECTIVES**

In response to the Superfund Amendments and Reauthorization Act (SARA) of 1986, the U.S. Environmental Protection Agency (USEPA) established a formal program called the Superfund Innovative Technology Evaluation (SITE) Program. The SITE Program was established to accelerate the development, demonstration, and implementation of innovative technologies at hazardous waste sites across the country. There are four parts to the SITE Program:

1. To identify and, where possible, remove impediments to the development and commercial use of alternative technologies.
2. To conduct a demonstration program of the more promising innovative technologies to establish reliable performance and cost information for site characterization and cleanup decision-making.
3. To develop procedures and policies that encourage selection of available alternative treatment remedies at Superfund sites.
4. To structure a development program that nurtures emerging technologies.

The objective of the first part of the program is to identify and evaluate these impediments and remove them or design methods to promote expanded use of alternative technologies. The demonstration portion of the SITE Program is a significant ongoing effort involving the Office of Research and Development (ORD), the Office of Solid Waste and Emergency Response (OSWER), USEPA Regions, and the private sector. The demonstrations will provide Superfund decision-makers with the information necessary to evaluate the use of these technologies in future cleanup actions. The third part of the SITE Program focuses on establishing methods for selecting treatment technologies for Superfund sites from the expanding range of available remedies, including these innovative technologies. Finally, the SITE Program provides a means of assisting in the development of emerging technologies towards a mutual goal.

## **PROJECT BACKGROUND**

A demonstration of the Retech Plasma Centrifugal Furnace (PCF) technology has been performed under the SITE Program. This system used an innovative thermal technology to treat soils and debris contaminated with hazardous organic chemicals, inorganic chemicals, and heavy metals. The process claims to vitrify metal-bearing solids and inorganic material into a monolithic nonleachable phase, and thermally destroy organic chemicals.

The study was conducted at the DOE's CDIF located in Butte, Montana, operated for the DOE by MSE, Inc. The test material used was a blend of soil [mine tailings obtained from the nearby Silver Bow Creek (SBC) Superfund Site] and 10% by weight No. 2 diesel oil. The mixture was spiked with high concentrations of zinc oxide and hexachlorobenzene. The combination of the soil and oil provided a test material which was high in concentrations of both heavy metals and organic material. The zinc oxide

and the hexachlorobenzene were spiked to ensure traceable compounds throughout the testing period.

This technology is significantly different from conventional incineration technologies in that the temperatures in the treatment zone are much higher and the gas flows are much lower than those typically encountered. Additionally, the treated soil is vitrified into a glass-like mass. The tests obtained process data on the system performance and the fate of metals and organics in the system. Removal or stabilization of inorganic and metal contaminants was assessed by pre- and post-test sampling and analysis. The DRE of this thermal technology was also determined by pre- and post-test sampling and analysis for organic materials. The results of this testing are presented in this report.

## **SECTION 3**

### **PROCESS DESCRIPTION**

#### **TEST SOIL**

Soil collected from the Silver Bow Creek (SBC) Superfund Site in Butte, Montana was placed in 11 32-gallon plastic containers with lids. The soil from the SBC site is categorized as a heavy metal-bearing soil and was previously characterized as part of the Streambank Tailings and Revegetation Study (STARS) conducted by the EPA in 1988 [1]. Zinc oxide and hexachlorobenzene were spiked into the dry SBC soil. Spiked SBC soil and 10% by weight No. 2 diesel oil were combined together in a cement mixer to provide a mixture containing 28,000 ppm zinc oxide and 1,000 ppm hexachlorobenzene. This mixture was homogenized by the rotating action of the mixer. To permit ease of handling and loading into the Retech Plasma Centrifugal Furnace, the blended mixture was poured from the mixer into 5-gallon metal containers and sealed for transport and storage.

Based on previous analyses, the leachability of the metals in the SBC soil was very low overall. Therefore, it was determined to spike the feed soil with a nonhazardous metal, zinc, to ensure an initial metal concentration in the feed high enough to allow an adequate evaluation of the effectiveness of treatment by the PCF in vitrifying inorganic constituents into a nonleachable mass.

Diesel oil was mixed with the feed soil to show that the process could treat wastes contaminated with high levels of organics. Ten percent (10%) was the maximum level of liquid combustibles that could be fed to the furnace equipped with the type of feeder present at the test site; levels of diesel oil greater than 10% ignite in the feeder because of the heat from the process. It is possible to treat contaminated wastes with a higher percentage of organics using a different feeder configuration assuming that the gas treatment system is sized correctly. Although the composition of the diesel oil was estimated prior to the Demonstration Tests, the soil/diesel oil feed mixture was left in the containers for an extended period of time after mixing activities and prior to the analysis conducted as part of the Demonstration Tests. Therefore, at the time of the tests, it was not certain that the concentrations of organic components in the diesel oil were suitable to evaluate the ability of the furnace to treat wastes contaminated with up to 10% organics. Hexachlorobenzene was spiked into the feed to ensure a traceable organic compound for evaluating DRE.

## **TEST LOCATION**

The Demonstration Tests were conducted using actual hazardous waste at the DOE CDIF in Butte, Montana. The use of this facility for the Demonstration Tests was the result of an interagency agreement between the EPA and the DOE. The CDIF is an engineering-scale development test facility, operated for the DOE by MSE, Inc. The CDIF is a major DOE test facility and part of the Idaho National Engineering Laboratory (INEL).

Retech's equipment was erected near the southwest corner of a 100-foot by 95-foot Component Test Building, which has a ten-ton bridge crane and a roll-up access door 12 feet wide by 20 feet high. The building is supplied with 480-volt power and is served by a recirculating cooling tower water system with heat rejection by a cooling tower. Macadam roads provide good access to the building.

## DETAILED PROCESS DESCRIPTION

The Retech Plasma Centrifugal Furnace is a remedial action process for soils contaminated with hazardous chemical wastes and/or heavy metals. The PCF is a thermal treatment process designed to convert contaminated soil into a chemically inert and stable glass and crystalline product. Figure 1 is a schematic of the pilot-scale unit utilized in the SITE Demonstration Tests. This unit is designated as a PCF-6, according to the six-foot diameter of its primary chamber.

The entire system is comprised of a thermal treatment system and an exhaust gas treatment system. The thermal treatment system consists of the feeder, the primary chamber, the torch, the afterburner, the secondary chamber, and the collection chamber. The exhaust gas treatment system consists of a quench tank, a jet scrubber, a packed-bed scrubber, a demister, and a stack blower.

### The Feeder

Hazardous waste is initially loaded manually into a screw feeder from sealed 5-gallon containers. The feeder unit is 3.5 feet high  $\times$  2.5 feet wide  $\times$  10 feet long and can hold approximately 120 pounds of feed. The outer stationary cylinder of the feeder is sealed at both ends. The fill end has a hinged door that opens to allow the inner cylinder to be loaded. The inner cylinder has welded internal screw flights that push the dirt charge toward the discharge chute as it is rotated by a hydraulic drive. After the feeder is loaded, it is sealed, and the waste is fed semi-continuously into the furnace through the discharge chute. An elbow in the discharge chute limits the size of waste particles fed into the furnace to be less than four inches in diameter. The operator controls the rotation of the feeder.



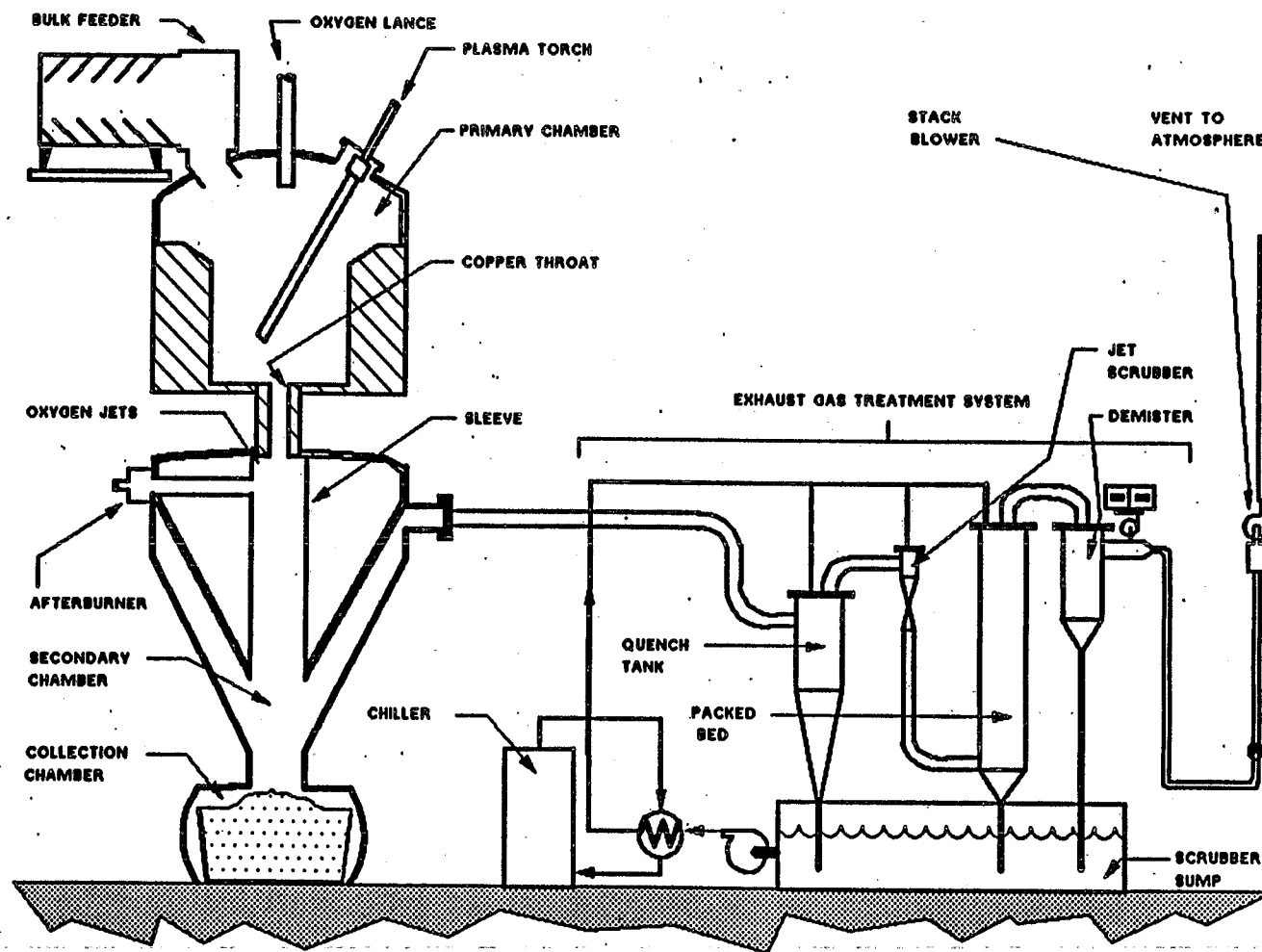


Figure 1. Schematic of Plasma Centrifugal Furnace System

When the feeder is empty, it is purged of potentially toxic gases to the primary chamber with an air purge. The feeder isolation valve is then closed, and the feeder is refilled. During the Demonstration Tests, the system operated in the semi-batch fashion, described here, at a rate of approximately 120 pounds per hour. Other feeders (not evaluated during these Demonstration Tests) that allow an increase in the feed rate and offer no limitation on the organic content of the waste feed can be used by the furnace.

In order to access to the inside of the primary chamber for maintenance and repair services, the feeder must be moved away from the primary chamber before opening the lid; it must be repositioned before the torch may be lit. During the Demonstration Tests, the ten-ton bridge crane in the CDIF was used to move the feeder.

### The Primary Chamber

The waste material drops from the feeder into the primary chamber, which is a rotating tub with a central orifice at the bottom. Solid material is retained in the primary chamber by centrifugal force. The primary chamber walls have an inner shell and water jacket welded between. Fifteen gallons per minute of cooling water at 30 psig circulate between the shell and the jacket. At the copper throat, an area of high heat flux, the cooling water flow area is reduced to increase the velocity of the cooling water to 40 ft/sec, increasing the cooling in this area.

Ports are located in the head of the lid for: the plasma torch, an off-axis feeder port, an oxygen lance, and four view ports. The entire lid subassembly is attached to a hinged structure that allows the lid to be hydraulically tilted through a 45° angle. A mechanical safety link provides a backup to the lid lift hydraulic cylinder locks.

The cavity below the primary chamber is maintained slightly positive with respect to furnace pressure so that any leakage at the furnace lip seal at the top of the spinning furnace wall will be in toward the hot furnace. This leakage will cool the furnace lip seal and help prevent contamination of the drive cavity below.

The primary chamber can hold approximately 1,800 pounds of contaminated waste; however, the size of the collection chamber limits the amount of waste that can be in the primary chamber to 1,000 pounds. The primary chamber has a diameter of 6 feet and a height of approximately 3.5 feet.

### The Plasma Torch

The Retech PCF makes use of a transferred plasma torch. The plasma torch uses electrical discharges to add energy to plasma torch gases in order to increase the gas temperature beyond that normally attainable by chemical reaction. The plasma torch produces a transferred arc that directly contacts a conducting portion of the primary chamber (either the copper throat or steel "doughnuts") located at the bottom of the spinning primary chamber. The heat generated by the plasma torch brings the waste material to temperatures sufficient to melt soil. The melting point for typical soil is on the order of 3,000°F. The waste is melted by this extreme heat, incorporating any inorganic and metal components into a stable material. Organic components are volatilized by the heat of the plasma and oxidized by the air used as the plasma gas. Oxygen may also be added from an oxygen lance in the primary chamber to enhance combustion of organics.

The plasma torch used in the PCF-6 is Retech's Model RP-250T plasma torch, developed for melting superalloys in the metallurgical industry. For this application, the torch, rated at 500 kW, used approximately 20 scfm of air as plasma gas. The torch runs on direct current (DC) provided by a power supply that uses 3-phase input.

Figure 2 shows a simplified presentation of the transferred plasma torch. Air is injected tangentially at an intermediate axial position inside the torch tube. One of the DC electric arc termination points is higher up inside the tube on an electrode. The arc travels out the end of the tube and terminates on the rotating copper throat (or steel doughnuts) below. The electrode and nozzle are cooled by a high velocity flow of distilled water. The torch is mounted on a spherical ball swivel joint that has x-, y-, and z-axis hydraulic positioning capability. The torch subassemblies are mounted on the primary chamber lid.

### The Afterburner

The gases evolved from the melting of the feed are drawn through the copper throat of the primary chamber and pass through a natural gas afterburner located in the secondary chamber, just downstream of the copper throat. The afterburner is utilized to combust any products of incomplete combustion (PICs) by providing additional heat input (beyond that supplied by the plasma torch) through another ignition source. It is sized to provide 200,000 Btu/hr and operates on a natural gas flame. The organics that are volatilized and oxidized are then drawn off to the exhaust gas treatment system.

### Secondary Chamber

A camera port in the secondary chamber allows observation of the gases and slag exiting the throat. If needed, oxygen may be added from oxygen jets located in the secondary chamber to enhance combustion of organics. A sleeve extending from the copper throat of the primary chamber, past the afterburner, and down to the end of the baffles in the secondary chamber, reduces the volume of gas required to be

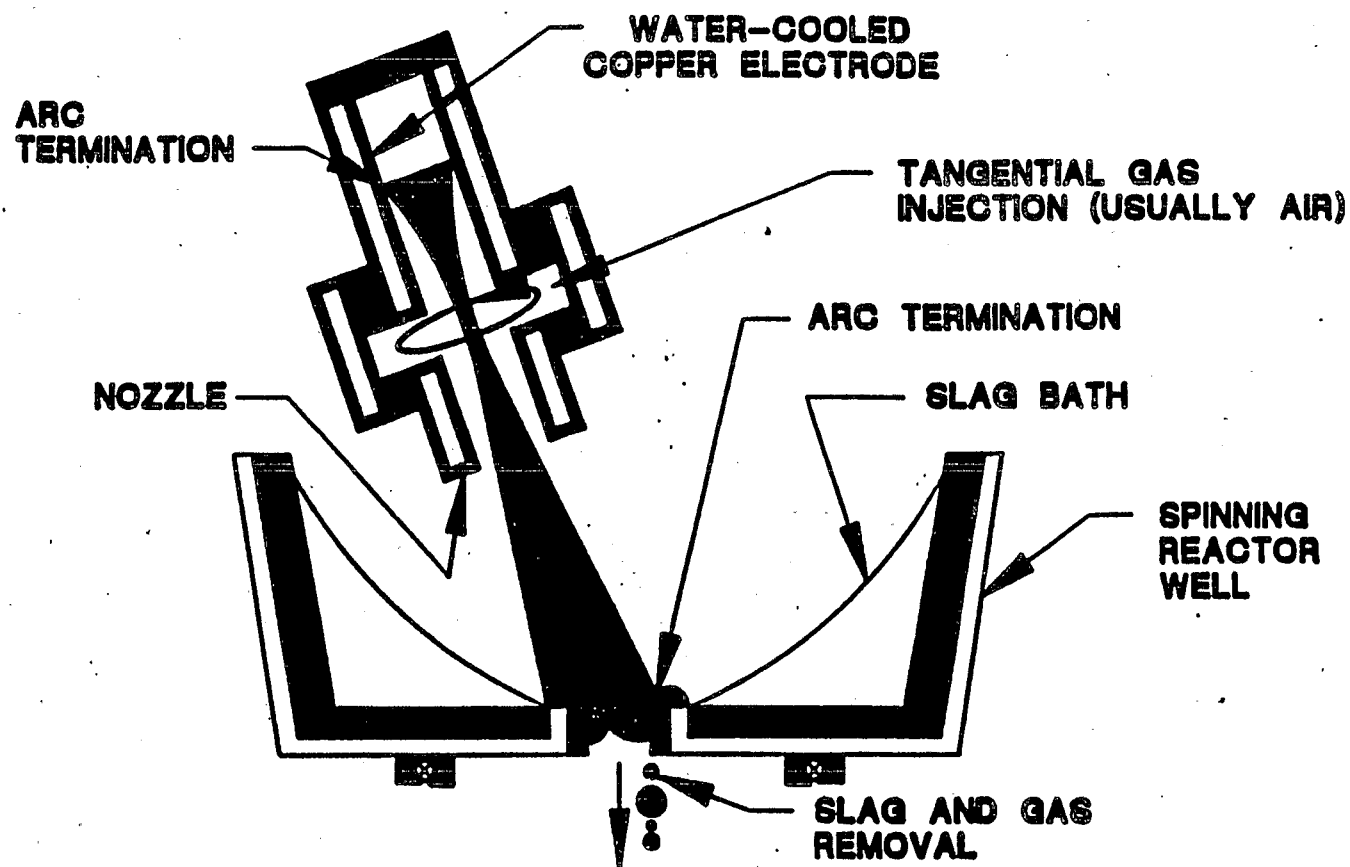


Figure 2. Retech's Centrifugal Furnace with Transferred Plasma Arc

heated in the secondary chamber by the afterburner, thus ensuring enough heat is available to complete the combustion of volatilized organics.

The secondary chamber walls have three inches of refractory lining to abate heat loss and protect the steel walls. These walls also form a jacketed vessel with 25 gpm cooling water circulating between them to maintain a safe operating temperature.

### Collection Chamber

The molten mass falls from the secondary chamber into a heavy pig mold located in the collection chamber. The collection chamber is a water-cooled, jacketed chamber that is bolted to the bottom of the secondary chamber. It houses a pig mold that can hold approximately 1,000 pounds of melted solids.

The collection chamber is cylindrically shaped on a horizontal axis. One end is closed off with a bolt-on, water-cooled blind flange. The other end has a water-cooled, hinged door equipped with a viewport. Pig molds may be loaded and unloaded through this door. Ten gpm of plant water at 30 psig cool this chamber subassembly.

The pig mold is made of 3/4-inch thick steel plate so it can withstand the sustained heat load of the cooling slag and the shock of the cascading charge. The mold has a rectangular plan view and both elevation views reveal a draft angle to aid in removal of the casting from the pig mold. The pig mold is on a removable tray, somewhat simplifying chamber decontamination.

## Exhaust Gas Treatment System

Figure 2 shows the exhaust gas treatment system designed for the Demonstration Tests. The gas stream is initially cooled in a quench tank before going to a jet scrubber that is designed to remove particulates. From the jet scrubber, the gas passes to a packed-bed scrubber to remove additional acid gases that may be present. A demister then removes moisture droplets entrained in the flow.

A mildly caustic solution is used in the quench tank, jet scrubber, and packed-bed to remove acid gases as well as particulates. The water passing through the exhaust gas treatment components exits from the bottom of each unit, back into the scrubber sump. The caustic reservoir that supplies the solution to the gas treatment units is maintained at a pH of 8.5. This is achieved by means of a 0.1 gph positive displacement pump that feeds the reservoir with sodium hydroxide, based on a signal from a pH sensor located at the discharge port of the scrubber pumps.

The scrubber sump is equipped with a 50-ton chiller to cool the scrubber water circulating through the exhaust gas treatment equipment, so that all the moisture can be removed from the exhaust gases. The chiller utilizes an external heat exchange system. The chiller coolant is previously cooled by means of an additional internal heat exchanger located within the chiller. The scrubber water is continuously cooled and recirculated through the exhaust gas treatment system.

Flowmeters are installed to ensure that correct flow rates of caustic solution are being applied to each of the gas treatment units. Flowrates are adjusted by means of metering valves at the inlet to each of the treatment units.

During normal operation, there is a negative pressure on the furnace due to the effect of the stack blower. This assures that, if there is a leak in the system, any leak would

be drawn into the furnace and not out into the operator area. All connections are water-cooled and O-ring sealed. The system is leak-checked before each test.

### Water Cooling Systems

All parts of the system exposed to high temperature are water cooled. There were three cooling water systems involved in the furnace components for the Demonstration Tests: a closed-circuit deionized water (D.I.) system; a closed-circuit water with rust inhibitor system; and a plant cooling water system.

The closed D.I. system cools the plasma torch, the ram, the electrode and the nozzle. The D.I. system pump delivers a total of 60 gpm at 80 psig. A heat exchanger transfers the heat gained by the above circuits to the plant cooling water system.

The closed water with rust inhibitor system has just one circuit--the primary chamber cooling circuit, flowing at 100 gpm and 50 psig (at the pump discharge). A heat exchanger is sized to pass a maximum of 300,000 BTU/hr from the furnace cooling circuit to the plant water system.

The plant cooling water system must not only cool the two cooling systems described above (60 gpm and 100 gpm, respectively), but must also cool the furnace lid, primary chamber, drive lid, secondary chamber, collection chamber, chiller, and hydraulic unit.



## The Control System

The Retech control system is designed to be fail-safe. Numerous interlocks and alarm circuits have been incorporated to reduce the risk of release of toxic material or equipment damage due to operator error or process failure.

The torch cannot be lit unless all of the console interlocks are satisfied. These interlocks include the torch gas pressure, the cooling water systems, the hydraulic system, the exhaust gas treatment system, the stack blower, the afterburner, and the chiller. The furnace is interlocked to shutdown if any of these systems or equipment fail. If the emergency stop is activated, only necessary support systems are left operating.

The plasma torch gas controls include a regulator, solenoid actuated on/off valves, gas rotameters, needle valves, and check valves. A pressure gauge and pressure switch are also mounted in the line on the way to the torch. Before torch start-up, the pressure regulator is set and the gas flow rate established. The operator controls the on/off valve from the control console. Power to the torch is automatically cut if the gas pressure falls below a predetermined level.

Power to the torch is controlled from the control console. Current can be varied by the operator with a twist of a dial. Cooling water to the torch is turned on manually at a supply manifold. Switches in the supply lines provide an interlock to cut power to the torch either at start-up or during operation if the water flows fall below preset minimums. A starter unit, used to initiate an arc inside the torch at start-up, is energized by the operator at the control console and de-energized by a timer in the controller. Torch position is controlled by the operator at the control console with a joy-stick. Position sensor outputs are used to limit deflection to safe locations.

The centrifugal speed is monitored and controlled at the console. A magnetic pickup uses the drive gear to generate the signal. Flow switches in the furnace cooling water return manifolds prevent the torch from operating if the furnace cooling system fails. The lid cannot be lowered hydraulically without a safety link being disconnected, thus protecting the equipment.

The output from the continuous emissions monitors are displayed at the console. An interlock is included to assure that the stack blower is turned on before the furnace is started. A pressure differential switch exists across the blower which shuts the system down in the event of blower failure.

Level controllers are equipped on the caustic makeup tank and reservoir for the water pump. If the level switches, in either case, are tripped on the low condition, an alarm lights an indicator on the control console.

To ensure complete combustion, the afterburner is interlocked to the feeder. If the afterburner is lost, the feeder is automatically stopped. The chiller is interlocked to the torch, the afterburner, and the feeder.

## **SECTION 4**

### **FIELD OPERATIONS DOCUMENTATION**

This section provides an overview of the field operations including a summary of the tests, a detailed description of typical operations, and an operational log of the Demonstration Test activities.

#### **TEST SUMMARY**

Demonstration testing of the Retech Plasma Centrifugal Furnace took place the week of July 22, 1991. The goal of this demonstration was to determine if the unit could effectively thermally treat soil contaminated with metals and up to 10% organic material and create a nonleachable matrix. The testing objectives are defined in Section 1 of this report and more specifically in the "Demonstration Plan for the Plasma Centrifugal Furnace Technology" [2].

For the demonstration, three test runs were performed using Silver Bow Creek mine tailings mixed with 10% by weight No. 2 diesel oil, and spiked to provide 28,000 ppm zinc oxide and 1,000 ppm hexachlorobenzene in the soil/oil mixture. The Demonstration Tests took place as scheduled on July 22, 24, and 26, 1991. It was anticipated that 960 pounds of the spiked soil would be treated for each test (2,800

pounds total). Instead, 1,440 pounds of the spiked soil were fed into the furnace and 1,137 pounds of treated material were generated during the three Demonstration Tests. Additional details regarding this discrepancy are provided later in this section and in Section 6, "PERFORMANCE AND DATA EVALUATION." For each Demonstration Test, samples of the feed soil, the treated soil, the scrubber liquor, and the stack emissions were collected to evaluate the performance of the technology. Samples were collected and analyzed in accordance with the Demonstration Test Plan with only minor changes in some of the sampling and analytical methods. These changes are detailed in Section 8.

The unit was installed into three levels of the MSE Component Test Building. The uppermost level of the system was located at ground level. This level housed the primary chamber and the feed screw, which extended to an elevation of approximately 17 feet. The feed screw was located on top of the unit and a platform with stairs allowed easy access to facilitate feeder loading. Twelve feet directly below, on the second level, was the secondary chamber, which received the soil and combustion gases for supplementary treatment with the assistance of an afterburner. The lower level (13 feet below the second level) contained the air pollution control system, designed to ensure that the treatment off-gas emitted to the atmosphere remained within permissible levels. The treated gas was directed back up to ground level, outside the building. Here, the exhaust gas ductwork was constructed to facilitate gas sampling. A vacuum blower drew the gas through the air pollution control system and maintained negative pressure in the furnace. After passing through the sampling configuration, exhaust gas exited at the top of the building through the stack. The entire process was controlled on the first level at a central control panel. Here the torch operator controlled the feed screw and the position of the torch. To assist in torch control, cameras, which gave a visual indication of the torch position, were installed in the primary chamber. All process parameters were monitored by an automated data acquisition system (DAS) which collected information every 30

seconds. Auxiliary equipment was monitored by MSE personnel performing visual inspections and recording data every 30 minutes.

Prior to the Demonstration Tests, MSE performed treatment tests using uncontaminated soil to familiarize themselves with equipment operation. During these shakedown tests, modifications were made to the original system design. These modifications included the installation of the afterburner in the throat of the furnace between the primary and secondary chambers, the installation of a chiller on the gas treatment system to compensate for this additional heat input, and the elimination of the surge tank. The detailed discussion of the process description is included in Section 3 of this report.

## DESCRIPTION OF OPERATIONS

Typically, operational procedures are conducted under standard process conditions. These operating conditions, coinciding with those used during the Demonstration Tests, are summarized in Table 1. To facilitate the treatment of different types of waste, the system has the potential for alternate conditions or configurations for parameters such as the feed rate, the feed material, and the air pollution control system; generally, the conditions remain the same for all operations. The operational procedures for the present configuration are described below.

As part of preparation for operation, the non-current-carrying end of the electrode is coated with Dow Corning vacuum grease and the current-carrying electrode thread is treated with an application of Copper Cote®. These materials are utilized as an aid to light-off procedures. Before initiating operations, the lid is opened to inspect the torch and the gas lines leading into the furnace. A general inspection of the entire system also takes place prior to light-off. Approximately 100 pounds of mild steel, in the form of 1-inch thick "doughnuts," are placed inside the chamber, encircling upper end

Table 1. Demonstration Test Operating Conditions

Parameter	Operating Range	Units
Feed Composition	Variable. Limited by 10% organics or no free liquid (for this feeder.)	—
Feed Rate	100 - 120	lbs/hr
Electrode Life	≈ 50	hr
Mild Steel "Doughnut" Mass	50 - 100	lbs
Torch Gas (Air) Flowrate	≈ 25	scfm
Torch Gas (Air) Temperature	> 15,000	°F
Oxygen Lance Flowrate	25 (during feeding)	scfm
Auxiliary Torch Gas (e.g., Argon, Helium) Flowrate	≈ 5	scfm
Afterburner Gas (Natural Gas) Flowrate	3 - 4	scfm
Furnace Well Rotation Rate During Treatment	50 - 70	rpm
Furnace Well Rotation Rate During Pour	10 - 30	rpm
Primary Chamber Temperature	> 2,100	°F
Afterburner Temperature	> 1,800	°F
Scrubber Liquor Generated	30 - 50	gal/hr treatment
Off-Gas Flowrate	≈ 110	scfm
Slag Mass	≤ 600	lbs/pour

of the throat. This provides a conductive surface on which the torch arc can be initiated and a lip that prevents untreated material from spilling into the secondary chamber. Typically, a 4- to 8-inch layer of previously treated material coats the inside of the primary chamber.

After a final check of process equipment, the torch arc is struck. The furnace chamber temperature initially rises rather rapidly, and off-gas temperatures climb steadily. Before any material can be fed into the furnace, operating procedures require that the primary chamber temperature must be at least 2,100°F, and the afterburner temperature 1,800°F. Three to five hours of operating time are necessary to achieve these temperatures, depending on the amount of residual heat in the primary chamber.

Feed material is generally placed into 5-gallon pails to facilitate ease and convenience during feeding. The pails are placed on the feeder platform prior to startup.

When the furnace is at or near operating conditions, the feed screw is manually loaded. Feeding is performed by opening the access door to the feed screw and placing soil into the screw chamber. A specially-designed bucket holder facilitates dumping the contents of the bucket and allows the material to pass down a shoot into the feeder. As material enters the feeder, the screw is turned by hand to evenly distribute the feed load along the screw. After an appropriate amount of feed (approximately 120 pounds) has been loaded into the feeder, the access door to the feeder is secured and purged with air of any residual contamination. The purged air is bled to the primary chamber of the furnace. This eliminates potential for worker exposure to toxic fumes when loading of the feeder is resumed. This feeding process has potential for automation, but is presently manually operated.

The feed screw is placed in motion, allowing material to enter the furnace. It takes approximately one hour to completely feed the 120 pounds of material in the screw to the furnace. When empty, the feeder is reloaded and additional material is fed to the furnace as described above until a maximum of 1,000 pounds has been fed. This corresponds to the capacity of the pig molds into which the molten soil is poured.

Process operations are continuously observed by operators and their supervisors. Process measurements shown in Table 2 are recorded manually or electronically by the DAS. Important process information is also calculated by this system. These calculated values are presented in Table 3.

Once fed into the furnace, treatment of the waste material is initiated with the plasma torch. The area closest to the copper throat and the mild steel doughnuts is the first location to be preheated. After the melted material in this vicinity is heated to conducting temperature, the torch is moved slowly to heat more of the bottom of the

**Table 2. Operating Parameters Monitored by the DAS**

<b>Temperatures</b>	<b>Pressures</b>
Argon Supply	Argon Supply
Oxygen Supply	Oxygen Supply
Service Air Supply	Service Air Supply
Furnace Off-Gas	Stack Gas Flowmeter Differential
Scrubber Outlet Gas	Stack Gas
Stack Gas	Drive Chamber
Primary Chamber Gas	<b>Flowrates</b>
Afterburner Gas	Argon Supply
Secondary Chamber Gas	Oxygen Supply
Melt	Service Air Supply
Stack Gas Composition	Afterburner Air
High CO	Afterburner Gas
Middle CO	<b>Miscellaneous</b>
Low CO	Torch Current
O <sub>2</sub>	Torch Voltage
SO <sub>2</sub>	Feeder Position
NO	Primary Chamber Rotation Speed
NO <sub>x</sub>	Switched Input Sensing
THC	

primary chamber and eventually the sidewall. This is continued until the entire contents of the primary chamber have been melted by the torch. Following this preheat period, the screw feeder is rotated to charge material at a uniform rate into the furnace. The feeder can be recharged and feeding repeated as previously described. After all the desired charge is melted, the natural gas afterburner (located downstream of the primary chamber, in the secondary chamber) is extinguished, and the furnace spin rate is slowed to approximately 25 rpm allow the pool to move inward and the melted soil to pour out of the bottom of the reactor, into the secondary chamber. The molted mass falls from the secondary chamber, through the



**Table 3. Operating Parameters Calculated by the DAS**

Heat Losses	
Scrubber Cooling	Torch Cooling
Centrifuge Cooling	Demister Cooling
Torch Ball Cooling	Primary Lid Cooling
Drive Chamber Cooling	Secondary Combustion Chamber Cooling
X-Y Plate Cooling	Side Ports Cooling
Total	
Flowrates	
Argon Supply Mass	Oxygen Supply Mass
Service Air Supply Mass	Stack Gas Volume
Stack Gas Mass	Total Mass In - Total Mass Out
Power	
Torch	

collection chamber, and into a heavy pig mold. The pouring process takes five to ten minutes for a 600-pound pig. The molten mass solidifies into a hard monolith in approximately 12 hours and can be disposed of in an appropriate landfill or otherwise utilized. The organics that are volatilized and oxidized are drawn off to the exhaust gas treatment system for subsequent conditioning prior to discharge to the atmosphere.

Routinely, the scrubber sump is drained, cleaned and charged with fresh water. Recharging activities for the scrubber sump are also performed whenever the waste feed is altered. New filters are installed into the scrubber recirculation lines as required, depending on particulate accumulation.

## OPERATIONAL LOG FOR THE DEMONSTRATION TESTS

Presented below are the field notes for the Retech Demonstration Tests. The notes briefly and chronologically summarize the operational events that took place during the tests.

### Test 1

The Retech Demonstration Tests began on Monday, July 22, 1991. Activities for Test 1 began at approximately 7:00 a.m. At 7:10 a.m., the pig mold, which would hold the treated material, was vacuumed out to remove any residual soil. A new torch electrode had been installed the previous evening and was coated with Dow Corning vacuum grease and Copper Cote®. The primary chamber was prepared for closure by coating the sealing surface with a new gasket. By 7:35 a.m., the furnace lid was fastened in place. At 7:40 a.m., the feed screw was placed on the furnace and secured into place. A process problem with a thermocouple and a plugged stack flowrate annubar were noted at 8:35 a.m.

After the process checkout had been completed at 9:05 a.m., the torch arc was struck, and the furnace warm-up was initiated. More than an hour and a half later, stack gas became visible from the stack. The gas appeared brown in color with a yellowish tint. Continuous emission monitoring equipment indicated that the stack gas exhibited high NO<sub>x</sub> values in the range of 7,000 to 10,000 ppm.

At 12:20 p.m., the open feed buckets were monitored with the organic vapor analyzer (OVA) field instrument to estimate the amount of volatile organics being emitted to the air around the feeder. The OVA did not detect any volatile organics in the air in the vicinity of the feed. Loading of the feed screw began at 12:25 p.m. By 12:35 p.m., the feeder loading was complete with 120 pounds of contaminated soil.

Because of Demonstration Test sampling activities, the time required to load the feeder was slightly longer than that required for typical operations.

By 1:10 p.m., the action of the screw feeder to the furnace was initiated for the first feed load. At this time, the primary reaction chamber temperature was 2,156°F, while the afterburner baffle temperature was 1,855°F. At 1:50 p.m., the sump pump tripped and the system was checked to determine the cause. The 1:50 p.m. trip of the scrubber occurred because one of the scrubber sump pumps was being starved of scrubber water. This caused the pump motor to overheat and trip the pump. Three minutes later, the scrubber sump pump feed line was cleared and the furnace was again activated. Feeding could not be resumed immediately because the primary chamber temperature was too low. By 2:20 p.m., the feeding resumed, and 20 minutes later, re-loading of the feed screw was started. As soon as the loading of the feeder was complete (at 2:49 p.m.), the action of the screw feeder was again initiated. The second feed load was fed by 2:50 p.m. At 3:25 p.m., scrubber pump #2 was taken off-line. At 3:30 p.m., the feed screw loading was started and by 3:39 p.m. feeding of third load began. By 4:15 p.m., the feed screw loading was started, and the feeding of the fourth and final began at 4:25 p.m.

At 4:37 p.m. and 4:48 p.m., the afterburner tripped and was restarted. At 5:03 p.m. the afterburner tripped again. A low pressure differential (20" H<sub>2</sub>O) across the blower was noted at this time. Normal operation requires a pressure differential of 24" H<sub>2</sub>O, thus, the operators experienced difficulties maintaining negative pressure in the primary chamber. The cause for the drop in pressure differential across the blower was particulate buildup within the blower. The resulting low negative pressure in the furnace was the cause of the afterburner failures. Operations were restarted, but feeding was not recommenced immediately due to inadequate temperatures in the afterburner. By 5:51 p.m., sampling and feeding of the final load were resumed. It was decided not to reload the feeder with any more soil as the problem with the particulates would only be aggravated. Therefore, the test was terminated.

At 6:14 p.m., the feeder was turned off, the afterburner was shutdown, and the melt was poured. By 6:36 p.m., the torch was off, all process equipment was secured, and the test was complete. Selected process parameters are summarized in Table 4.

## Test 2

Test 2 for the Retech Plasma Centrifugal Furnace system took place on July 24, 1991. At 8:20 a.m., the torch arc was struck. By 10:35 a.m., the primary chamber was at operating temperature, but the afterburner baffle temperature was too low to begin feeding. Once the baffle reached 1,800°F, the feed screw loading started. By 11:15 a.m., the feeder was loaded, and the feed personnel had exited the sampling platform. At 11:20 a.m., feeding of the first load began. Between 12:10 p.m. and 12:25 p.m., the feed screw was reloaded and the feeding of the second load commenced. At 1:10 p.m., a third feeder load was started. These activities were complete at 1:16 p.m. The feeding of the third load into the furnace started at 1:18 pm. Operators were once again having trouble maintaining negative pressure in the furnace because of the low pressure differential across the blower. At 2:10 p.m., the

Table 4. Summary of Selected Process Parameters

Parameter	Test 1	Test 2	Test 3	Total
Test Soil Fed (lbs)	480	360	600	1,440
Treated Soil Poured (lbs)	277	265	595	1,137
Scrubber Water Generated (gal)	155	≈ 150	≈ 800 *	1,105
Mild Steel Added	75	54	202 **	331

\* Prior to Test 3, the scrubber was flushed and recharged. This generated approximately 100 gallons. Additionally, the scrubber was continuously blown down during this test in an attempt to alleviate the problem of particulate buildup in the blower. The total amount of scrubber water collected during Test 3 (also the conclusion of the demonstration testing) was approximately 900 gallons.

\*\* Prior to the feeding of any test soil for Test 3, 58 pounds of mild steel were added to the furnace. When the furnace was restarted (6:33 p.m.), an additional 144 pounds of mild steel were added.

blower tripped the torch, and adequate furnace pressure could not be maintained. The torch arc was reinitiated, and the test proceeded until a pour could be completed.

At 2:27 p.m., the afterburner was shut down, and the melt was poured. By 2:37 p.m., the system was shut down, secured, and Test 2 was complete.

### Test 3

Demonstration Test 3 was conducted on July 26, 1991. The troublesome blower was replaced by a blower with a larger motor. Lighting of the torch took place at approximately 8:20 a.m. The furnace began to heat up at 8:23 a.m.

At 9:53 a.m., the furnace tripped. A leak, which caused distilled water to drip into the primary chamber, had developed in the torch cooling system. The problem was caused by a side arcing of the torch which burned a hole in the torch casing. The furnace was opened and repairs were made by grinding clean the torch tip and welding the hole shut. By 12:40 p.m., the repairs were complete. The torch was again struck, and the furnace began to heat back up.

At 3:30 p.m., the furnace was near processing temperatures, and feeder loading started. By 3:35 p.m., feeder loading was completed. The feeding of the first load to the furnace was started at 3:50 p.m. By 4:47 p.m., the loading of the feeder with a second load had begun. At 4:54 p.m., the feeder was fully loaded. This was followed immediately by the feeding of the second load to the furnace. Approximately one hour later, feeder loading of the third load was started and promptly completed. By 6:00 p.m., the feeding of the third load had begun.

At 6:07 p.m. the afterburner tripped, temperature in the top baffle dropped, and the feeder was stopped. The feeder was restarted at 6:11 p.m. At 6:24 p.m., the

afterburner tripped again, the furnace temperature dropped, and the feeder was stopped. The afterburner was relit at 6:27 p.m., and the feeder was restarted at 6:30 p.m. It was later determined that the afterburner trips had been caused by the operator of the scrubber unit. During the course of the test, the two 10 micron filters at the discharge of the sump pumps had become clogged. The operator changed out each of the filters while the furnace was still operational. This caused a pressure drop in the scrubber supply water which, in turn, tripped the afterburner.

The time was 6:59 p.m. when the feeder loading activities began for the fourth load of feed. At 7:06 p.m., when the feeder was loaded, the feeding of the fourth load started. Feeder loading began at 7:59 p.m. and was finished eight minutes later. The feeding of a fifth load immediately followed. By 9:06 p.m., the feeding of the fifth load was completed and the feeder was stopped. At this point, the torch operator prepared the melt for pouring. At 9:23 p.m., the pouring of the melt was initiated. By 9:26 p.m., the melt was poured, the system was powered down, and the test was complete.

## **SECTION 5**

### **SAMPLING AND ANALYSIS PROGRAM**

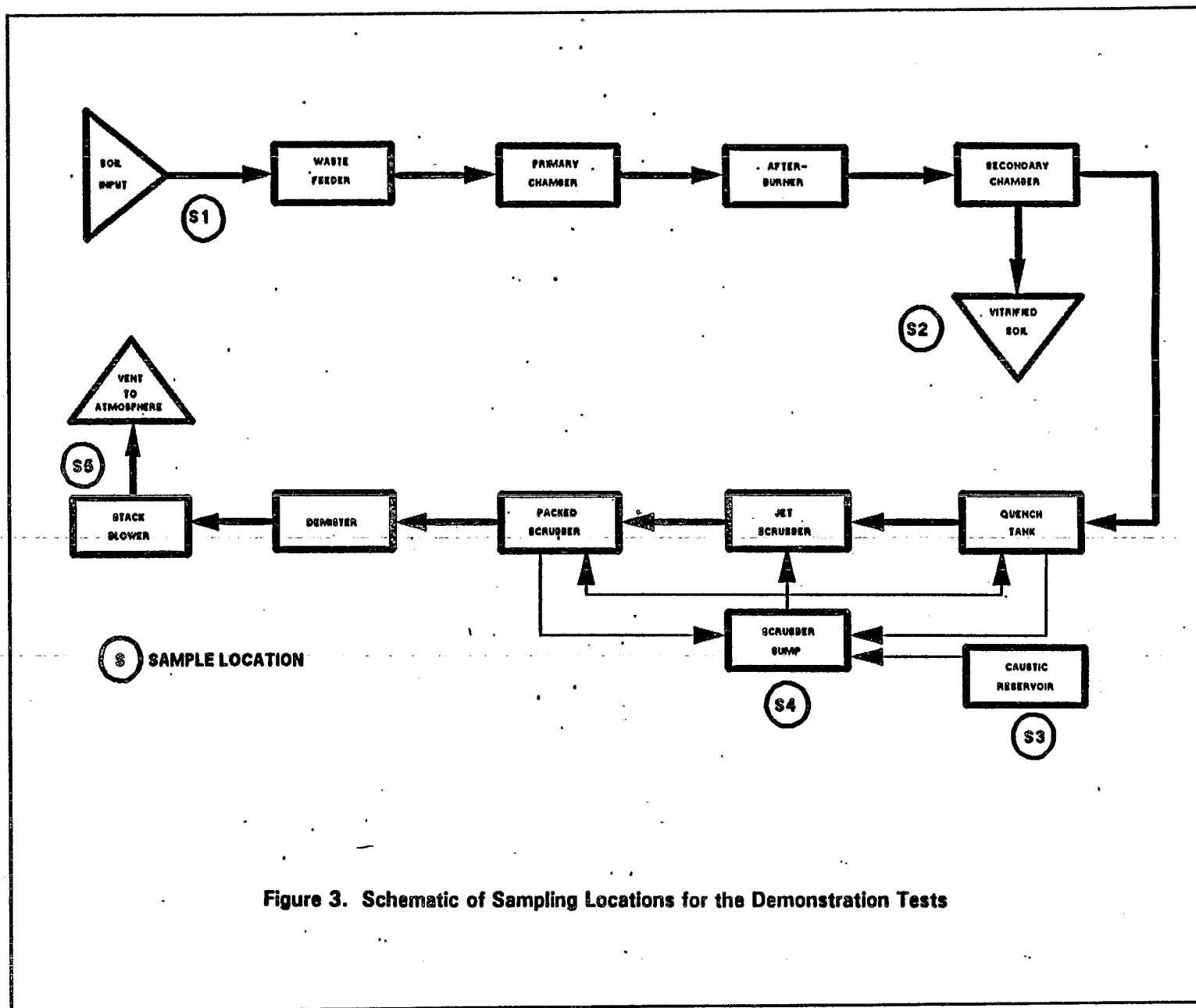
#### **SAMPLING PROTOCOL**

As part of the SITE Program, a sampling strategy was designed and employed to evaluate the performance of the Plasma Centrifugal Furnace technology developed by Retech, Inc. The sampling protocol was developed based on the critical test objectives stated in Section 1.-

Extensive monitoring of process equipment was also performed to collect data required for analytical and economic calculations necessary to properly evaluate this technology. These monitoring parameters are presented in Section 4 of this report.

Samples were collected in accordance with the "Demonstration Plan for Plasma Centrifugal Furnace Technology." Minor changes to the original sampling plan were made. These changes are discussed in detail in Section 8 of this report. Figure 3 presents a schematic of the sampling locations for the Demonstration Tests.

To evaluate the technology, three replicate tests were performed using similar process conditions. Accordingly, the sampling plan for each test was similar. The demonstration consisted of three tests to ensure that adequate information could be





gathered to properly evaluate the technology and limit analytical bias from one set of data. The types of samples collected for each of the tests are presented in Table 5. Originally, it was also planned to collect samples of the scrubber solids. Due to the limited amount of mass trapped in the scrubber filters (less than 0.5% wt.), it was not possible to obtain enough sample for the required analyses. Hence, the scrubber solid and liquid samples were collected and analyzed together.

All liquid, solid, and gas samples were collected for analysis of the parameters listed in Table 5. Sampling containers were obtained from I-Chem Research and were cleaned to EPA protocol specifications. Miscellaneous sample containers, such as petri dishes, XAD-2 resins, Tenax®, etc. were provided by the sampling subcontractor and precleaned to specifications outlined in the Demonstration Plan.

Process measurements were made by MSE. The process was equipped with sensing equipment fitted with transmitters (i.e., thermocouples, flowmeters, etc.) to monitor all important parameters. The data acquisition system (DAS) checked each monitoring point and logged information every 30 seconds. Table 2 presents the process measurements made by MSE using the automated DAS. From these measurements, the DAS calculated several operational parameters as shown in Table 3. Visual monitors were provided in the control area to display these parameters to the operators. Additional measurements of the auxiliary equipment were collected by MSE at 30-minute intervals. These measurements included:

- furnace temperatures and pressures;
- torch parameters (i.e., pressure and flow of torch gas, and torch power supply);
- deionized and centrifuge water temperatures, pressures, and flow rates; and
- system pressures for the lid, feeder, exhaust, drive, and scrubber.

Table 5. Samples Collected During Demonstration Tests

Sample Location <sup>a</sup>	Sample Name	Sample Type	Parameter	Number of Samples	
				P	D
TEST 1					
S1	Feed Soil	Discrete	Volatiles	3	1
		Discrete	Semivolatiles	3	3 <sup>b</sup>
		Discrete	Metals Scan	3	3 <sup>b</sup>
		Composite	Dioxins/Furans	1 <sup>c</sup>	1 <sup>c</sup>
		Composite	TCLP	1	1
		Composite	Higher Heating Value	1	
		Composite	Chloride	1	
		Composite	Moisture (as received)	1	
S2	Treated Soil	Discrete	Semivolatiles	1	1
		Discrete	Metals Scan	1 <sup>b</sup>	1
		Discrete	Dioxin/Furans	1	1
		Discrete	TCLP	1	1
		Discrete	Bulk Density	1	
S4	Scrubber Liquor Before Test	Grab	Volatiles	1	1
		Grab	Semivolatiles	1	1
		Grab	Metals Scan	1	1
		Grab	Dioxins/Furans	1 <sup>c</sup>	1 <sup>c</sup>
S4	Scrubber Liquor After Test	Grab	Volatiles	1	1
		Grab	Semivolatiles	1	1
		Grab	Metals Scan	1	1
		Grab	Dioxins/Furans	1 <sup>c</sup>	1 <sup>c</sup>
S4	Scrubber Discharge Solids <sup>a</sup>	Grab	Volatiles	1	1
		Grab	Semivolatiles	1	1
		Grab	Metals Scan	1	1
		Grab	Dioxins/Furans	1 <sup>c</sup>	1 <sup>c</sup>

(Continued)

Table 5: (Continued)

Sample Location*	Sample Name	Sample Type	Parameter	Number of Samples	
				P	D
TEST 1 (CONTINUED)					
S5	Stack Gas	VOST (SW-846 M0030)	Volatiles	6	1
		Gas Canister (EPA Compendium MTO-14)		3 <sup>a</sup>	1 <sup>a</sup>
		EPA MM5	Semivolatiles	1	
		Multiple Metals	Metals Scan	1	
		EPA MM5	Dioxins/Furans	1	
		EPA M5	HCl Particulates	1	
		CEMs	O <sub>2</sub> , CO <sub>2</sub> , CO, NO <sub>x</sub> , SO <sub>2</sub> , THC	Continuous	
TEST 2					
S1	Feed Soil	Discrete	Volatiles	3	
		Discrete	Semivolatiles	3	3 <sup>b</sup>
		Discrete	Metals Scan	3	3 <sup>b</sup>
		Composite	Dioxins/Furans	1 <sup>c</sup>	
		Composite	TCLP	1	
		Composite	Higher Heating Value	1	
S2	Treated Soil	Discrete	Semivolatiles	1	1
		Discrete	Metals Scan	1	1
		Discrete	Dioxin/Furans	1	1
		Discrete	TCLP	1	1
		Discrete	Bulk Density	1	
S4	Scrubber Liquor Before Test	Grab	Volatiles	1	1
		Grab	Semivolatiles	1	1
		Grab	Metals Scan	1	1
		Grab	Dioxins/Furans	1 <sup>c</sup>	1 <sup>c</sup>

(Continued)

Table 5. (Continued)

Sample Location*	Sample Name	Sample Type	Parameter	Number of Samples	
				P	D
TEST 2 (CONTINUED)					
S3	Scrubber Makeup	Grab	Volatiles	1	
		Grab	Semivolatiles	1	
		Grab	Metals Scan	1	
		Grab	Dioxins/Furans	1 <sup>c</sup>	
S4	Scrubber Liquor After Test	Grab	Volatiles	1	1
		Grab	Semivolatiles	1	1
		Grab	Metals Scan	1	1
		Grab	Dioxins/Furans	1 <sup>c</sup>	1 <sup>c</sup>
S4	Scrubber Discharge Solids*	Grab	Volatiles	1	1
		Grab	Semivolatiles	1	1
		Grab	Metals Scan	1	1
		Grab	Dioxins/Furans	1 <sup>c</sup>	1 <sup>c</sup>
S5	Stack Gas	VOST (SW-486 M0030)	Volatiles	6	1
		Gas Canister (EPA Compendium MTO-14)		3 <sup>c</sup>	1 <sup>c</sup>
		EPA MM5	Semivolatiles	1	1
		Multiple Metals	Metals Scan	1	
		EPA MM5	Dioxins/Furans	1	1
		EPA M5	HCl Particulates	1	
		CEMs	O <sub>2</sub> , CO <sub>2</sub> , CO, NO <sub>x</sub> , SO <sub>2</sub> , THC	Continuous	

(Continued)

Table 5: (Continued)

Sample Location*	Sample Name	Sample Type	Parameter	Number of Samples	
				P	D
TEST 3					
S1	Feed Soil	Discrete	Volatiles	3	
		Discrete	Semivolatiles	3	3 <sup>b</sup>
		Discrete	Metals Scan	3	3 <sup>b</sup>
		Composite	Dioxins/Furans	1 <sup>c</sup>	1 <sup>c</sup>
		Composite	TCLP	1	
		Composite	Higher Heating Value	1	
S2	Treated Soil	Discrete	Semivolatiles	1	1
		Discrete	Metals Scan	1	1
		Discrete	Dioxin/Furans	1	1
		Discrete	TCLP	1	1
		Discrete	Bulk Density	1	
S4	Scrubber Liquor Before Test	Grab	Volatiles	1	1
		Grab	Semivolatiles	1	1
		Grab	Metals Scan	1	1
		Grab	Dioxins/Furans	1 <sup>c</sup>	1 <sup>c</sup>
S4	Scrubber Liquor After Test	Grab	Volatiles	1	1
		Grab	Semivolatiles	1	1
S4	Scrubber Discharge Solids*	Grab	Volatiles	1	1
		Grab	Semivolatiles	1	1
		Grab	Metals Scan	1	1
		Grab	Dioxins/Furans	1 <sup>c</sup>	1 <sup>c</sup>

(Continued)

Table 5. (Continued)

Sample Location*	Sample Name	Sample Type	Parameter	Number of Samples	
				P	D
TEST 3 (CONTINUED)					
S5	Stack Gas	VOST (SW-846 M0030)	Volatiles	6	1
		Gas Canister (EPA Compendium MTO-14)		3°	1°
		EPA MM5	Semivolatiles	1	
		Multiple Metals	Metals Scan	1	1
		EPA MM5	Dioxins/Furans	1	
		EPA M5	HCl Particulates	1	1
		CEMs	O <sub>2</sub> , CO <sub>2</sub> , CO, NO <sub>x</sub> , SO <sub>2</sub> , THC	Continuous	

Sample locations keyed to Figure 4.

Two of these samples were separate discrete samples collected at different times from the primary sample. One true duplicate was collected at the same time as the primary sample and analyzed for precision.

Samples were not analyzed since dioxins/furans were not detected in the treated soil or in the stack gas.

Since sufficient scrubber solids were not obtained for a separate analysis, scrubber liquor with suspended solids was analyzed.

Samples were not analyzed since there was neither breakthrough in the VOST cartridges nor saturation of the GC/MS detector during analysis of the VOST samples.

Primary Sample

Duplicate Sample

calculated parameters included:

plant water temperatures and flowrates; and  
off-gas heat removal data and chiller operating data.

starting the Demonstration Tests, extensive planning and evaluations were  
cted to ensure that representative samples would be collected to achieve project

objectives. Samples were collected at the locations noted in Figure 3. Below is a discussion of the procedures used to collect samples at these locations.

#### Feed Soil

Samples of the feed soil were collected during each loading of the feed screw. These samples were collected by obtaining a scoop of material from the top and middle of each 5-gallon pail that was fed to the furnace. These samples were collected using a small (1-cup) stainless steel scoop and depositing the sample in a 4-L glass jar. After each collection, the jar was sealed and agitated by hand to thoroughly mix its contents.

At the end of the test, the material that had been collected in the jar was portioned into aliquots and stored in separate containers for each selected analysis. Samples for all analytical parameters were collected using this procedure with the exception of the samples obtained for volatile organic analysis (VOA). Samples for VOA were collected at intervals specified in the QAPP during feeder loading. These discrete samples were immediately placed into 40-mL VOA vials and stored at 4°C.

#### Treated Soil

After each test, the treated soil was poured into a rectangular steel mold ("pig") and allowed to cool. After the material had solidified and cooled to approximately 100°F, sampling was conducted. These events took place the morning after treatment for Tests 1 and 2 and two days later for Test 3. The treated soil from Test 3 was too hot to sample the morning following treatment.

Sampling of the treated soil was conducted using a drill equipped with a diamond-tipped 2-inch coring bit. The drill was mounted on a specially designed stand which spanned the pig and could be firmly attached in place. This provided a sturdy, stable platform to operate the drill. Once mounted, the stand allowed the drill to operate similar to a drill press. The drill bit was cooled by using a Milli-Q water system. Cooling water was placed in a container, which could be pressurized by hand, and piped to the drill bit. When drilling was initiated, the cooling water was allowed to flow to the bit. The drill bit was lowered into place at selected locations within the pig to collect sample cores. Each 4- to 5-inch core sample took approximately 5 minutes to obtain. When the bit was raised from the slag, the core sample remained in the bit. There were, on occasion, some broken pieces that remained in the drilled hole. The broken fragments were removed from the hole using a stainless steel screwdriver. The core within the bit was dislodged by removing the bit from the drill, sliding a length of pipe down the internal shaft of the bit, and ramming the core until it fell from the bit. The entire core length (unbroken) depended on the depth of the treated soil in the pig. In most cases, the core was recovered in broken fragments. Pieces obtained from each core ranged in size from 1 to 2 inches.

Several cores were obtained from different locations within the treated soil pig for each test. After collecting a sufficient amount of material from each test, the sample cores were composited into a single sample for each test. Compositing was performed by placing the core fragments between two cotton cloths. A hammer was used to break the fragments into smaller pieces. After reducing the fragment size to less than one-half inch, the pieces were thoroughly mixed and placed into sample containers for analysis of each designated analytical parameter.

Samples of the Milli-Q water were also collected and shipped to the laboratory for analysis to check for contamination and account for any potential loss of material from the vitrified soil during the drilling process.



### **Scrubber Liquor**

Samples of the scrubber liquor were collected before and after each test. The samples collected at the beginning of the test were obtained from a tap in the scrubber recirculation lines. The scrubber was flushed clean at the beginning of each test and recharged with fresh tap water. Before samples were collected, the recirculation pumps were turned on to allow the water to thoroughly mix. A bucket was placed under the tap, and scrubber water was allowed to flow until 1 to 2 gallons were collected and the sample tap flushed. This was performed to ensure that the samples collected were representative of the actual contents of the scrubber and not line contamination. After purging the line, the 1-L sample bottles were placed under the tap and filled. Samples for volatile organic analysis were also collected using this technique and substituting the 1-L sample bottles with 40-mL VOA vials.

Samples of the scrubber liquor were also collected immediately following each test run. When the system was secured, samples were obtained from the same tap as the pre-test. These samples were collected using the same procedures described above.

### **Scrubber Caustic**

As with the scrubber liquor, a representative sample of the scrubber caustic in the sump was collected after Test 2. This sample was obtained from the discharge end of the scrubber makeup pump. The tubing from the discharge end was removed from its connection into the scrubber tank and placed into the sampling containers. The pump was allowed to operate until the sample containers were filled.

## Stack Gas

The stack gas was sampled for a number of parameters. All gas samples were collected downstream of the blower. The stack gas was collected from the exhaust duct located on the outside of the MSE test building. Several sampling ports were installed in the duct to accommodate the gas sampling trains. Each port was installed so that the spacing between them was at least 8 pipe diameters to facilitate isokinetic sampling when required. All piping in the exhaust duct was 3" sch 10 304L SS with an ID of 3.26". The sampling ports themselves were 3" x 3" tees with threaded adapters for the sampling probes. All stack gas probes (with the exception of the standard pitot tube and thermocouple probe) were designed to allow only the nozzle to protrude into the gas stream and thus prevent significant flow disturbances.

A port similar to the sample train ports, only with a 1" adaptor, was also installed to facilitate continuous emission monitoring equipment. This port was located upstream of all other ports to maintain sample integrity that may have been lost from in-leakage due to the installation and removal of other sample trains.

## Volatiles

The stack gas was sampled for volatile organic compounds (VOCs) using SW-846 Method 0030, volatile organic sampling train (VOST). This method is designed to provide analytical information on volatile organics with boiling points less than 100°C. Gas samples were collected on pairs of Tenax®-Tenax®/charcoal cartridges as described in the method. VOST samples were collected for each Demonstration Test. Tests 1 and 2 were conducted utilizing a single sample train to collect VOST samples throughout each test. Test 3 employed both a primary train and a duplicate train which operated during the same time frame as the primary train. To prevent overload on the resin cartridges, varying volumes of samples were collected during each test. Table 6 presents information on the samples collected for each test run.

Table 6. VOST Sample Identification

Time	Sample ID#	Sample Description
Test 1 (07/22/91)		
1400	SAIC-0129	20-L Pair
1540	SAIC-0130	20-L Pair
2530	SAIC-0131	Field Blank
1715	SAIC-0132	10-L Pair
1735	SAIC-1033	10-L Pair
1800	SAIC-0134	5-L Pair
1825	SAIC-0134	5-L Pair
1841	SAIC-0161	3-L Pair
1841	SAIC-0165	Condensate
1841	SAIC-0199	Trip Blank
Test 2 (07/24/91)		
1100	SAIC-0255	Field Blank
1116-1156	SAIC-0256	20-L Pair
1210-1250	SAIC-0257	20-L Pair
1302-1322	SAIC-0258	10-L Pair
1336-1356	SAIC-0259	10-L Pair
1429-1442	SAIC-0260	6.75-L Pair
1443	SAIC-0272	Condensate
Test 3 (07/26/91)		
2145	SAIC-0381	Primary Condensate
2145	SAIC-0382	Duplicate Condensate
1558-1638	SAIC-0386	Duplicate 20-L Pair
1558-1638	SAIC-0387	Primary 20-L Pair
1649-1729	SAIC-0389	Duplicate 20-L Pair
1648-1728	SAIC-0399	Primary 20-L Pair
1737-1757	SAIC-0390	Duplicate 10-L Pair
1738-1758	SAIC-0391	Primary 10-L Pair
1808-1828	SAIC-0392	Duplicate 10-L Pair
1809-1829	SAIC-0393	Primary 10-L Pair
1841-1851	SAIC-0394	Duplicate 5-L Pair
1841-1851	SAIC-0395	Duplicate 5-L Pair
2127-2130	SAIC-0396	Duplicate 1.69-L Pair
2127-2130	SAIC-0397	Primary 1.69-L Pair
1900	SAIC-0398	Field Blank

In the event of breakthrough of the VOST cartridges or saturation of the GC detector, gas canister samples were also collected as a backup for the VOST samples. The gas canister samples were collected per EPA Compendium Method TO-14. As with the VOST, Tests 1 and 2 were sampled with a single canister sampling train and Test 3 included a duplicate canister sampling train. Six-liter samples were collected in evacuated stainless steel canisters.

### **Semivolatiles and Dioxins/Furans**

Samples for semivolatiles and dioxins/furans were each collected from the stack using the Modified Method 5 (MM5) sampling trains in accordance with the method specified in SW-846 (Method 0010). The MM5 train is designed to sample gaseous and particulate pollutants with boiling points greater than 100°C. Samples are pulled from the stack isokinetically and then passed through a filter and a porous polymeric resin to trap the components of interest.

Samples were collected nominally at approximately 0.5 scfm until a total of 106 ft<sup>3</sup> of sample were obtained. During sample retrieval in the field, each component of the sample train was removed and rinsed with methylene chloride. This rinse was collected and sent to the laboratory for analysis. For the dioxin/furan MM5 sample trains, toluene was used instead of methylene chloride. All other procedures took place as described in the method.

One MM5 sample train was used to collect semivolatile stack samples for Tests 2 and 3. Test 1 semivolatile stack samples were collected in duplicate. One sample train was used to collect dioxin/furan stack samples in Tests 1 and 3, while Test 2 sampling was performed in duplicate.

## **Metals**

Samples of the stack gas for metals analysis were collected using the multiple metals train. This method is similar the MM5 method sampling methodology and is described in SW-846 draft Method 0012.

For the multiple metals method, nitric acid, peroxide, potassium permanganate and sulfuric acid are utilized in the impinger system to remove contaminants from the gas stream. Samples are collected isokinetically and particulates are captured on a filter (similar to Method 5) which is later digested for analysis. For these tests, a minimum of 30 ft<sup>3</sup> was required to meet Demonstration Test objectives. Samples were collected at a rate of approximately 0.5 scfm.

One multiple metals train was used to collect gas samples for Tests 1 and 3. A duplicate sample train was installed for Test 2.

## **Particulates/HCl**

The stack gas was sampled for particulates and hydrogen chloride (HCl) using the EPA Method 5 (M5) sampling train. This method involves isokinetic sampling of the stack gas similar to the MM5 sample trains. Here the impinger system employs sodium hydroxide and silica gel to determine contamination levels in the gas stream.

The gas was sampled at a rate of 0.5 scfm until a total of 30 cubic feet were obtained for each sample. After the samples were collected, the train was removed to a clean area for sample recovery utilizing acetone and deionized water.

## Continuous Emission Monitoring

Continuous Emission Monitors (CEMs) were used for each of the test runs to monitor the stack gas concentrations of CO<sub>2</sub>, CO, O<sub>2</sub>, SO<sub>2</sub>, NO<sub>x</sub>, and THC. Samples were collected by inserting a stainless steel probe into one of the 1" sampling ports. Gas was withdrawn and transported through a heated Teflon® sample line to the instrument trailer. Once inside the trailer, the gas was conditioned before entering the analytical equipment. Conditioning was performed by passing the gas through several short-stemmed impingers immersed in an ice bath to remove any water that was entrained in the gas. Following the impingers, the gas was directed through a glass fiber filter which removed particulates. The gas was pulled through the system by a Teflon®-bladder diaphragm pump. Gas exiting the pump was sent to a manifold which supplied sample to each of the CEM instruments.

## **ANALYTICAL PROTOCOLS**

Analytical protocols were selected to provide reliable data. In most cases, the Demonstration Test samples were analyzed using standard EPA-approved methods. There were some variations and modifications made to these methods as noted in Section 8 of this report. The following describes the analytical methods used for this demonstration.

### Volatile Organics

Analyses for VOCs of all solid and liquid samples obtained during the Demonstration Tests were done in accordance with SW-846 Method 8240. This method is a GC/MS method in which the sample is introduced to a purging tube and an inert gas is bubbled through the sample. The volatiles are removed from the sample and swept

into the gas phase. The gas is then collected and concentrated in a trap that contains a sorbent material. After the sample has been sufficiently purged, the trap is rapidly heated to desorb the volatiles into a gas chromatograph. The sample is separated in the gas chromatograph and sent to a mass spectrometer for detection. The mass spectrometer is calibrated by spiking reagent water with pollutants of interest and analyzing under the same conditions as the samples.

Before purging the standards and samples, internal standards and surrogates are added to the purge tube. Quantitation of the sample is performed by comparing the response of the samples to that of the standard. Corrections are made for the recovery of the internal standards.

The VOST samples were analyzed using SW-846 draft Method 5041 and SW-846 Method 8240. Method 5041 was required to desorb the volatiles from the VOST tubes. For the analysis of the VOST tubes, the GC/MS was first calibrated using a flash evaporation technique which involved the loading of the standards on a pair of VOST cartridges. The VOST cartridge was then placed in a clamshell heater to rapidly desorb the volatiles. An inert carrier gas backflushed the volatiles off the VOST cartridges. The volatiles passed through a sparge tube with 5 mL of reagent water containing surrogate and internal standards. The volatiles were collected on the concentrator tube in the purge and trap device. Following the desorption, the analysis proceeded as described in SW-846 Method 8240. Samples were analyzed under the same conditions as the standards.

If the results of the VOST tubes had exceeded the calibration range of the GC/MS, it would have been necessary to analyze the gas canister sample for volatiles. Since the results of the VOST samples were satisfactory, the gas canisters were not analyzed.

## Semivolatiles

Semivolatile samples collected during the Demonstration Tests were analyzed using SW-846 Method 8270. Method 8270 is a GC/MS method in which a sample extract is injected into a gas chromatograph. The chromatograph splits the components of interest which are then detected by the mass spectrometer. Surrogate standards were added to the sample at the time of extraction to measure the extraction efficiencies. Internal standards were added to the sample extracts before analysis. Calibration of the mass spectrometer was performed in accordance with the procedures prescribed in the method. Method 8270 was used to analyze the feed soil, treated soil, TCLP extract for the treated soil, scrubber liquor before and after each test, the scrubber caustic, and the stack gas samples.

To prepare the samples for analysis by SW-846 Method 8270, the compounds of interest must first be extracted from the media of interest. Liquid samples were extracted using Method 3520, continuous liquid-liquid extraction. Solid samples, including the XAD-2 resin from the MM5 sample train, were extracted using Method 3540, soxhlet extraction. The treated soil from the test had to be pulverized into a fine powder before it was extracted. This was accomplished by running the material through a crusher and placing the smaller pieces in a mechanical mortar and pestle until a fine powder was obtained.

## Dioxins and Furans

Analysis for polychlorinated dioxins and furans was performed using SW-846 draft Method 8290, which is a high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS) method. It was not anticipated that dioxin and furans would be present in any of the matrices. However, hexachlorobenzene, which has the potential of forming these compounds, was spiked into the feed soil. Method 8290



was selected to detect dioxins and furans at very low levels. The sampling plan called for the analysis of the treated soil and the stack gas to determine if these compounds were present. If these compounds were detected at significant levels in these matrices, the remaining samples would be analyzed. Since dioxin and furans were not detected at significant levels in the stack gas or the treated soil, these additional samples were not analyzed.

Method 8290 is a rigorous method for the detection of polychlorodibenzodioxins and polychlorodibenzofurans (PCDDs/PCDFs) which involves matrix-specific sample preparation and cleanup. Before extraction, the samples are spiked with specific amounts of nine isotopically ( $^{13}\text{C}_{12}$ ) labeled PCDDs/PCDFs. The samples then undergo the extraction procedure and extract cleanup. The final extract is prepared by adding two recovery standards to determine the percent recoveries of the PCDD/PCDF congeners and the hexa-, hepta-, and octachlorinated PCDD/PCDF congeners. One to two  $\mu\text{L}$  of the extract are injected into an HRGC/HRMS capable of performing selected ion monitoring and resolving powers of at least a 10 percent valley definition. Compounds are detected and identified based on their elution at their exact retention time, per the calibration, and the simultaneous detection of the two most abundant ions in the molecular ion region. Confirmation is performed by a comparison of the ratios of the integrated ion abundance of the molecular ion species to their theoretical abundance ratios. Quantitation is achieved in conjunction with a multipoint calibration curve for each targeted compound.

### Metals

All of the sample matrices collected during the Demonstration Tests were analyzed for metals. All liquid samples were digested per Method 3010 of SW-846. The filters that collected particulate matter from the multiple metals trains were digested using SW-846 Method 3050. A modification of Method 3051 was used for the digestion

of all soil samples for metals analyses. This is a microwave digestion procedure. The modification to this method requires the use of hydrofluoric acid in combination with hydrochloric acid and nitric acid to completely dissolve the sample rather than to leach the sample. A total digestion was particularly necessary for the metals analyses of the treated soil because of the limited leachability of metals in this matrix. The method was also used for the feed soil samples so that reported values are comparable when performing a material balance. The glassified soil was crushed prior to digestion as described for semivolatile analysis.

Analyses of the digestates were performed using primarily SW-846 Method 6010 which is inductively coupled plasma atomic emission spectroscopy method (ICP). This method measures element-emitted light by optical spectrometry. The quantity of light emitted is directly proportional to the amount of material present in the sample. Arsenic concentrations were measured using SW-846 Method 7060. This method is a graphic furnace procedure in which a sample of the digestate is atomized in a graphic tube furnace. The absorption of a specific wavelength of light is proportional to the arsenic concentration. Selenium was measured by SW-846 Method 7740 which is also a graphic furnace technique. The procedure is the same as the arsenic method but uses a different wavelength of light. Mercury was analyzed by Method 7471 which is a cold-vapor atomic absorption method. The mercury is reduced to the elemental state and aerated from solution in a closed system. The vapor passes through a cell position in the light path of an atomic absorption spectrophotometer. Absorbance is measured on a strip chart recorder where the height of the peak is a function of the mercury concentration.

#### Toxicity Characteristic Leaching Procedure

Feed soil and treated soil were tested for leachable metals and semivolatiles organic compounds using the Toxicity Characteristic Leaching Procedure (TCLP). TCLP

analyses were conducted in accordance with the procedures outlined in SW-846 Method 1311. The TCLP method is designed to determine the mobility of organic and inorganic analytes present in liquid, solid, and multiphase wastes. The method involves selecting an appropriate extraction fluid, then extracting the waste by agitation for  $18 \pm 2$  hours. The extract is then collected by filtration and subjected to the required analytical procedures. Semivolatiles were extracted and analyzed using the procedures defined in SW-846 Methods 3520/8270. Metals were analyzed per SW-846 methods outlined above.

TCLP requires that solid waste pass through a 9.5 mm standard sieve. Since the treated soil was a solid monolith, particle size reduction was necessary. This was accomplished by crushing and grinding the material as described previously.

#### Chloride Analysis

Samples collected in the M5 sample train were analyzed for HCl using a Dionex Model 2010 ion chromatograph following Method 27 from the "FGD Chemistry and Analytical Methods Handbook", Radian Corporation, Volume 2, July 1984. This method involves ion separation on an ion exchange column and detection of these ions conductimetrically.

Chloride analysis of feed soil samples were performed in accordance with ASTM Method D-2361.

#### Higher Heating Value

The higher heating value of the test soil was determined using a bomb calorimeter in accordance with ASTM Method D2015-85. The method involves charging a known

quantity of material inside a bomb calorimeter, igniting it, and determining the amount of heat released.

### Particulate Matter

The particulate determination from the M5 sample train was determined using a gravimetric procedure. A pre-weighed filter was used in the sample train, then reweighed after the test. The acetone rinsates generated during sample recovery were allowed to evaporate and the residue was also weighed. The amount of residue from the rinsate was added to the residue collected on the filters to determine the total particulate catch.

### Density

The bulk density of the treated soil was determined using EPA Method 25A. The samples were placed in a pre-weighed graduated cylinder to a prescribed volume. The cylinder was re-weighed and the density was determined.

### Continuous Emission Monitoring

Gas emissions were continuously monitored using on-line instrumentation. The data obtained from these instruments were recorded every 2 seconds using a computer based data acquisition system. Although measured, SO<sub>2</sub> data was later determined to be unusable because of interferences caused by high NO<sub>x</sub> levels in the gas stream. All CEM instruments were calibrated using certified gas standards. Below is a discussion of the analysis method for each parameter.

## **Carbon Monoxide**

A Bendix Model 85-105CA analyzer was used to measure CO in the stack gas according to EPA Method 10. The instrument operates by using a dispersive infrared analyzer which measures the concentration of CO by infrared absorption at a characteristic wavelength.

## **Carbon Dioxide**

A MSA Model 303 non-dispersive infrared analyzer was used to detect the concentration of CO<sub>2</sub> in the stack gas according to EPA Method 3A. This monitor operates by measuring the absorption of infrared radiation at a characteristic wavelength.

## **Oxygen**

A Taylor Model 540A oxygen analyzer was used to determine the concentrations of O<sub>2</sub> in the stack gas in accordance with EPA Method 3A. The analyzer measures O<sub>2</sub> concentrations based on the magnetic properties of O<sub>2</sub>.

## **Sulfur Dioxide**

A TECO Model 40 pulsed fluorescent analyzer was used to measure SO<sub>2</sub> in the stack gas continuously (EPA Method 6C). The instrument detects SO<sub>2</sub> based on the absorption of ultraviolet radiation. As the molecule returns to the ground energy state, fluorescence occurs. The amount of fluorescence is related to the concentration of SO<sub>2</sub>.

## **Nitrogen Oxides**

A TECO Model 10 analyzer was used for NO<sub>x</sub> measurements in the stack gas according to EPA Method 7E. The instrument works by converting all of the nitrogen oxides present in the sample to nitric oxide. The nitric oxide is then reacted with ozone. This reaction produces a chemiluminescence proportional to the NO<sub>x</sub> concentration in the sample. The chemiluminescence is detected by a high-sensitivity photomultiplier.

## **Total Hydrocarbons**

A Beckman Model 400A was used to continuously measure the concentration of hydrocarbons present in the stack gas in accordance with EPA Method 25A. This instrument uses a hydrogen flame ionization detector which ionizes the sample as it passes through the flame. The carbon atoms in the sample are ionized to produce positive cations and electrons. These charged particles are collected and produce a current which is measured. The current generated is directly proportional to the concentration of hydrocarbons present in the sample.

## SECTION 6

### PERFORMANCE AND DATA EVALUATION

#### INTRODUCTION

Three Demonstration Tests were performed to evaluate the effectiveness of the Tech, Inc. Plasma Centrifugal Furnace (PCF-6) in treating the waste feed matrix described in Section 3 and to evaluate the feasibility of employing similar, but full-scale units at hazardous waste treatment facilities throughout the country. To facilitate this evaluation, the following "critical" and "noncritical" objectives were established:

ical:

to characterize the residues produced at optimum operation including Destruction and Removal Efficiency (DRE), fate and transport of metals, and residue quality;

to identify pre- and post-feed waste treatment requirements.

to evaluate the ability of the furnace to effectively vitrify inorganic and metal constituents within a soil into a monolithic nonleachable mass. (Zinc oxide was spiked into the soil at a level of 28,000 ppm.)

- to determine if the furnace can meet 99.99% DRE for the target analytes in a soil contaminated with up to 10% organics. (Hexachlorobenzene was spiked into the feed soil at 1,000 ppm so that a DRE of 99.99% could be positively determined. DREs for other target analytes, i.e., SW-846 Method 8240 and 8270 compounds as listed in the Quality Assurance Project Plan (Section 5 of the Demonstration Plan), were determined if these compounds were present at high enough levels in the feed soil.)

**Noncritical:**

- to achieve heat and mass balances;
- to characterize the performance of the process;
- to identify the need for secondary treatment;
- to isolate operational problems in the field;
- to identify solutions to potential problems;
- to identify government policy and regulatory requirements;
- to evaluate potential uses of the process;
- to provide a comparison against competitive technologies;
- to develop operating costs; and
- to determine the useful life of the equipment.

In addition to allowing an evaluation of the technology for potential Superfund applications, the activities and results of this testing will also provide assistance to DOE in their evaluation of the technology for the remediation of hazardous waste sites under their jurisdiction.

All detailed analytical results are presented in Volume II of this Report. Generally, only results that have undergone data reduction appear in this section.



## TOXICITY CHARACTERISTIC LEACHING PROCEDURE

The PCF is designed to encapsulate inorganic compounds in the vitrified slag and render the treated soil nonleachable. Testing activities have demonstrated that the process can effectively bind inorganic compounds into the treated soil. The Toxicity Characteristic Leaching Procedure (TCLP) was performed on both the feed soil and the produced slag. The feed soil was tested to establish initial values for the leachability of organic and inorganic compounds. The vitrified slag underwent TCLP to meet the testing objectives.

TCLP analysis of the feed soil for metals showed that the only elements which exhibited significant leachability characteristics were: calcium, sodium, and the spiked zinc. Table 7 summarizes the results of the TCLP metals analysis of the feed soil. The presence of sodium in the leachate is not unexpected because of its high concentration in the soil and the fact that it is a weakly dissociable metal. This means that sodium, unlike other metals, is readily soluble. If the solution is even slightly acidic (as in the TCLP) this phenomenon is enhanced. None of the eight RCRA characteristic metals found in the feed soil leachate were above the regulatory limit. The evaluation of the leachability of the vitrified slag was based on calcium and zinc. Calcium was chosen, in addition to zinc, because of its tendency to leach from the feed soil. Sodium was not monitored because of its unusual solubility characteristics as explained above.

The treated soil TCLP metals analysis is also shown in Table 7. None of the metals, with the exception of sodium, showed any strong characteristic for leaching. Sodium is probably present in the leachate for the reasons stated above and was not considered in this evaluation. Both tracer metals, calcium and zinc, showed significant reductions in leaching properties in the treated soil as compared to the feed. In fact, all of the metals, with the exception of aluminum and iron showed reduced leachability characteristics. The leachability for the aluminum in both feed

Table 7. TCLP Results for Demonstration Tests

Compound	Average Feed Soil Leachate Concentration (mg/L)	Treated Soil Leachate Concentration		
		Test 1 (mg/L)	Test 2 (mg/L)	Test 3 (mg/L)
Metals				
Aluminum	0.23 J	0.45 J	0.415 J	0.32 J
Barium	0.14	0.078	0.085	0.075
Cadmium	0.067	<0.039	<0.039	<0.039
Calcium	175	2.1 J	2.55 J	2.05 J
Copper	4.6	0.15	0.355	0.305
Iron	0.063	2.5	2.95	31.2
Magnesium	8.12	<0.039	<0.039	<0.039
Manganese	4.82	0.057	0.061	0.245
Nickel	0.022	<0.011	0.010 J	0.11
Potassium	4.58	<0.70	<0.70	<0.70
Sodium	1,475	1,500	1,400	1,400
Vanadium	0.099	<0.0043	<0.0043	<0.0043
Zinc	982	0.45	0.36	0.30
Semivolatiles				
Hexachlorobenzene	<0.0010	<0.0010	<0.0010	<0.0010
2-Methylnaphthalene	0.282	<0.0019	<0.0019	<0.0019
Naphthalene	0.397	<0.0026	<0.0026	<0.0026

J Estimated result. Indicates that the result is less than the quantitation limit. The quantitation limit is defined as 5 times the detection limit.

< Indicates that this constituent was not detected at or above the detection limit.

and treated soil is low and the values reported for the treated soil are only estimates (less than the quantitation limit). Therefore, it is quite probable that the leachability of aluminum from the feed soil as compared to that from the treated soil did not change. The increase in leachability of iron in the treated soil is probably because approximately 100 pounds of mild steel were placed in the furnace to aid in initiating the arc of the torch (see Section 4). This considerably increased the iron content of the slag in comparison to the feed soil.

The only organic constituents that were found to be leachable from the feed soil were 2-methylnaphthalene and naphthalene, as shown in Table 7. Although the feed soil was spiked with a high level of hexachlorobenzene (1,000 ppm), it did not leach from the soil. No organic compounds were found to leach from the treated slag.

The Toxicity Characteristic Leaching Procedure requires samples to be ground into small particles. In this manner, a large amount of surface area is available for leaching. Since the PCF produces a monolithic slag after treatment, the surface area per pound of treated soil is much smaller than that of the feed soil. The TCLP results, therefore, present a conservative assessment of the actual leachability of the monolithic slag.

#### DESTRUCTION AND REMOVAL EFFICIENCY

The Destruction and Removal Efficiency (DRE), used to determine organic destruction, is determined by analyzing for the Principal Organic Hazardous Compound (POHC) in the feed soil and the stack gas. The DRE may be calculated as follows:

$$\text{DRE (\%)} = \frac{\text{mass in} - \text{mass out}}{\text{mass out}} \times 100\%$$

For these tests, the POHC was hexachlorobenzene. The estimated mean level of hexachlorobenzene, based on all the feed soil samples for the three tests was 972 ppm (see Table 8). The 95% confidence interval for the estimated mean was 864 to 1,080 ppm. No hexachlorobenzene was detected in the stack gas, therefore, all hexachlorobenzene DREs determined are based on the detection limit from the appropriate tests. Table 9 gives these DREs based on the 95% confidence interval of the feed soil and the detection limit for each test.

Table 8. Organic Compounds in the Demonstration Test Soil

Compound	lb/100 lb feed	ppm
Volatiles*		
Benzene	9.91E-05	0.991
Ethyl Benzene	2.84E-03	28.4
Toluene	1.81E-03	18.1
Xylene	1.34E-02	134
Semivolatiles**		
Hexachlorobenzene	9.72E-05	972
2-Methylnaphthalene	4.58E-05	458
Naphthalene	1.52E-05	150
Phenanthrene	6.62E-06	66.2

\* 1,2-Dichloroethane and methyl ethyl ketone were both detected a few samples, but only at low levels.  
 \*\* Other compounds were detected in a few samples, but only at low levels.

Table 9. DRE Results for Demonstration Tests

Compound	Test 1	Test 1 Duplicate	Test 2	Test 3
Hexachlorobenzene				
Lower 95% Confidence Interval Limit	>99.9964	>99.9982	>99.9990	>99.99989
Mean	>99.9968	>99.9984	>99.9991	>99.99990
Upper 95% Confidence Interval Limit	>99.9971	>99.9986	>99.9992	>99.99991
2-Methylnaphthalene				
Lower 95% Confidence Interval Limit	>99.9853	>99.9930	>99.9958	>99.99960
Mean	>99.9872	>99.9939	>99.9964	>99.99965
Upper 95% Confidence Interval Limit	>99.9891	>99.9948	>99.9969	>99.99970

As can be seen from Table 9, the estimated average DRE values for this test ranged from >99.9968% to >99.9999% for a highly chlorinated compound, hexachlorobenzene. It can be reasonably assumed that this level of DRE (if measurable) can be achieved for most chlorinated or halogenated compounds.

The Silver Bow Creek soil was mixed with 10% by weight No. 2 diesel oil, in addition to being spiked with hexachlorobenzene. Analysis of the mixed feed soil indicated that sufficient 2-methylnaphthalene, another semivolatile compound, was present at sufficiently high levels in the feed to determine a significant DRE for each test. The 95% confidence interval for 2-methylnaphthalene was 390 to 526 ppm with an estimated mean of 458 ppm as shown in Table 8. This level of contamination in the feed soil leads to the range of DREs given in Table 9, again based on detection limits because none of this compound was detected in the stack gas.

Total xylenes, a group of volatile compounds, were also found in sufficient quantities in the feed soil to determine a significant DRE. The 95% confidence interval for the estimated mean of the total xylenes was 128 to 139 ppm. The DREs associated with this confidence interval for the three tests were >99.9929% to >99.9934%. Throughout each of the Demonstration Tests, multiple Volatile Organic Sampling Train (VOST) samples were taken. VOST samples were obtained using SW-846 Method 0030 which designates specific volumes collected over a short duration. The DREs presented are an average over all three tests based on the 95% confidence interval of xylenes in the feed soil. Averages of all the DREs can be taken because the detection limits obtained are the same for all the VOST samples.

Overall, the PCF appeared to be very efficient in destroying both volatile and semivolatile compounds when both the primary reaction chamber and the afterburner were operating.

#### **ACID GAS REMOVAL AND PARTICULATE EMISSIONS**

Measured HCl emission rates ranged from 0.0007 to 0.0017 lbs/hr. The chlorine concentration in the feed soil for Test 1 was 0.066%. This leads to a HCl removal efficiency of 98.5%. Because of the low chlorine input, the regulatory requirement

of the larger of either 4 lbs/hr or 99% removal [40 CFR (07/01/91 Edition) §264.343(b)] was met. The removal efficiency may not be meaningful because of the low chlorine input. However, it appears that even if the chlorine in the feed had been higher, an HCl removal efficiency of 99% could be achieved.

As shown in Table 10, the particulate emissions during each of the three tests exceeded the regulatory limit of 0.08 grains/dscf [40 CFR (07/01/91 Edition) §264.343(c)]. These emission rates have not been corrected for 7% oxygen. The 7% oxygen correction factor is  $14\% / (21\% - O_2\%)$ . The oxygen correction is required by RCRA for all hazardous waste incinerators except those operating under the condition of oxygen enrichment. The purpose for correcting for 7%  $O_2$  in conventional incineration systems is to account for the dilution factor in the stack gas caused by using excess air for combustion. For the Retech process, pure oxygen is fed to the primary chamber through an oxygen lance as soon as feeding of the first batch of soil begins. The  $O_2$  is continually introduced to the furnace throughout the remainder of the treatment process. The  $O_2$  content of the stack gas when no soil is being fed to the furnace (i.e., between feeding cycles, during recharging of the feeder), is in excess of 21%. As stated above, RCRA does not require the 7%  $O_2$  correction factor for hazardous waste incinerators operating under oxygen enrichment [40 CFR (07/01/91 Edition) §264.343(c)]. Therefore, the PCF is exempt from this correction factor. During feeding of the soil to the furnace, the stack gas  $O_2$  level drops to approximately 11%. Therefore, if the correction is to be applied only during the feeding cycle (when presumably the particulates are being generated), then the values given in Table 10 should be increased by a factor of 1.4.

Table 10. Particulate Results for Demonstration Tests

Parameter	Test 1	Test 1 Duplicate	Test 2	Test 3
Particulate Concentration (grains/dscf)	0.341	0.240	0.422	0.410
Particulate Emissions (lb/hr)	0.342	0.238	0.418	0.423

The amount of particulates captured by the air pollution control system was extremely small. This is demonstrated, in part, by the low level of scrubber solids present in the sump. There was less than 0.5% total solids in the scrubber sump tank. The consequence of this high particulate loading during the Demonstration Test was a substantial buildup of particulate matter in the exhaust blower after the air pollution control system. The pressure differential across the exhaust blower was reduced because of the particulate build-up, and the first two Demonstration Tests were shortened due to this problem. A larger blower was installed for the third Demonstration Test, which was completed as scheduled, but particulate build-up still persisted.

The Silver Bow Creek Superfund Site soil was extremely dry and dusty before it was mixed with the diesel oil. Even after mixing the soil with the diesel oil, it was very free-flowing with no standing liquid. It is possible that the fines from this feed may not have been retained in the melted soil in the primary reaction chamber and simply passed through the treatment process and the scrubbing unit and into the exhaust blower and stack gas. If the dust did pass through the treatment process, it would be expected that a well-designed wet scrubbing system would be capable of capturing the particulates. If the feed soil does not contain any halogenated compounds, then the process does not require a wet scrubbing system and a baghouse could be used to control the particulate emissions. Judicious selection of the most suitable air pollution control device is necessary before effective implementation of this technology can be achieved.

## **AIR EMISSIONS**

The air emissions consisted primarily of products of incomplete combustion (PICs) and particulates. Table 11 presents a summary of the semivolatile organic compounds emitted in the stack gas. For the case of the emitted semivolatile organics, the most

Table 11. Stack Gas Composition During the Demonstration Tests

Compound	Test 1		Test 2		Test 3	
	lbs/100 lbs feed	ppm	lbs/100 lbs feed	ppm	lbs/100 lbs feed	ppm
Semivolatiles						
Acetophenone	<2.15E-06	<0.38	4.63E-06 J	0.71 J	2.30E-06	0.031
Benzoic Acid	3.77E-04	6.7	3.29E-04	4.9	1.98E-04	2.6
Benzyl Alcohol	<4.95E-07	<0.0099	<1.65E-07	<0.0033	3.76E-07 J	0.0056 J
Butylbenzylphthalate	<3.28E-07	<0.0023	<1.09E-07	<0.0008	3.76E-07 J	0.0056 J
Dibutylphthalate	<9.93E-07 B	<0.0077 B	4.48E-06 JB	0.030 JB	1.15E-06 B	0.0067 B
Diethylphthalate	<5.41E-05 B	<0.70 B	2.54E-05 B	0.28 B	2.50E-05 B	0.24 B
2,4-Dinitrophenol	2.13E-05 J	0.25 J	1.27E-05 J	0.13 J	5.74E-06	0.050
bis(2-Ethylhexyl)phthalate	7.36E-05 B	0.41 B	5.08E-05 B	0.24 B	4.69E-06 B	0.019 B
Naphthalene	1.68E-05 B	0.26 B	8.96E-06 B	0.12 B	1.15E-05 B	0.134 B
Nitrobenzene	<3.13E-07 J	<0.0055 J	2.84E-06 J	0.042 J	1.56E-06	0.021
2-Nitrophenol	<8.56E-06 J	<0.13 J	2.09E-06 J	0.028 J	1.36E-05	0.16
4-Nitrophenol	1.15E-05 J	0.18 J	<1.43E-05	<0.22	1.01E-05	0.12
Metals						
Aluminum	5.02E-04 B	1.34 B	9.56E-04 B	2.10 B	5.67E-04 B	1.14 B
Antimony	1.18E-05	0.031	4.16E-05	0.091	2.62E-05	0.053
Arsenic	1.88E-03 B	5.02 B	2.98E-03 B	6.54 B	2.47E-03 B	4.97 B
Barium	4.26E-05 B	0.114 B	8.09E-05 B	0.177 B	6.11E-05 B	0.123 B
Beryllium	1.63E-07	0.001	3.58E-07	0.001	2.91E-07	0.001
Cadmium	1.76E-05	0.047	3.99E-05	0.088	2.76E-05	0.056
Calcium	8.41E-04 B	2.51 B	1.74E-03 B	3.81 B	9.60E-04 B	1.93 B
Chromium	3.39E-04 B	0.804 B	6.57E-04 B	1.44 B	7.27E-04 B	1.46 B
Copper	1.63E-03 B	4.35 B	4.71E-03 B	10.3 B	2.04E-03 B	4.09 B
Iron	1.51E-02 B	40.2 B	3.01E-02 B	66.1 B	4.51E-02 B	90.6 B
Lead	9.28E-04 B	2.48 B	2.29E-03 B	5.02 B	1.60E-03 B	3.21 B
Magnesium	2.01E-04 B	0.535 B	3.26E-04 B	0.716 B	2.47E-04 B	0.497 B
Manganese	1.14E-04 B	0.305 B	2.48E-04 B	0.544 B	3.20E-04 B	0.643 B
Mercury	1.38E-06 B	0.004 B	2.92E-06 B	0.006 B	5.09E-06 B	0.010 B
Nickel	6.65E-05 B	0.177 B	1.10E-04 B	0.372 B	1.74E-04 B	0.351 B
Potassium	3.76E-03	10.0	7.11E-03	15.6	6.69E-03	13.4
Selenium	5.02E-06	0.013	1.11E-05	0.024	6.40E-06	0.013
Silver	2.88E-06	0.008	6.32E-06	0.014	4.65E-06	0.009
Sodium	2.26E-03 B	6.02 B	4.26E-03 B	9.35 B	3.78E-03 B	7.56 B
Thallium	5.52E-06	0.015	1.35E-05	0.030	1.25E-05	0.025
Vanadium	2.76E-05	0.074	6.57E-05	0.144	4.51E-05	0.091
Zinc	3.01E-02	80.3	8.36E-02 B	183 B	5.53E-02 B	111 B

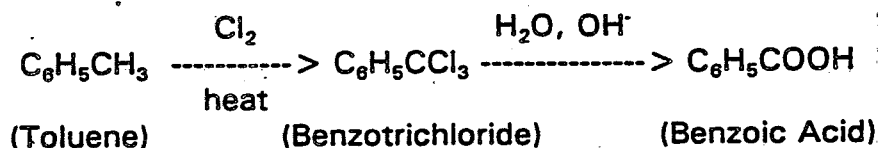
B Indicates that this compound was detected in a blank.

J Estimated result. Indicates that result is less than the quantitation limit. The quantitation limit is defined as five times the detection limit.

< Not detected at or above the detection limit.



dominant compound released in the stack gas is benzoic acid at an average concentration of approximately 4 ppm. The occurrence of benzoic acid in the stack gas is to be expected as both toluene (from the diesel oil) and chlorine (from the hexachlorobenzene) were present in the feed soil. These two compounds, with the addition of heat, readily form benzoic acid as shown in the basic reaction presented below:



The water and hydroxide required to complete the reaction were provided by the scrubber.

- A group of nitrated compounds was found in the stack gas at low levels (< 0.3 ppm). These compounds were formed because of the high levels of NO<sub>x</sub> and trace quantities of organic compounds in the stack gas interacting with the scrubber liquor spray. Other compounds such as the phthalate groups and naphthalene were also present but were found in the field blanks as well.

In addition to the target compounds identified by the SW-846 Method 8270 list, the next 20 highest peaks from the chromatograms were investigated for compound identification and semi-quantification. A review of the chromatograms and the spectral data showed that some Tentatively Identified Compounds (TICs) were present in the gas stream. The first Demonstration Test yielded a relatively clean chromatogram that was somewhat comparable to the field blank. No TICs could be positively identified, but it appeared that some of the unknowns contained oxygen somewhere in the molecular structure and some of the unknowns were nitrogen-containing compounds.

Test 2 data appears to be notably different from the Test 1 data. The chromatogram indicated that several higher molecular weight compounds were detected. The TIC data shows that these compounds were unidentifiable carbonic acids. It should be noted that there were no positive identifications made on the specific type of carbonic acid, but the pattern was present in several of the TICs. Carbonic acids are formed by reacting carbon dioxide with water at high temperature. This may suggest that the torch cooling water leak detected during Test 3 could have developed during Test 2 (see Section 4). Because of this leak, Test 3 was aborted early and restarted after the leak had been repaired. Compounds that eluted in the early stages of the chromatogram contained nitrogen and oxygen within their molecular structure.

Data from Test 3, collected after the leak was repaired, was very similar to the TIC information gathered from Test 1. In general, only low levels of semivolatile organic compounds were identified in the gas stream.

Very low levels of volatile organic compounds were detected in the exhaust gas stream. The most common of these compounds was benzene at an average concentration of approximately 19 ppbv. Benzene and substituted benzenes are prevalent in many forms throughout diesel oil and hence benzene is a readily formed PIC. Other identified compounds in the stack gas tended to be chlorinated organics which were not identified in the feed soil at the detection limits achieved for the testing and therefore can possibly be PICs.

Sampling and analysis for polychlorodibenzodioxins (PCDDs) and polychlorodibenzofurans (PCDFs) in the exhaust gas stream was accomplished during the Demonstration Tests. The results of these analyses indicated that no PCDDs or PCDFs were formed in the stack gas. Although some PCDDs and PCDFs were detected in some of the samples analyzed, the levels detected were lower than the corresponding blank sample detection limit. For example, a particular isomer of a PCDD was detected with 10 picograms of catch; however, the field blank reported

a nondetect with a detection limit of 20 picograms of catch. The variation of the detection limits is a function of the matrix being analyzed and the resolution of the analytical instruments used to quantify the samples.

Metal emissions (summarized in Table 11) were almost exclusively in the solid phase. Very little of the metals was found in the impingers of the sampling trains. The only significant vapor phase metals detected were calcium and mercury. A very volatile metal such as mercury is expected to be found in the vapor phase. Arsenic, copper, iron, lead, potassium, and zinc were in abundance in the stack gas in the solid phase. The copper in the stack gas is suspected to originate from the throat of the furnace and the torch electrode as copper was not present in high quantities in the feed soil. The presence of iron in the stack gas, at 66 ppm, was a consequence of the high levels of this element in the feed soil and the need for using a mild steel doughnut for start-up purposes. Arsenic, at approximately 6 ppm in the stack gas, and lead, at 4 ppm, were not retained in the treated soil as they are volatile metals (arsenic sublimates) and most probably evaporated from the soil while it was being treated in the furnace. Potassium was found in the stack gas because of its high initial levels in the feed soil (see TEST SOIL AND TREATED SLAG, below). The high level of zinc in the stack gas, at approximately 125 ppm, was a consequence of the high spiking level of this element and the high volatility of this metal in the temperature range encountered within the furnace. Additionally, the presence of chlorine with the zinc at this temperature range rapidly increases the volatility of zinc. In all cases (except mercury) the air pollution control system should have captured the metals. As shown later, the scrubber liquor did not contain high levels of metals after each of the tests.

Based on these results, it appears that not all of the volatile metals are captured in the molten soil at the completion of treatment. If this is the case, then these volatile metals should be captured by the gas treatment system (assuming it is correctly designed). A percentage of the volatile metals originally found in the soil would

appear in the scrubber liquor, therefore, possibly requiring treatment of the liquor prior to disposal.

## TEST SOIL AND TREATED SLAG

As stated previously, the feed soil consisted of a mixture of Silver Bow Creek Superfund Site soil, which is classified as a high metal-bearing soil, and 10% by weight No. 2 diesel oil. Into this mixture, zinc oxide and hexachlorobenzene were spiked to provide 28,000 ppm and 1,000 ppm, respectively. (The corresponding amount of zinc is 22,500 ppm zinc). Section 3, **TEST SOIL**, provides additional information regarding the preparation of the waste feed.

Analysis of the feed soil showed that it contained volatile compounds consistent with those associated with diesel fuel: benzene, toluene, ethyl benzene, and xylene (BTEX). These compounds were detected in the quantities presented in Table 8. The semivolatile compounds found most predominantly in the feed soil were the spiked hexachlorobenzene and 2-methylnaphthalene (see Table 8). Other diesel-based compounds were also found in the soil, but at levels that could not be accurately quantified by SW-846 Method 8270. Gas chromatography/mass spectrometry (GC/MS) analysis also indicated the presence of large numbers of TICs. These TICs are typical of those found in diesel oil and consisted mainly of compounds of naphthalene and benzene. The metals found most abundantly in the feed soil were aluminum, calcium, iron, potassium, sodium, and zinc.

Volatile organic analysis was not performed on the treated soil as no volatile compounds were considered to exist in the slag after it had reached its melting point temperature. The only semivolatile organic compounds found in the treated soil were low levels of two phthalate compounds which were probably sampling or analytical

contaminants. This agrees with the TCLP analysis of the slag discussed earlier in which no semivolatile compounds leached from the slag.

For the first Demonstration Test, 480 pounds of feed soil was fed to the furnace and a steel "doughnut" weighing 74.5 pounds was added. As the feed soil contained 10% organics, which appeared to have been destroyed, a calculated 506.5 pounds of slag should have been poured into the collection chamber. The mass of treated soil collected in the slag chamber was 277 pounds, the remainder being retained within the reaction chamber to provide a "skull" for the next test. (A skull is a layer of melted material around the interior of the primary chamber which reduces the chamber's volume and acts as an insulator to protect the refractory.) Therefore, a mass balance that yields meaningful results cannot be performed on this technology since a portion of material from each test can potentially remain in the reactor at the end of treatment. It is possible, though to compare the concentration of the inorganic elements in the feed soil with that of the collected slag, taking into account the destruction of the 10% organics (by weight) and assuming that none of the elements are concentrated in the poured slag. Table 12 gives the concentrations of the metals in feed and in the slag for all three of the tests. The feed is an average of all feed samples from the three tests. This table shows that a large percentage of the metals from the feed soil are retained within the vitrified slag. Exceptions to this trend are generally the volatile metals: arsenic, lead, mercury, and zinc. These volatile metals have been found, as stated earlier, to be exiting the system through the exhaust stack. In addition, some of these metals can be found in the scrubber liquor.

To evaluate the fate of the feed soil metals, a comparison can be made of the behavior of a non-volatile metal such as aluminum to that of a volatile metal such as zinc. Test 3 provides a good basis for examination since 600 pounds of waste were fed into the furnace and 595 pounds were poured. The feed soil utilized during Test 3 contained 29.6 pounds of aluminum. A total of 26.0 pounds were detected in the treated soil. This represents 88% of the aluminum originally present in the feed soil.

Traces of aluminum (0.00340 pounds total) were also detected in the stack gas. A small increase in the aluminum concentration from the pre-test scrubber liquor to the post-test scrubber liquor was also noted. The remainder of the aluminum which has not been accounted for may be due to sampling and analytical variation.

For zinc, 13.9 pounds were fed to the furnace in the feed soil during Demonstration Test 3. A total of 5.24 pounds (38%) was retained in the treated soil. The stack gas contained 0.332 pounds of zinc. A large portion of the zinc plated out as particulate matter in the blower and the long exhaust gas duct. Again, sampling and analytical

Table 12. Metals in the Demonstration Test Feed Soil and Treated Soil

Element	Average Feed Soil (ppm)	Treated Soil		
		Test 1 (ppm)	Test 2 (ppm)	Test 3 (ppm)
Aluminum	49,400 B	51,200	46,000	43,700
Arsenic	201	12.0 J	16.0 J	11.3 J
Barium	508	523	480	453
Calcium	12,500	28,800	20,500	18,000
Chromium	23.6 J	500	510	617
Copper	591	780	1,500	827
Iron	36,900 B	160,000	150,000	213,000
Lead	426	98.3 J	115 J	120 J
Magnesium	4,650	5,720	4,600	4,670
Manganese	814	1,850	1,900	2,570
Mercury	1.00	<0.133	<0.133	<0.133
Nickel	NA	270	265	287
Potassium	19,360	15,500	16,000	14,700
Sodium	10,200	8,180 B	8,650	7,830
Vanadium	77.3 J	59.8 J	59.5 J	50.0 J
Zinc	23,200	6,480	9,050	8,800

B Indicates that this compound was detected in a blank.

J Estimated result. Indicates that the result is less than the quantitation limit. The quantitation limit is defined and 5 times the detection limit

NA Not Analyzed

variation may have contributed, in part, to the apparent discrepancy between the zinc in the feed soil and the zinc in the treated soil and the stack gas.

The increase in iron concentration can be explained by the initial presence of the carbon steel doughnut. The concentrations of chromium and nickel both increase in the treated slag because, prior to the Demonstration Tests, stainless steel had been incorporated into the furnace to form part of the skull. The increase in the copper concentration is probably because of the melting of portions of the copper throat during treatment. In addition, soil treated prior to the Demonstration Tests, and hence part of the skull, had been high in calcium, manganese, and potassium.

Only the treated soil (not the feed soil) was analyzed for PCDDs and PCDFs. The levels of PCDDs and PCDFs in the treated soil were very low. However, as described earlier for the stack gas emissions, the detection limits for the blank samples were higher than the levels of PCDDs and PCDFs detected in the samples. It is therefore reasonable to conclude that no PCDDs or PCDFs were formed by the treatment process, and if any dioxins/furans were in the feed soil, they were destroyed by the intense heat of the process.

## **SCRUBBER LIQUOR**

The pre-test scrubber liquor for each of the three Demonstration Tests contained very little in the way of organic compounds. A metals scan on the pre-test scrubber liquor showed that, generally, only low levels of inorganic elements were present. This was expected since, prior to each test, the scrubber sump was flushed and filled with deionized water.

The post-test scrubber liquor did not contain any significant quantities of organic compounds. Nitrated compounds and phthalates were the only compounds present.

The nitrated compounds were most likely produced from the high levels of  $\text{NO}_x$  in the exhaust gas reacting with the water from the scrubber and any organic compounds present. The phthalates and volatile organic compounds probably entered the scrubber sump from the scrubber make-up liquor. The lack of organic compounds in the scrubber liquor and, as stated earlier, the absence of volatile or semivolatiles organic compounds in the exhaust gas, indicates that combustion of the organic compounds was complete.

The scrubbing unit was very inefficient in the capture of the inorganic compounds. The scrubber did capture some of the volatile metal elements but not at the levels that would typically be expected from a well-designed system. As stated previously, the exhaust gas contained a variety of metals that should have been captured by the scrubbing unit. The types of metals found in the scrubber liquor were similar to those found in the stack gas; that is, arsenic, iron, and zinc were the elements in abundance. High sodium levels found in the liquor were a consequence of the scrubber make-up (sodium hydroxide).

## CONTINUOUS EMISSION MONITORS

Throughout each of the three tests,  $\text{CO}$ ,  $\text{CO}_2$ ,  $\text{O}_2$ ,  $\text{NO}_x$ , and Total Hydrocarbons (THC) were monitored continuously to present a real time image of the combustion process and to determine if regulatory standards were being exceeded. Figures 4 through 8 present CEM plots for Demonstration Test 3, typical of those generated throughout the demonstration.  $\text{SO}_2$  was also measured, but the data collected was not considered suitable. High levels of  $\text{NO}_x$  in stack gas are known to interfere with  $\text{SO}_2$  meters, and the interference indicates the presence of  $\text{SO}_2$  when none is actually present.



PPMV CARBON MONOXIDE

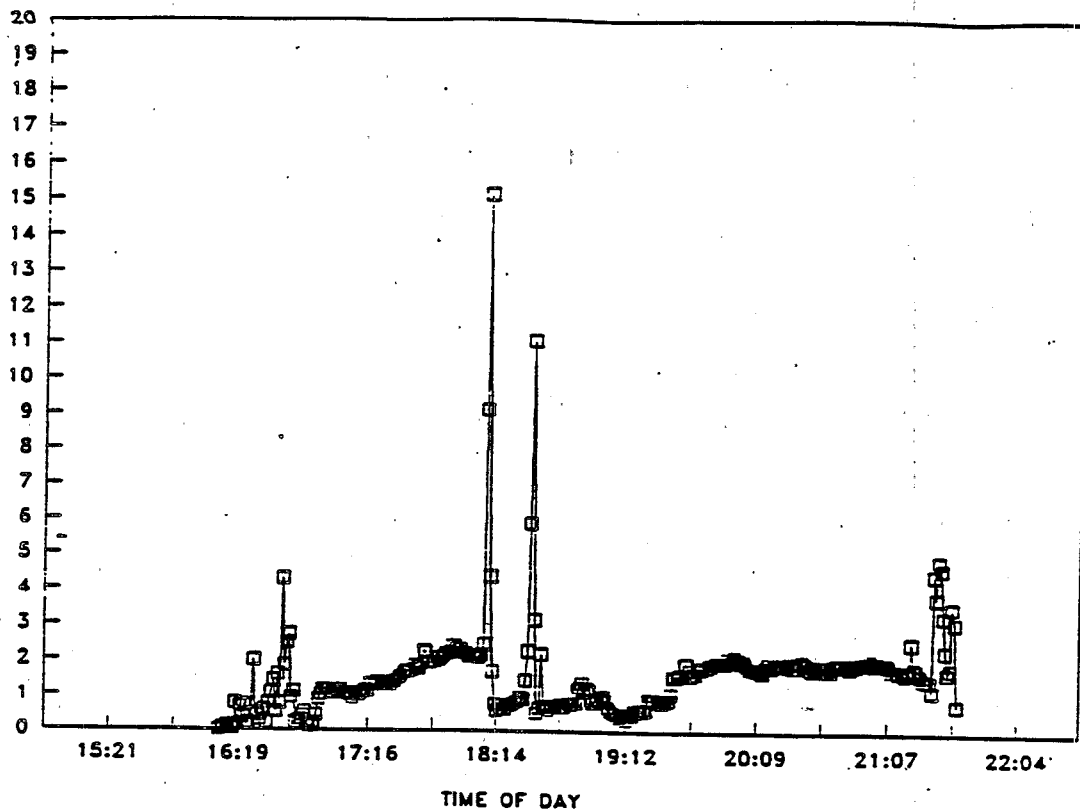


Figure 4. CO Plot for Demonstration Test 3

PERCENT CARBON DIOXIDE

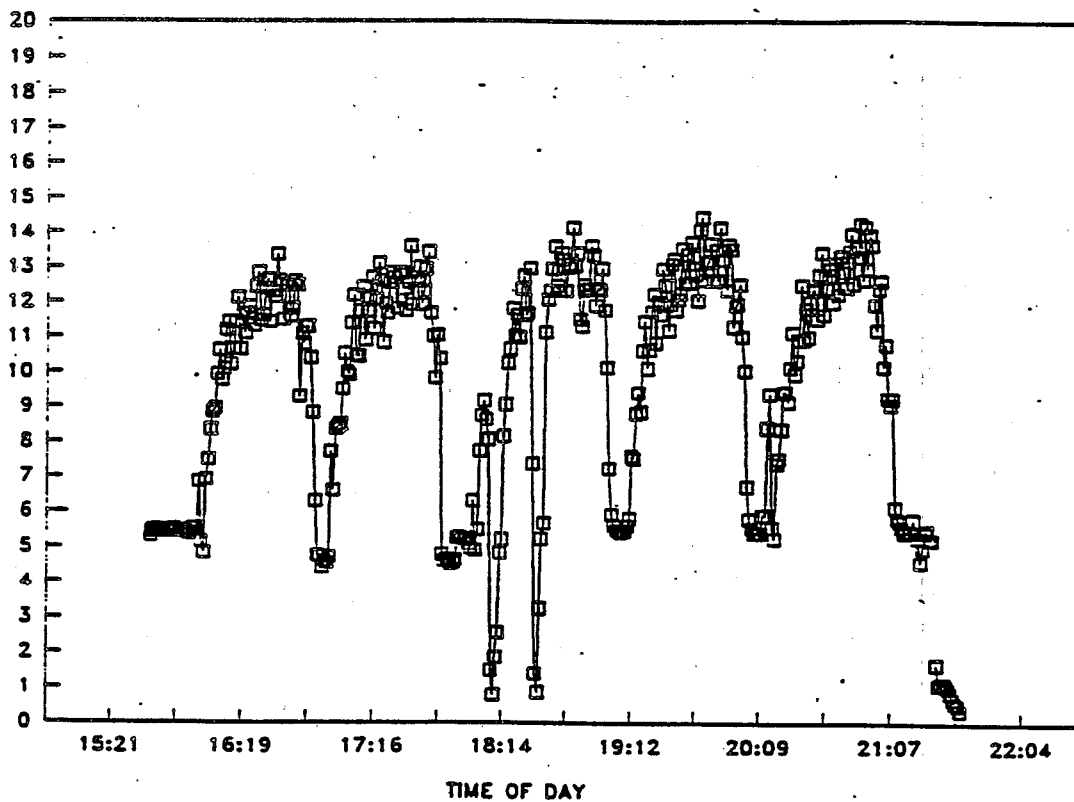


Figure 5. CO<sub>2</sub> Plot for Demonstration Test 3

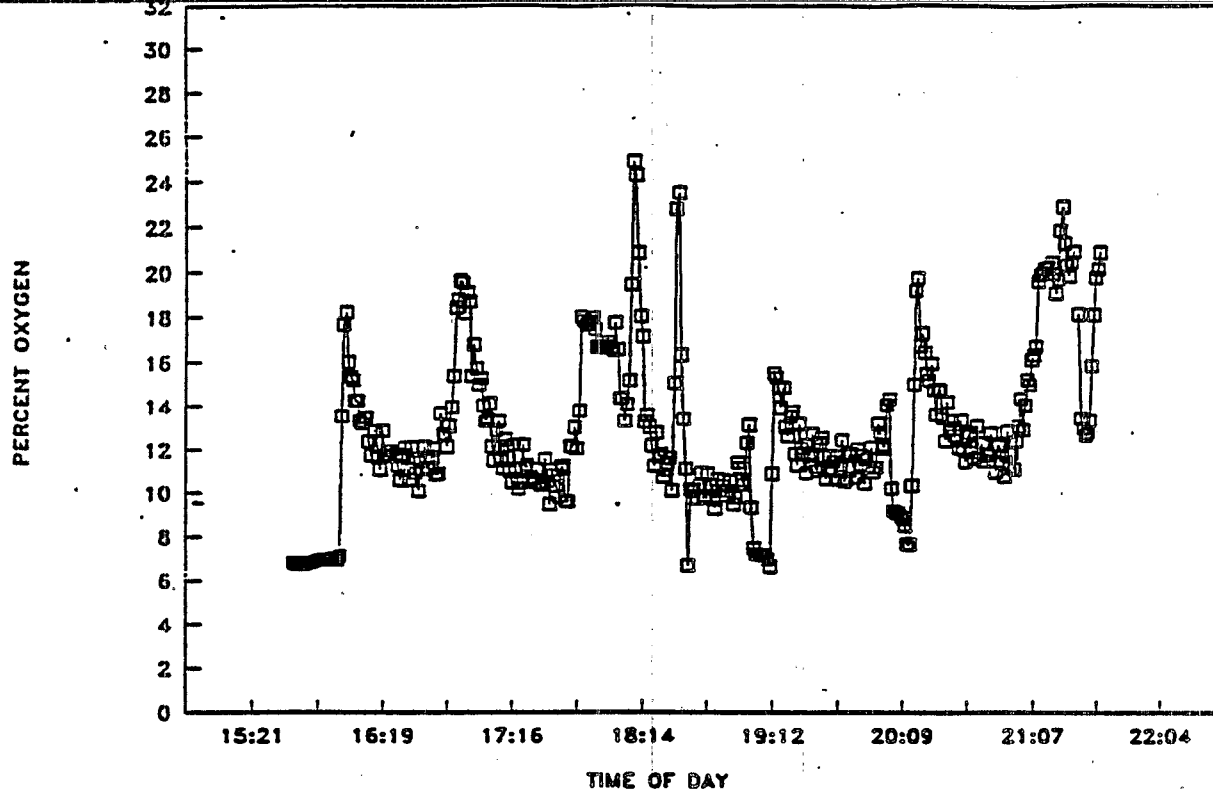


Figure 6. O<sub>2</sub> Plot for Demonstration Test 3

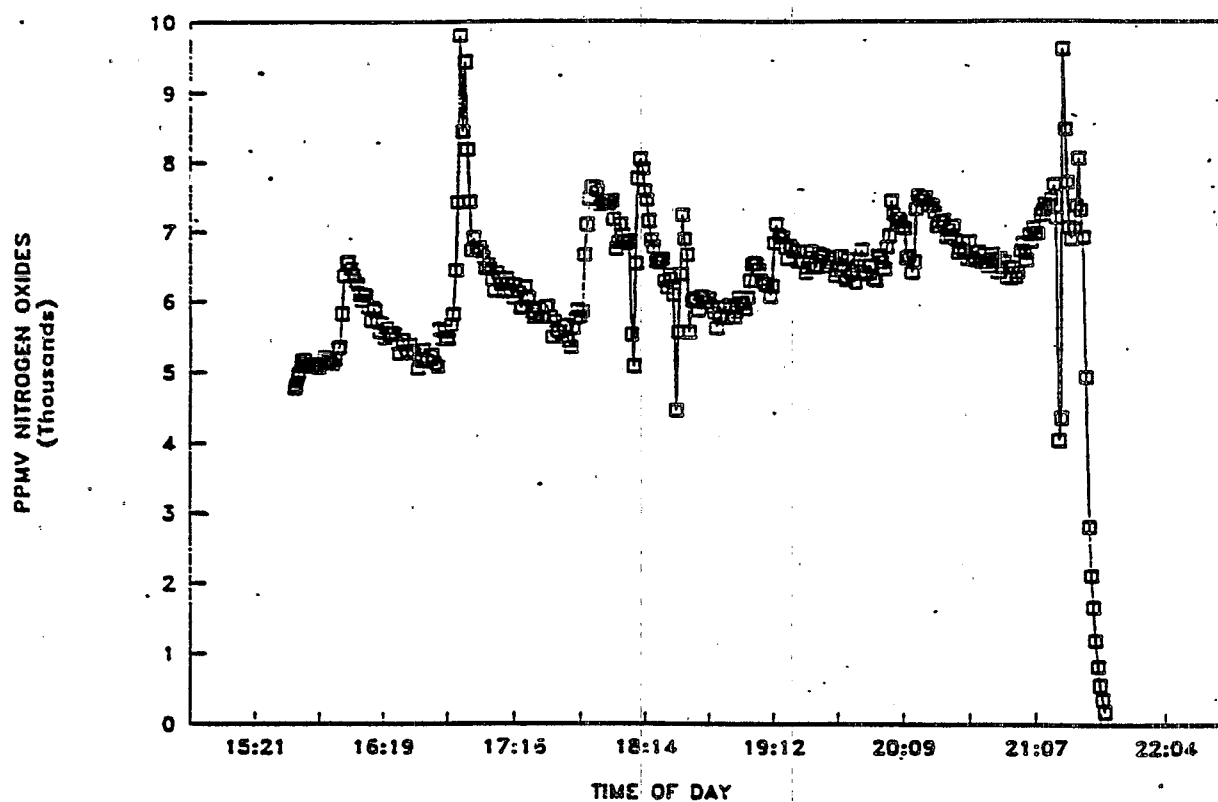


Figure 7. NO<sub>x</sub> Plot for Demonstration Test 3

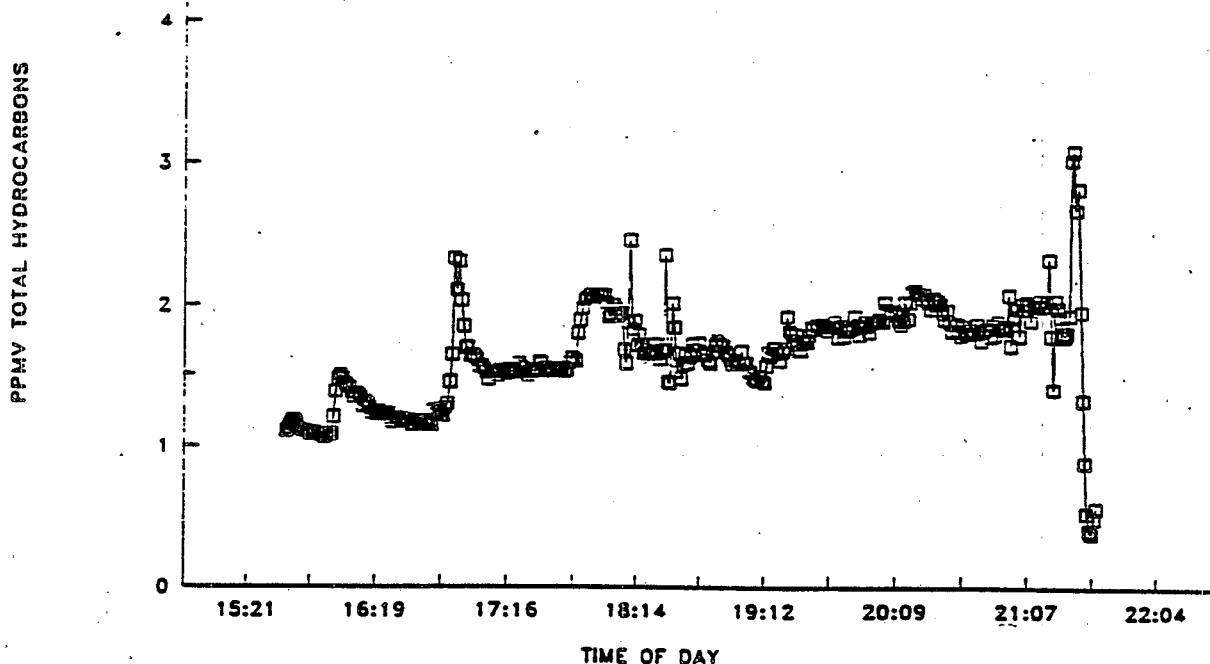


Figure 8. THC Plot for Demonstration Test 3

Since the installation of the afterburner in the secondary combustion chamber the level of Total Hydrocarbons exiting the system has been low ( $<4$  ppm) even with at least 10% organics in the feed soil. This gives a good indication that effective thermal destruction of the organic compounds is occurring. Another indication of the ability of the process to treat organic contaminated media is the low levels of CO in the exhaust (approximately 1.4 ppm) and the level of CO<sub>2</sub> (approximately 8%). O<sub>2</sub> monitors showed significant variation throughout the treatment process as pure O<sub>2</sub> was fed to the primary chamber at approximately 18 scfm while waste was being fed to the furnace.

High levels of NO<sub>x</sub> are a consequence of this process if air is used as the torch gas. The torch gas passes through the extremely hot arc of the plasma, thus oxides of nitrogen are readily formed. Testing to date, has shown that the average concentration of NO<sub>x</sub> in the stack gas is approximately 5,000 ppm (uncorrected to 7% O<sub>2</sub> as explained earlier). The oxygen lance operated intermittently rather than continuously during Tests 1 and 3. During Test 2, however, the oxygen was fed at a steady rate over the entire treatment time, so the NO<sub>x</sub> values corrected to 7% O<sub>2</sub>

may easily be calculated, if desired. This correction is not required by RCRA regulations because the system operates under oxygen enrichment. The average uncorrected NO<sub>x</sub> value during Test 2 was 5,467 ppm; the average NO<sub>x</sub> value corrected to 7% O<sub>2</sub> was 8,514 ppm.

The uncorrected average NO<sub>x</sub> value for testing (5,000 ppm) corresponds to an emission rate of 2.5 lbs/hr (based on operation 365 days/yr, 24 hrs/day). Federal requirements state that the NO<sub>x</sub> emission rate must be less than 9.2 lbs/hr (40 tons/yr based on operation 365 days/yr, 24 hrs/day). The PCF does not exceed this 9.2-lbs/hr standard, but the concentration in the stack gas is high. The emission rate is low because of the low flowrate of the exhaust gas (approximately 110 scfm). This flowrate is dependent on the size of the torch used. The feed rate of the soil does not influence the level of NO<sub>x</sub> in the exhaust assuming the same torch is used for the different feed rates. This is because the torch uses the same amount of torch gas regardless of the soil feed rate. If a torch larger than that used in the Demonstration Tests is to be employed, then the use of a NO<sub>x</sub> reduction technology should be investigated.

## **FURNACE OPERATION**

Since all three Demonstration Tests were designed to be identical in nature, operating conditions during the tests were relatively constant. These operating conditions are described in detail in Section 4, **FIELD OPERATIONS DOCUMENTATION**. The feed material was identical in each case, a mixture of Silver Bow Creek soil and 10% by weight No. 2 diesel oil, spiked to provide 28,000 ppm zinc oxide (22,500 ppm zinc) and 1,000 ppm hexachlorobenzene. The feed rate for each test was 120 lbs/hr. Although the mass of material to be fed during each test was anticipated to be 960 pounds, the actual weight of the feed was 480, 360, and 600 pounds for Tests 1, 2, and 3, respectively. The corresponding weight of the treated soil generated during

the tests was 277, 265, and 595 pounds. The difference between the mass fed and the mass poured during each test can be accounted for by the retention of material inside the chamber as part of the skull as described earlier in this section. As demonstrated by these values, the skull was progressively built up throughout the course of the Demonstration Tests.

The torch power ranged from an average of approximately 410 kW during Test 3 to nearly 460 kW during Tests 1 and 2. The total power consumption of the torch ranged from 3,308 kWh (Test 1) to 4,720 kWh (Test 3). As anticipated, the total power consumption for Test 3 was greater than the other two tests because of the extent of its duration. The torch gas in each case was air with a flowrate of 23 to 24 scfm.

The furnace is operated so that a minimum temperature of 2,100°F is achieved in the primary reaction chamber and a minimum temperature of 1,800°F is reached in the afterburner before feeding of the waste is initiated. The reactor chamber temperature, once it stabilized, achieved an average value of approximately 2,250°F. The afterburner temperature averaged around 1,800°F (slightly higher during Test 3) once the system reached operating range. The off-gas flowrate was maintained at approximately 110 scfm during all three Demonstration Tests.

The scrubber liquor generated during each of Tests 1 and 2 was close to 150 gallons. During Test 3, this value was greatly exceeded due to frequent blowdowns of the scrubber in an attempt to reduce particulate loading on the blower downstream. Nearly 800 gallons of scrubber liquor were generated during Test 3.

The PCF-6 is a high maintenance process that is subject to frequent stoppage because of equipment failure. During the first Demonstration Test, a stoppage occurred when a scrubber sump pump overheated and tripped the system. The test was also shortened because of particulate build-up in the exhaust blower. This same problem

caused the second Demonstration Test to be completed prior to treatment of all the feed. While warming up the primary chamber for the third Demonstration Test, the torch developed a cooling water leak. This led to the furnace being out of operation for approximately three hours while the leak was repaired. Experience of operating the PCF-6 has shown that secondary arcing within the primary chamber is the most common form of equipment breakdown. Torch cooling water leaks result when this occurs, and the torch ram needs to be welded, plugging these pinhole leaks. With respect to preventive maintenance, torch electrode replacement is the most regular of the procedures that need to be carried out to ensure uninterrupted operation. The electrodes must be replaced approximately every 60 to 100 hours of furnace operation.

The plasma torch provides a substantial amount of thermal energy to the feed, however, to protect the equipment from being damaged by this heat, cooling circuits are utilized. For the PCF-6 the cooling circuits that remove the highest percentage of the heat from the process are the torch cooling circuit (31%), the primary chamber cooling circuit (39%), and the scrubber unit (10%). For optimum operation of the furnace, it would be anticipated that the majority of the heat removed from the system would be from the collection chamber and the scrubbing unit. For the Demonstration Tests, the specific energy consumption was approximately 8 kWh/lb. Physical data indicates that, ideally, a specific energy requirement for melting soils is approximately 0.3 kWh/lb. Therefore, there is considerable room for design and operational improvement of the PCF system.

## **SECTION 7**

### **CONCLUSIONS**

A number of conclusions have been drawn based on this SITE Demonstration Test. These conclusions have been briefly addressed in the **EXECUTIVE SUMMARY** (Section 1) and are discussed in detail in this section.

#### **LEACHABILITY OF THE TREATED SOIL FOR INORGANIC COMPOUNDS**

The PCF process is designed to encapsulate inorganic compounds in a vitrified slag and render the treated soil non-leachable. The Toxicity Characteristic Leaching Procedure (TCLP) was performed on both the feed soil and the treated slag to determine if the process can effectively bind inorganic compounds into the treated soil. The feed soil was tested to establish initial values for the leachability of inorganic and organic compounds. The vitrified slag underwent TCLP to meet testing objectives. For inorganics, the feed soil only exhibited significant leachability characteristics for: calcium (175 mg/L), sodium (1,475 mg/L), and the spiked zinc (982 mg/L). Sodium, was not selected as a tracer compound since it is a weakly dissociable metal and thus behaves differently from typical metals. The treated soil did not show strong leachability for any metals except sodium which leached at approximately the same level as in the feed soil. Both tracer metals, calcium and zinc, showed significant reductions in leaching properties as a result of treatment.

Overall, testing activities demonstrated that the process can effectively bind inorganic compounds into the treated soil.

#### **LEACHABILITY OF THE TREATED SOIL FOR ORGANIC COMPOUNDS**

The only organic compounds that were found to be leachable from the feed soil for the Demonstration Tests were naphthalene and 2-methylnaphthalene. Although the feed soil was spiked with high levels of hexachlorobenzene (1,000 ppm), it did not leach from the soil. No organic compounds were found to leach from the treated slag.

#### **DESTRUCTION AND REMOVAL EFFICIENCY OF TARGET ANALYTES**

The Destruction and Removal Efficiency (DRE) is based on the concentration of the target analyte in the feed soil and the stack gas. For the Demonstration Tests the mean level of hexachlorobenzene, based on all the feed soil samples for the three Demonstration Tests, was 972 ppm. The 95% confidence interval for the estimated mean of the hexachlorobenzene spiked into the feed soil was 864 to 1,080 ppm. No hexachlorobenzene was detected in the stack gas in any of the three tests, therefore, all hexachlorobenzene DREs were determined based on the detection limit from the appropriate test. The estimated average DRE for each of the three Demonstration Tests, based on the confidence interval of the feed soil and on the analytical instruments' detection limits for the stack gas analysis, were >99.9976%, >99.9991%, and >99.9999%, respectively.

The concentration of 2-methylnaphthalene, a semivolatile compound, was found to be between 390 and 526 ppm in the feed soil. Again, none of this compound was detected in the stack gas for any of the Demonstration Tests, leading to estimated



average DREs, based on instrument detection limits, of >99.9906%, >99.9964%, and >99.9996%, for Demonstration Tests 1, 2, and 3, respectively.

Total xylenes, volatile compounds, were found in the feed soil with a 95% confidence interval of 128 and 139 ppm. This led to an estimated average DRE of total xylenes, for all tests, based on instrument detection limits, of >99.993%. The reasoning for presenting only one DRE for a volatile compound is given in Section 6.

Overall, the PCF process appears to be very efficient in destroying both volatile and semivolatile compounds when both the primary reaction chamber and the afterburner are operating.

## **STACK GAS EMISSIONS**

### **HCl and Particulate Matter Emissions**

HCl emissions were very low for all three tests. Measured HCl emission rates for the Demonstration Tests ranged from 0.0007 to 0.0017 lbs/hr. The chlorine concentration in the feed soil for Test 1 was 0.066%. This leads to a HCl removal efficiency of 98.5%. The removal efficiency may not be meaningful because of the low chlorine input. However, it appears that if the feed contained a higher concentration of chlorine, then a HCl removal efficiency of 99% could be achieved.

The average particulate emissions to the atmosphere for each of the three Demonstration Tests was 0.374 grains/dscf. This exceeded the RCRA regulatory limit of 0.08 grains/dscf [40 CFR (07/01/91 Edition) §264.343(c)].

## Volatile and Semivolatile Organic Compound Emissions

Small quantities of volatile and semivolatile compounds were formed as products of incomplete combustion (PICs) in the plasma furnace. The most dominant semivolatile compound released in the stack gas was benzoic acid, at an average concentration of approximately 4 ppm. The occurrence of benzoic acid in the stack gas is to be expected as both toluene (from the diesel oil) and chlorine (from the hexachlorobenzene) were present in the feed soil. These two compounds, with the addition of heat, readily form benzoic acid as discussed in Section 6. Low levels of nitrated compounds were found in the stack gas at low levels ( $< 0.3$  ppm). These compounds were formed because of the high levels of  $\text{NO}_x$  and trace quantities of organic compounds in the stack gas interacting with the scrubber liquor spray. The volatile compound found in the greatest abundance in the stack gas was benzene, at approximately 19 ppbv. Benzene and substituted benzenes are prevalent in many forms throughout diesel oil in the feed, and hence benzene is a readily formed PIC.

## Dioxins/Furans Emissions

Sampling and analysis for polychlorodibenzodioxins (PCDDs) and polychlorodibenzofurans (PCDFs) in the exhaust gas stream was accomplished during the Demonstration Tests. The results of these analyses indicated that no PCDDs or PCDFs were formed in the stack gas. Although some PCDDs and PCDFs were detected in some of the samples analyzed, the levels detected were lower than the corresponding blank sample detection limit.

## Metal Emissions

The only significant vapor phase metals detected were calcium and mercury. A very volatile metal such as mercury is expected to be found in the vapor phase. Arsenic,

copper, iron, lead, potassium, and zinc were in abundance in the stack gas in the solid phase. Lead, at approximately 4 ppm in the stack gas, and arsenic, at 6 ppm, were not retained in the treated soil as they are volatile metals (arsenic sublimates) and most probably evaporated from the soil while it was treated in the furnace. Potassium was found in the stack gas because of its high initial levels in the feed soil. The high level of zinc in the stack gas, at approximately 125 ppm, was a consequence of the high spiking level of this element and the high volatility of zinc in the temperature range encountered within the furnace. With the exception of mercury, the air pollution control system should have captured the metals.

The Demonstration Tests results show that not all of the volatile metals were captured in the molten soil at the completion of treatment. If this is the case, these volatile metals should be captured by a correctly designed gas treatment system.

## **AIR POLLUTION CONTROL SYSTEM**

### **Particulates Captured by the Air Pollution Control System**

The amount of particulates captured by the air pollution control system was extremely small. This is demonstrated, in part, by the low level of scrubber solids present in the sump. There was less than 0.5% total solids in the scrubber sump tank. The high particulate loading during the Demonstration Tests caused substantial build-up of particulate matter in the exhaust blower downstream of the air pollution control system.

The Silver Bow Creek Superfund Site soil was extremely dry and dusty, both before and after it was mixed with diesel oil. The mixed feed soil was very free-flowing with no standing liquid. It is possible that the fine particles of this dusty feed may not have been retained in the primary reaction chamber in the melted soil and simply

passed through both the treatment process and the scrubbing unit to the exhaust blower and stack. A well-designed scrubbing system should be capable of capturing the particulates.

#### Organics Captured by the Air Pollution Control System

The pre-test scrubber liquor for each of the three Demonstration Tests contained very little organics. This was expected since, prior to each test, the scrubber sump was flushed and filled with deionized water. The post-test scrubber liquor did not contain any significant quantities of organic compounds. Only nitrated compounds and phthalates were present in the scrubber liquor. The nitrated compounds were most likely from the high levels of  $\text{NO}_x$  in the exhaust gas reacting with the water from the scrubber and any organic compounds present. The phthalates and volatile organic compounds probably entered the scrubber sump from the scrubber make-up liquor or in the case of phthalates were possibly a laboratory contaminant. The lack of organic compounds in the scrubber liquor and the absence of volatile or semivolatile organic compounds in the exhaust gas, indicates that combustion of the organic compounds was complete.

#### Inorganics Captured by the Air Pollution Control System

The scrubbing unit was very inefficient in capturing inorganic compounds. Only small quantities of inorganic compounds, mainly volatile metals, were captured by the scrubber, and not at the levels that would be expected from a well-designed system. The metals found in the scrubber liquor were similar to those found in abundance in the stack gas; that is, arsenic, iron, and zinc. High sodium levels found in the liquor were a consequence of the scrubber make-up (sodium hydroxide).

## CONTINUOUS EMISSION MONITORS

Throughout each of the Demonstration Tests, CO, CO<sub>2</sub>, O<sub>2</sub>, NO<sub>x</sub>, and Total Hydrocarbons (THC) were monitored continuously to present a real time image of the combustion process and to determine if regulatory standards were being exceeded. SO<sub>2</sub> was also measured but the data collected was not considered suitable. High levels of NO<sub>x</sub> in a stack gas are known to interfere with SO<sub>2</sub> meters, and the interference indicates the presence of SO<sub>2</sub> when none actually exists. During the Demonstration Tests, the THC exiting the system was low (<4 ppm), even with 10% organics in the feed. The exhaust gas contained low levels of CO (approximately 1.4 ppm) and a level of approximately 8% CO<sub>2</sub>. These levels of THC, CO, and CO<sub>2</sub> give a good indication that effective thermal destruction of the organic compounds is occurring.

Oxygen monitors showed significant variation throughout the treatment process as pure O<sub>2</sub> is fed to the primary chamber at approximately 18 scfm while waste is being fed to the furnace. High levels of NO<sub>x</sub> are a consequence of this process if air is used as the torch gas, as it was during the Demonstration Tests. The torch gas passes through the extremely hot arc of the plasma, thus oxides of nitrogen are readily formed. NO<sub>x</sub> emission rates during the Demonstration Tests averaged approximately 5,000 ppm (2.5 lbs/hr). However, because of the low stack gas flowrates (110 scfm) the total emissions of NO<sub>x</sub> were below the regulatory requirements of 9.2 lbs/hr (40 tons/yr based on operation 365 days/yr, 24 hrs//day). If a torch larger than that used in the Demonstration Tests is to be employed, then the use of a NO<sub>x</sub> reduction technology should be investigated.

## **SYSTEM PERFORMANCE AND RELIABILITY**

The components of the PCF can be broken down into two main categories; the thermal treatment section and the gas clean-up system. The furnace unit demonstrated that it was entirely capable of processing the waste feed, however, the gas clean-up system did not perform up to expectations. In fact very little particulate matter or organic or inorganic compounds were found in the scrubber sump at the conclusion of the tests.

The entire system is a high maintenance unit. During the course of the Demonstration Tests, the exhaust gas blower failed twice (because of the high particulate loading in the flue gas), the torch developed a deionized water leak, and numerous preventive maintenance activities took place. The on-line factor for the process during the Demonstration Tests was 70%.

## **COST OF COMMERCIAL OPERATION**

Several cost scenarios can vary the unit cost of operation for the furnace. The cost of operation is strongly dependent on two factors: the on-line factor and the feed rate. The present configuration of the feeder, furnace, and slag collector allows an average feed rate of 120 lbs/hr. However, feed rates of 500 and 1,000 lbs/hr could be achieved with a few minor furnace modifications. A larger Plasma Centrifugal Furnace is operational in Switzerland that can process contaminated soil at a feed rate of 2,200 lbs/hr. If the PCF used during the Demonstration Tests is operated at a feed rate of 500 lbs/hr, with an on-line factor of 70% and a total treatment volume of 2,000 tons, then it is estimated that the cost per ton of this technology is \$1,816/ton of contaminated waste treated. For the larger-scale PCF, operating at a feed rate of 2,200 lbs/hr, and the same on-line factor and total treatment volume, the cost is

estimated at \$757/ton. Although the on-line factor for the Demonstration Tests was 70%, a more realistic on-line factor would be approximately 60%.

## **SECTION 8**

### **QUALITY ASSURANCE**

Quality Assurance (QA) may be defined as a system of activities whose purpose is to provide to the producer or user of a product or a service with assurance that it meets defined standards of quality with a stated level of confidence. A QA program is a means of integrating the quality planning, quality assessment, quality control (QC), and quality improvement efforts of various groups in an organization and to enable operations to meet user requirements at an economic level. Included are all actions taken by personnel, and the documentation of laboratory performance as specified in the Quality Assurance Project Plan (QAPP). The QA program is an essential part of a sound analytical protocol used by individuals and laboratories to detect and correct problems in a measurement process or to demonstrate statistical control. The objective of this quality assurance program is to reduce measurement errors to agreed-upon limits and to produce results of acceptable and known quality.

#### **INTRODUCTION**

To achieve testing objectives (see Section 1) and to obtain data of known quality, a detailed QAPP was prepared for this Demonstration Test. The QAPP specified the necessary guidelines to ensure that the measurement system was in control. The QAPP also detailed information on the process measurements as well as the analytical



approach to ensure data of high quality could be obtained to verify vendor claims and achieve project objectives. Certain key measurements were used to determine the leachability of the treated soil and the Destruction and Removal Efficiencies (DREs) for the organic material. Measurements designated as critical for evaluating leachability and DREs (previously defined as numerical objectives for the demonstration) consisted of semivolatile organic compounds in the feed soil, semivolatiles in the stack gas, along with semivolatile and metal TCLP measurements for the treated soil. Specifically, hexachlorobenzene and zinc were spiked into the feed soil to evaluate these parameters. In addition to the above critical measurements, other parameters which were designated as secondary critical measurements included metals, semivolatiles, dioxins/furans, particulates, HCl/chlorine, carbon monoxide, for all matrices and semivolatiles and metals TCLP for the feed soil. Other non-critical parameters included carbon dioxide, oxygen, nitrogen oxides, sulfur dioxide, total hydrocarbons, higher heating value, bulk density, moisture, and volumetric flow. Each of these measurements, along with their respective QC data is discussed in the following sections.

The QAPP outlines several modifications made to standard analytical methods. These modifications were made to allow collected data to be evaluated in terms of project objectives. Modifications were made to surrogate spike compounds, matrix spike compounds, and target analyte lists, along with sample preparation methods. These deviations are presented in the following sections and discussed in detail later in Section 8 under **"MODIFICATIONS AND DEVIATIONS FROM THE QAPP."** During the Demonstration Test, there were no modifications or deviations made pertaining to the collection of field data.

As part of the QAPP, audits of both the field and laboratory operations were performed. The intent of these audits was to ensure that measurement techniques were performed in accordance with the guidance set forth in the QAPP. Results of these audits are discussed in this section under **"AUDIT FINDINGS."**

The following sections describe procedures used to determine the quality and usability of the measured data obtained for this Demonstration Test.

## PROCEDURES DEFINING DATA QUALITY CONTROL AND USABILITY

To assess the quality of the data generated for this Demonstration Test, two important data quality indicators are of primary concern: precision and accuracy. Precision can be defined as the degree of mutual agreement characteristics of independent measurements as the result of repeated applications of the process under specified conditions. Accuracy is the degree of agreement of a measured value with the true or expected value.

Precision is measured by matrix spike and sample duplicates. In most cases precision was evaluated by expressing, as a percentage, the difference between results of matrix spike or sample duplicates for a single parameter. The relative percent difference (RPD) was calculated as:

$$RPD = \frac{\text{Maximum Value} - \text{Minimum Value}}{[(\text{Maximum Value} + \text{Minimum Value})/2]} \times 100\%$$

For data sets with greater than two points, the coefficient of variance (CV) or the relative standard deviation (RSD) were used to assess precision. The CV (or RSD) can be calculated as:

$$CV \text{ (or RSD)} = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

To determine the agreement between a measured value and the true value, accuracy was measured. Accuracy can be expressed as the relative error (%RE) calculated as:

$$\%RE = \frac{\text{Measured Value} - \text{Theoretical Value}}{\text{Theoretical Value}} \times 100\%$$

To determine the accuracy of several organic and inorganic parameters, known quantities of analytical and/or surrogate standards were spiked into selected samples. Equipment used to provide data for this project were tested for accuracy through the analysis of calibration check standards and laboratory control samples. To determine the recovery of these spikes, the following equation was used:

$$\% \text{ Recovery of Spike} = \frac{C_{ss} - C_{us}}{C_{sa}} \times 100\%$$

where:  $C_{ss}$  = analyte concentration in spiked sample

$C_{us}$  = analyte concentration in unspiked sample

$C_{sa}$  = analyte concentration added to sample

During the course of the sample analyses, several samples were spiked to determine the matrix effect on analyte recoveries. These samples were selected at random by the analytical laboratory performing the work.

Another important aspect of assessing data quality is completeness. Completeness is a measure of the amount of valid data produced from the total effort compared to the total amount of data originally planned for the project. Specific completeness objectives were delineated in the QAPP for all measurements.

For this demonstration, the completeness objectives were met for all analytical parameters. Some samples had poor surrogate recoveries or other QC indicators outside QAPP control limits; however, most of these data points are still useable and are discussed in greater detail in the following sections. There were only a few minor losses of data due to problems associated with poor quality control indicators. In addition, minor losses of data were also due to laboratory preparation of samples and breakage of sample containers during shipment. Some of the semivolatile feed soil samples were analyzed two days beyond their specified extract holding times which presented another possible loss of data. Reviewing the QC information and the data associated with these samples, however, indicates that these data are still useable for this Demonstration Test. (See discussion later in this section under "SAMPLE HOLDING TIMES.")

To determine if a measurement is valid, it must be reproducible and comparable. Comparability expresses the extent with which one data set can be compared to another. To generate comparable results, standard methods, which are widely accepted, along with strict analytical protocols were used. These methods were clearly specified in the QAPP and reviewed and approved before data or samples were collected. Methods that were non-standard, such as the Multiple Metals Train (MMT), were also cited in the QAPP and the appropriate analytical protocol were appended. This allowed the results generated by this test to be reproducible by other investigators using these methods.

Although several precautions were taken to generate data of known quality through the control of the measurement system, the data must also be representative of true conditions. Representativeness refers to the degree with which analytical results accurately and precisely represent actual conditions present at locations chosen for sample collection. During the development of the QAPP and the analysis of the samples, great care was taken to ensure that the samples were representative of actual process conditions. Specific sampling methods are discussed in Section 5.

Of critical concern was the sampling of the treated soil. Since the treated soil was poured from the furnace as a molten mass, it was not possible to collect a well-mixed grab sample. To obtain a representative sample, the molten mass was allowed to cool and harden into a monolith. A diamond-tipped drill bit was used to cut 2" cores from random locations in the monolith. When the cores were removed, they were collected for homogenization. In the field, the collected cores were smashed with a sledge hammer between cotton cloth to reduce their size. The shattered fragments were thoroughly mixed and placed into appropriate sample containers for shipment to the laboratory. In the laboratory, the sample was further reduced in size by grinding the fragments in a mechanical mortar and pestle. Once the material was in a powder-like form, aliquots were collected for specific analyses.

#### **ANALYTICAL QUALITY CONTROL**

Quality control was measured in each of the analytical parameters through the use of surrogate spikes, matrix spikes, blanks, and field and laboratory duplicates. Surrogate spikes are added to the matrices before extraction and provide an indication of the extraction efficiency. Matrix spikes provide an indication of how well target analytes can be recovered in sample matrices. Matrix spike and laboratory duplicates provide information on laboratory precision. Blanks provide an indication of contamination that may have been introduced to the samples in the field, during shipment, and during laboratory preparation. Field duplicates also provide information concerning sample representativeness. Good agreement among field duplicates indicates that the matrix sampled is homogeneous and that representative samples have been collected. In addition to these QC parameters, criteria was placed on the analytical laboratory to ensure that the results were performed using acceptable methodologies as outlined in the QAPP. For each measured parameter, internal QC procedures were designed to control and assess the performance of the analytical system. The following sections discuss the results of these QC indicators for each analytical parameter.

Each of the following sections addresses the accuracy and precision associated with all measurement parameters. As previously noted, completeness objectives were satisfied for every parameter.

### Volatile Organics

Volatile organic samples were collected from the feed soil, scrubber liquor before and after each run, and the scrubber makeup. In addition, volatile emissions were analyzed by the collection of volatile organic sample train (VOST) samples as discussed in Section 5. Samples of the feed soil were collected from the 5-gallon feed pails during the loading of the feeder. Liquid samples were collected from the scrubber sump by allowing water to purge the samples lines. Once the line was purged, the flow was diverted into standard 40-mL VOA vials. These samples were analyzed using SW-846 Method 8240. This method uses a purge-and-trap analyte concentration procedure prior to analysis by gas chromatograph/mass spectrometry (GC/MS).

As outlined in the QAPP, each day the GC/MS was operated, a mass axis calibration was performed and the spectral fragmentation pattern was checked through the use of bromofluorobenzene (BFB). The BFB was required to pass the criteria specified in the method. The instrument was calibrated for the target volatiles through the analysis of standards across five concentration ranges. Each day, this calibration was checked to ensure that instrument conditions did not change and that the initial calibration data was valid. Calibrations were required to pass criteria for system performance check compounds and calibration check compounds as specified by SW-846 Method 8240. Calibrations were verified every 12 hours. All samples were spiked with internal standard compounds to provide accurate quantitations relative to the calibration standards. Internal standard recoveries were acceptable for each

analysis. Each sample was also spiked with surrogate standards to monitor the purging efficiency of the sample. Spike data is summarized in the following sections.

#### **Feed Soil Volatiles**

Volatile organics in the feed soil was designated as a secondary critical parameter for this demonstration. Specifically, xylene concentrations were used in the same manner as semivolatiles in the feed soil when determining DREs for evaluating project objectives. Due to the high levels of target analytes in the feed soil, the samples were analyzed using the SW-846 Method 8240 medium level protocol. This entailed extracting a known quantity of the sample with a known volume of methanol. A portion of the methanol extract was then analyzed using conventional purge-and-trap procedures. Surrogate recoveries for the feed soil volatile organic samples are summarized in Table 13. As noted in this table, all surrogate recoveries met the specified recovery control limits.

Matrix spikes for the feed soil were not performed because of a mistake made on the chain of custody records sent to the analytical laboratory. In viewing the results of all of the feed soil samples, it can be seen that there is very good agreement among these results (see Appendix D). The standard deviation for analyte concentrations were well within QAPP precision specifications. These data were expected to show good agreement because the feed soil was homogenized prior to sampling. In addition, surrogate spike recoveries, as noted above, were within specified control limits. Therefore, it is believed that the absence of matrix spikes has a limited impact on data quality and does not adversely affect project objectives.

Two sets of duplicate samples of the feed soil were collected and analyzed during the first test run. Results of these duplicates are presented in Table 14. This table summarizes the results of the detected compounds. Compounds that do not appear on the table were not detected. The feed contained high levels (ppm quantities) of

Table 13. Volatile Surrogate Summary Data

Compound	No. of Results <sup>a</sup>	Mean Recovery (%)	Std. Dev. (%)	Min (%)	Max (%)	No. of Results Outside Limits		Control Limits (%)
						Low	High	
Feed Soil								
1,2-Dichloroethane-d <sub>2</sub>	17	78	4	74	89	0	0	70 - 121
1,4-Bromofluorobenzene	17	101	4	92	107	0	0	74 - 121
Toluene-d <sub>8</sub>	17	97	3	88	101	0	0	81 - 117
Liquors <sup>b</sup>								
1,2-Dichloroethane-d <sub>2</sub>	13	82	5	79	96	0	0	76 - 114
1,4-Bromofluorobenzene	13	103	3	99	109	0	0	86 - 115
Toluene-d <sub>8</sub>	13	99	3	96	104	0	0	88 - 110

- <sup>a</sup> Number of field samples and field duplicates. (Does not include matrix spikes, blanks, and recovery checks.)
- <sup>b</sup> Includes one scrubber make-up water sample.

Table 14. Feed Soil Volatile Duplicate Sample Results (Test 1)

Description	Compound	Primary Value (ug/kg)	Duplicate Value (ug/kg)	RPD	Objectives
					Precision RPD
1 <sup>st</sup> Set	Benzene	680	1,000 J	38	50
	1,2-Dichloroethane	.	1,700 J	NC	50
	Ethyl Benzene	22,000	23,000	4	50
	Methyl Ethyl Ketone	1,100 J	.	NC	50
	Methylene Chloride	3,300 JB	3,700 JB	11	50
	Toluene	16,000	17,000	6	50
	Xylenes	130,000	130,000	0	50
2 <sup>nd</sup> Set	Benzene	940 J	910 J	3	50
	Ethyl Benzene	24,000	21,000	13	50
	Methylene Chloride	2,000 JB	.	NC	50
	Toluene	18,000	17,000	6	50
	Xylenes	140,000	120,000	15	50

- .
 Indicates that this compound was not detected.
- B
 Indicates that this compound was detected in a blank.
- J
 Estimated Result. Indicates that this compound was detected below the quantitation limits. The quantitation limit is defined as 5 times the detection limit.
- NC
 Not calculated.



organic compounds that are typical of diesel fuels. The feed soil samples show excellent agreement between samples. The compounds methylene chloride, 1,2-dichloroethane, and MEK were detected in one sample but not the others. This was most likely caused by the introduction of laboratory contamination or values detected near the detection limit. As noted in Table 14, RPD values were not calculated for these parameters.

Field blanks collected for the feed soil for the first two tests were clean with the exception of low levels of methylene chloride. The field blank for Test 3 contained benzene at 12  $\mu\text{g/kg}$ , chlorobenzene at 19  $\mu\text{g/kg}$ , and some methylene chloride. These low levels have no significant impacts on data quality. A complete summary of the volatile feed soil data is presented in Appendix D.

The only volatile compound used for computing DRE were total xylenes because of their high concentration in the feed soil. This is seen in a review of the project conclusions (see Section 7). Xylenes were well within precision objectives as shown in Table 14 and because all surrogates were within specified objectives for all samples, only data of known and sufficient quality were used for evaluating this conclusion in relation to stated project objectives.

#### **Volatile Scrubber Water Samples**

Scrubber water samples (a secondary critical measurement) were collected and analyzed per the procedures outlined in the QAPP with no modifications. Results of these samples indicated little or no significant volatile compounds were detected.

Scrubber samples were spiked with surrogate standards before analysis. These results are also presented in Table 13. For the scrubber liquor samples, all surrogates were recovered within the control limits.

As with the feed soil, matrix spikes were not performed in this matrix because of an error on the chain of custody records. Matrix spikes for these samples would have been of limited use since there were no significant levels of volatile target compounds found in these samples. Matrix spikes would have indicated potential recovery problems associated with specific target analytes, however, surrogate spikes showed reasonable recoveries indicating no recovery problems for general chemical compound classification. The absence of matrix spikes is therefore not believed to adversely affect the quality of data for these samples. In addition, no critical project conclusions were based upon data from scrubber samples. Specifically, scrubber water data were only used in supporting emission data for evaluation of the emission control system.

Duplicate samples of the scrubber water were collected and analyzed before and after each Demonstration Test. These data are presented in Table 15. The scrubber water samples are also in good agreement for the compounds that were detected. In general these samples were very clean with little or no target analytes detected. There are some variations that yield high RPD values for methylene chloride. Methylene chloride has also been identified as a laboratory contaminant which probably attributed to the poor precision of this compound in these samples.

The field blank collected for the scrubber liquor contained low levels of toluene, MEK, and methylene chloride. A sample of the scrubber make-up was collected and analyzed in the unlikely event that unexplained volatile results were obtained in the scrubber. No target compounds were detected except MEK at 44  $\mu\text{g/L}$ , methylene chloride at 1.6  $\mu\text{g/L}$ , and toluene at 1.6  $\mu\text{g/L}$ . These results have no impact on scrubber volatile data quality.

#### **Volatile Organic Sampling Train Gas Samples**

Samples of the gas stream were collected using the volatile organic sampling train (VOST) to determine the types and quantity of volatile organic emissions generated

**Table 15. Scrubber Water Volatile Duplicate Sample Results**

Compound	Primary Value (µg/L)	Duplicate Value (µg/L)	RPD	Objectives	
				Precision	RPD
Test 1					
PRE-TEST					
Chlorobenzene	*	3.4 J	NC		30
Methyl Ethyl Ketone	31 J	33 J	6		30
Methylene Chloride	1.8 JB	20 B	167		30
POST-TEST					
Methylene Chloride	44 B	16 B	93		30
Test 2					
PRE-TEST					
Methyl Ethyl Ketone	1600	1800 J	12		30
Methylene Chloride	1.1 JB	14 JB	171		30
Xylenes	1.6 J	*	NC		30
POST-TEST					
Methyl Ethyl Ketone	200	260	26		30
Methylene Chloride	15 B	5.4 B	94		30
Test 3					
PRE-TEST					
Methylene Chloride	22 B	6.2 B	112		30
POST-TEST					
Benzene	0.85 J	0.88 J	3		30
Chloroform	2.4 J	2.3 J	4		30
Dibromomethane	*	0.78 J	NC		30
Methyl Ethyl Ketone	1.3 J	*	NC		30
Methylene Chloride	26 J	22 J	17		30
Toluene	0.51 J	0.63 J	21		30

\* Indicates that this compound was not detected.

B Indicates that this compound was detected in a blank.

J Estimated Result. Indicates that this compound was detected below the quantitation limit. The quantitation limit is defined and 5 times the detection limit.

NC Not calculated.

during the Demonstration Tests. These data are considered as secondary critical measurements used primarily for evaluating emission levels and to determine if PICs were being formed. Total xylenes, as previously noted, were used in determining DREs as stated in the conclusions and these data were considered critical. VOST samples were collected under the guidance of SW-846 Method 0010. For each of the Demonstration Tests, several VOST samples were collected. The normal sampling procedure calls for the collection of 20 L of sample to be collected for each Tenax® pair. To ensure that the samples did not become overwhelmed and saturate the resin, several pairs were collected at various sample volumes (see Section 5) during each Demonstration Test. In addition, before the melt was poured into the pig, a fresh set of Tenax® traps was installed to measure any sudden release of volatile contaminants during the pouring process. When these samples were returned to the laboratory, they were analyzed in accordance with Methods 5041 and 8240. Similar procedures, were used to calibrate and tune the mass spectrometer for VOST analysis. Calibration of the instrument was performed by using a flash volatilization technique. Here, standards were loaded onto a clean set of Tenax® traps per Method 5041 specifications. These standards were then desorbed and analyzed using the same procedures employed while analyzing samples.

Surrogate recoveries for VOST analyses are summarized in Table 16. As noted in this table, there were some poor surrogate recoveries that were not within specified control limits. It is not possible to re-run VOST samples once they have been desorbed from their Tenax® cartridges. In reviewing the raw data, it can be noted that several of the poor recoveries occurred for Test 3. This could have been caused by chromatographic interference. Sample SAIC-390, 10-L duplicate, had very poor recovery for all of the surrogates. This would indicate a potential problem with this sample. Some samples contained two surrogates that were not within the control limits. Data from these samples may be in question due to poor desorption or analytical interference. Samples that contain one surrogate out of the control window have not been significantly affected. Although some surrogates were not

Table 16. Surrogate Recoveries for VOST Analyses

Compound	No. of Results <sup>a</sup>	Mean Recovery (%)	Std Dev (%)	Min (%)	Max (%)	No. of Results Outside Limits		Control Limits (%)
						Low	High	
1,2-Bromofluorobenzene	24	71	47	4	139	8	0	50 - 150
1,2-Dichloroethane-d <sub>4</sub>	24	75	24	6	107	2	0	50 - 150
Toluene-d <sub>8</sub> <sup>b</sup>	23	90	56	3	300	4	1	50 - 150

<sup>a</sup> Number of field samples and field duplicates. (Does not include matrix spikes, blanks, and recovery checks.)

<sup>b</sup> Recovery of toluene-d<sub>8</sub> was not calculable in one of the VOST samples due to interferences.

recovered well in some of the VOST tubes, it does not appear that this seriously affects VOST data quality because the quantities detected in the VOST samples do not contribute significantly to process emissions.

One set of method spikes (MS and MSD) was performed for the VOST analyses. This was done by spiking a blank VOST tube and then performing the analysis as specified by Method 5041. These results are summarized in Table 17. Accuracy and precision objectives were not specified in the QAPP for these samples, however, the results indicate the precision for this matrix was acceptable for each compound except toluene which had a RPD of 38 percent. Accuracy was acceptable for these spikes with the exception of high recoveries for benzene (about 135 percent) and low chlorobenzene recoveries (60 and 51 percent).

Table 17. VOST Method Spike Results

Compound	Spike 1 % Recovery	Spike 2 % Recovery	RPD
Benzene	138	133	4
Chlorobenzene	60	51	16
1,1-Dichloroethene	103	98	5
Toluene	117	80	38
Trichloroethene	109	111	2

Duplicate VOST samples were collected during Test 3. This was accomplished by installing a second VOST into the exhaust duct. Samples were collected throughout the duration of the test. The analytical results of these duplicates are presented in Table 18. A total of six duplicate pairs were collected for this run. No specific control limits for precision were placed in the QAPP for the VOST duplicates. The precision for most compounds exceeded the generally accepted guidance of 30 percent RPD. Benzene appeared to have higher RPD values than most other compounds. Specific reasons for this discrepancy are not clear. Values for methylene chloride also exhibit high RPDs in some samples which were most likely due to laboratory contamination. Although there was high variability associated with these duplicate results, it does not appear that this seriously affected VOST data quality because the quantities detected in the VOST samples did not contribute significantly to process emissions as noted by the project conclusions. If, in the worst case, the VOST values are off by a factor of 2, volatile emissions would still be considered irrelevant and would not impact project objectives. In addition, no xylenes were detected in the stack gas and therefore DRE calculations are not affected.

Field blanks were collected on each run day. Field blanks (FB) were clean with the exception of low levels of methylene chloride detected in the FB for Test 3. Eight mg of 1,1,1-trichloroethane were detected in the Test 2 FB. Trip blanks were analyzed for Test 1 and Test 2. No detectable levels of volatile contaminants were found in these samples. These blank results did not have any significant impact on data quality. A complete presentation of all of the VOST data is presented in Appendix C.

For two 10-L primary and duplicate samples (SAIC-0391 and SAIC-0392) and a 20-L primary sample (SAIC-0130), only the Tenax®/charcoal tubes were analyzed because the front-half cartridges broke during shipment. The back half cartridge is used if high levels are encountered and breakthrough occurs in the first Tenax® tube. Since very few compounds were detected at significant levels in any of the other samples, and a total of 30 samples split among the 3 tests were analyzed, loss of data is expected

Table 18. VOST Duplicate Sample Results (Test 3)

Description	Compound	Primary Value (ppbV)	Duplicate Value (ppbV)	RPD
1 <sup>st</sup> Pair	Benzene	3.36	2.48	30
	Chloroform	0.23	0.26	12
	Methylene Chloride	*	0.35	NC
	Toluene	0.52	*	NC
	1,1,1-Trichloroethane	0.20	0.17	16
2 <sup>nd</sup> Pair	Benzene	4.91	9.86	67
	Chloroform	3.11	4.01	25
	Methylene Chloride	0.24	0.33	32
	Toluene	*	0.70	NC
	1,1,1-Trichloroethane	0.12	0.11	9
3 <sup>rd</sup> Pair	Benzene	9.94	7.59	27
	Chloroform	1.04	0.70	39
	Methylene Chloride	*	0.38	NC
	Toluene	*	1.32	NC
	1,1,1-Trichloroethane	0.23	0.22	4
	Vinyl Chloride	3.13	*	NC
4 <sup>th</sup> Pair	Benzene	4.05	36.47	160
	Chloroform	2.78	6.26	77
	Ethyl Benzene	*	1.14	NC
	Methylene Chloride	1.09	0.52	71
	Tetrachloroethene	*	0.28	NC
	Toluene	0.50	6.84	173
	1,1,1-Trichloroethane	0.39	0.22	56
	Trichloroethene	*	0.36	NC
	Xylenes	*	1.28	NC

(Continued)

Table 18. (Continued)

Description	Compound	Primary Value ppbV	Duplicate Value ppbV	RPD
5 <sup>th</sup> Pair	Benzene	25.44	37.39	38
	Carbon Tetrachloride	*	0.32	NC
	Chloroform	8.50	9.48	11
	Methylene Chloride	1.02	2.97	98
	Toluene	3.09	2.99	3
	1,1,1-Trichloroethane	0.51	0.44	15
6 <sup>th</sup> Pair	Benzene	116.31	23.59	133
	Chloroform	6.43	3.93	48
	Methylene Chloride	3.57	7.49	71
	Toluene	6.97	4.91	35

\* - Indicates that this compound was not detected.

NC - Not calculated

to be minimal even though these 3 samples were not analyzed. Loss of these three samples does not impact project completeness objectives.

#### Volatile Organic Gas Canister Samples

As back-up stack gas sample for volatile organics, gas canister samples were collected in the event that the VOST tubes became saturated. These canisters were collected in accordance with EPA Compendium Method TO-14. There were very few compounds that exceeded the instrument calibration during the VOST analyses (benzene and chloromethane in two samples). None of the detected compounds saturated the GC/MS detector. A preliminary review of the VOST data indicated that the analysis of canisters was not required and therefore, as stated in the QAPP these analyses were not performed.



## Semivolatile Organics

Semivolatile organic samples were collected from several locations for each of the Demonstration Tests. The semivolatile analyses for the feed soil and the stack gas were designated as critical parameters for this demonstration and were used to determine DREs. All other matrices were secondary critical measurements. Semivolatile samples were collected for the feed soil, treated soil, scrubber liquor before and after each test, the scrubber make-up, and the stack gas. Below is a discussion of each of these analyses.

### **Feed Soil Semivolatiles**

Samples of the feed soil were collected from each 5-gallon pail that was fed to the unit during each of the Demonstration Tests. A small metal scoop was used to collect approximately 100 g of soil from several locations within each of the 5-gallon pails. This sample was placed into a glass jar for compositing. After the addition of each scoop, the jar was agitated by hand to facilitate mixing. At the end of the Demonstration Tests, appropriate portions of the composite sample were sent to the laboratory for semivolatile analyses. To ensure that a representative sample was studied, 60 grams of the composite sample was extracted using SW-846 Method 3550. Since the samples contained high levels of diesel and hexachlorobenzene, special care was required to bring the target analytes to within the detection limits of the instrument without exceeding the calibration range. Sample extracts were "cleaned" to remove possible interferences of the diesel fuel by gel permeation chromatography (GPC) and neutral alumina prior to analysis. Because of the high levels of target compounds in the samples, surrogate spiking was performed at similar levels with three surrogate compounds: 2-fluorobiphenyl, methylnaphthalene-d<sub>10</sub>, and nitrobenzene-d<sub>5</sub>. These surrogates were selected because of their chemical similarities to the compounds of interest in the feed. In addition, consideration as to the amount of spike needed and the high concentration required, was also a factor in selecting

these project specific surrogates. The usual SW-846 Method 8270 surrogates added at typical concentrations would not satisfy project QC objectives. Inclusion of these three surrogates was approved prior to the demonstration.

Following extraction, the extracts were analyzed in accordance with SW-846 Method 8270. Hexachlorobenzene was not recovered in the samples after the neutral alumina clean-up. The fraction of the extract that was run through GPC, but not neutral alumina, did give good recoveries of hexachlorobenzene. Hence only this fraction was used to provide all semivolatile results for this study.

Each day samples were analyzed, the mass spectrometer was mass axis calibrated using PFTBA. Following this, DFTPP was analyzed to check the spectral fragmentation patterns of the GC/MS. The spectrum generated was required to pass the ion abundance criteria presented in the QAPP before sample analyses could begin. The mass spectrometer was then calibrated with a multipoint calibration curve for each of the target compounds specified in the QAPP criteria. This calibration curve was required to pass minimum response factor criteria for the SPC compounds and stability criteria for the CCC. For this demonstration, hexachlorobenzene was added to the routine list of CCCs. This would help ensure a stable response for this critical compound. Every 12 hours the mass spectrometer's calibration was checked by analyzing a single standard and comparing the response to that of the multipoint calibration curve. The instrument also was required to pass the DFTPP tuning criteria. Each sample was spiked with internal standards for quantitation of target compounds. Internal standard recoveries were monitored and were required to remain within established laboratory control limits.

Surrogate results for the feed soil samples are presented in Table 19. It may be noted by reviewing this table that the surrogate recoveries for these samples are generally good. Feed sample 2, Test 3, exhibited high recoveries for all three surrogates. The average recovery for these surrogates was 150 percent. In viewing the raw data, it

Table 19. Semivolatile Feed Sample Surrogate Recoveries

Compound	No. of Samples <sup>a</sup>	Mean Recovery (%)	Std. Dev. (%)	Min. (%)	Max. (%)	No. of Results Outside Limits		Control Limits (%)	
						Low	High		
2-Fluorobiphenyl	15	89	21	70	155	0	1	30	115
1-Methylphtalene-d <sub>10</sub>	15	86	23	57	159	0	1	50	150
Nitrobenzene-d <sub>5</sub>	15	64	25	39	135	0	1	23	120

<sup>a</sup> Number of routine field samples and field duplicates, not including blanks, matrix spikes, and recovery checks.

appears that the quantitations are about 1.5 times higher than the average of the other feed samples. These data, therefore, were not used for the calculation of DREs.

It appears that the incorrect amount of internal standard was added to this extract causing mis-quantitation of detected compounds. No other limitations for these data are indicated by these surrogate recoveries.

Because of the high levels of hexachlorobenzene and diesel fuel oil in the feed samples, it was not possible to perform a matrix spike using normal procedures outlined in SW-846 Method 8270. It was not feasible to spike all of the matrix spike compounds at levels high enough to be detected after diluting the extract. Since hexachlorobenzene was one of the semivolatile compounds designated as critical in the QAPP for these Demonstration Tests, it was spiked (at 1,000 ppm) into Silver Bow Creek tailing soil that was previously mixed with No. 2 diesel oil and spiked with zinc oxide but no hexachlorobenzene. The matrix spike results for the feed soil are presented in Table 20. As noted by the data presented in this table, the results of

Table 20. Feed Soil Matrix Spike Results (Test 1)

Field ID	Compound	% Recoveries			RPD	Objectives	
		MS	MSD	Mean		Accuracy (% Rec.)	Precision (RPD)
SAIC-159/160 MS	Hexachlorobenzene	123	116	120	5.9	Det. - 152	50

these spikes are satisfactory. In addition to matrix and surrogate spikes, a single sample was to be extracted twice to obtain extraction efficiency information on this matrix. For the target analytes, this double extraction was proposed because of the highly complex matrix and because only hexachlorobenzene could feasibly be spiked into the feed soil. While this procedure was performed by the laboratory, both extracts were inadvertently combined and run as a single sample rather than analyzed separately. However, because most of the hexachlorobenzene spike was recovered in the feed samples, and because all samples had similar levels of hexachlorobenzene with very little difference in concentration, the lack of these additional extraction results has not greatly impacted data quality.

Replicate samples were collected during each test run. For each test, the QAPP specified that three primary and two duplicate samples be collected. Since the same feed stock of material was used for each test, and each sample was an aliquot of the feed composite, it was not possible to distinguish between a specific primary and duplicate sample. Selecting a primary and a random duplicate sample from a specific run could bias precision and accuracy information. Results for the feed soil samples are presented in Table 21. Data presented in this table is evaluated two ways. The first part presents test-specific information pertaining to each of the five aliquots from the composite feed sample for each test. The summary information provides an evaluation of the statistics of all three Demonstration Tests. These results indicate good agreement among the detected compounds. This indicates that the soil was well-mixed and homogeneous samples were collected.

Field blanks collected on each run day were clean. The results from the laboratory extraction blanks were also clean. Data quality was therefore not impacted by contamination introduced in the field or the laboratory for these samples. A complete presentation of these sample results is presented in Appendix D.

Table 21. Duplicate Semivolatile Feed Sample Results

Compound	Sample 1 ( $\mu\text{g/kg}$ )	Sample 2 ( $\mu\text{g/kg}$ )	Sample 3 ( $\mu\text{g/kg}$ )	Sample 4 ( $\mu\text{g/kg}$ )	Sample 5 ( $\mu\text{g/kg}$ )	Mean	Std. Dev.	RSD
Test 1								
Acenaphthene	*	*	16 J	*	*	NC	NC	NC
Dibenzofuran	*	*	*	*	13 J	NC	NC	NC
Fluorene	*	*	36 J	*	37 J	NC	NC	NC
Hexachlorobenzene	850	750	990	900	900	878	88	1.14
2-Methylnaphthalene	470	480	440	380	440	442	39	8.82
Naphthalene	140	120	130	110	130	126	11	9.05
Phenanthrene	60 J	60 J	65 J	55 J	60 J	60	4	5.89
Test 2								
Dibenzofuran	*	*	14 J	20 J	*	NC	NC	NC
Fluorene	*	*	36 J	36 J	*	NC	NC	NC
Hexachlorobenzene	1100	1000	950	950	800	960	108	11.29
2-Methylnaphthalene	420	400	420	400	370	402	20	5.10
Naphthalene	140	140	160	130	120	138	15	10.75
Phenanthrene	60 J	60 J	65 J	70 J	60 J	63	6	9.78
Test 3								
Acenaphthene	17 J	**	*	*	*	NC	NC	NC
Anthracene	9.7 J	**	*	*	*	NC	NC	NC
Dibenzofuran	16 J	**	15 J	16 J	12 J	15	2	12.83
Fluorene	43 J	**	45 J	46 J	30 J	41	7	18.14
Hexachlorobenzene	980	**	1100	930	780	948	133	13.98
2-Methylnaphthalene	490	**	310	550	430	445	102	23.03
Naphthalene	170	**	180	170	130	163	22	13.65
Phenanthrene	72 J	**	74 J	68 J	54 J	67	9	13.46
Summary	Mean		Standard Deviation		RSD			
Hexachlorobenzene	927		107		11.57			
2-Methylnaphthalene	429		59		13.69			
Naphthalene	141		21		15.13			
Phenanthrene	63		6		9.66			

\* Indicates that this compound was not detected.

\*\* Sample results not used because of high surrogate recoveries as noted previously.

B Indicates that this compound was detected in a blank.

J Estimated Result. Indicates that this compound was detected below the quantitation limit. The quantitation limit is defined as 5 times the detection limit.

NC Not calculated.

## Semivolatile Treated Soil

Once the molten mass had cooled, samples of the treated soil were collected for semivolatile analyses. Samples were obtained by using a drill core as described in Section 5 and earlier in this section. These samples were sent to the laboratory for further size reduction and extraction using SW-846 Method 3540. In addition to the core samples, water used to cool the drill bit was sampled and analyzed with the treated soil samples. These cooling water samples were collected to measure any losses of semivolatile organic material due to the drilling. These liquid samples were extracted using SW-846 Method 3520. No modifications were required to either extraction procedure because it was anticipated that these samples would contain little or no semivolatile organic material. Each sample was spiked with the normal surrogates required for SW-846 Method 8270. All of the samples were analyzed using SW-846 Method 8270. The quality control procedures; tuning, calibration, etc, utilized for these extracts are described later in this section.

Table 22 presents the surrogate results for these samples. Included in this table are the surrogate results of the cooling water samples. The results of the surrogates are excellent. All of the recoveries are within the specified limits with the exception of

Table 22. Semivolatile Treated Soil Surrogate Recoveries

Compound	No. of Results*	Mean Recovery (%)	Std. Dev. (%)	Min. (%)	Max. (%)	No. of Results Outside Limits		Control Limits (%)
						Low	High	
2-Fluorobiphenyl	8	90	6	80	98	0	0	30 - 115
2-Fluorophenol	8	89	7	73	94	0	0	25 - 121
2,4,6-Tribromophenol	8	94	7	85	104	0	0	19 - 122
Nitrobenzene-d <sub>5</sub>	8	89	6	77	98	0	0	23 - 120
Phenol-d <sub>5</sub>	8	91	8	75	97	0	0	24 - 113
Terphenyl-d <sup>14</sup>	8	103	15	77	121	0	1	18 - 137

\* Number of routine field samples and field duplicates, not including blanks, matrix spikes, and recovery checks.

one high recovery of terphenyl-d<sub>14</sub> (188%) in the cooling water sample that was collected before the water was subjected to drilling.

Results of the matrix spikes performed on the treated soil are presented in Table 23. As indicated by these results, there does not appear to be a recovery problem associated with this matrix.

**Table 23. Semivolatile Treated Soil Matrix Spikes**

Field ID	Compound	% Recoveries			RPD	Objectives	
		MS	MSD	Mean		Accuracy (% Rec.)	Precision (RPD)
SAIC-0220 MS	1,2,4-Trichlorobenzene	97	103	100	6.0	38-107	23
SAIC-0220 MS	1,4-Dichlorobenzene	82	83	83	1.2	28-104	27
SAIC-0220 MS	2-Chlorophenol	99	94	97	5.2	25-102	50
SAIC-0220 MS	2,4-Dinitrotoluene	98	93	96	5.2	28-89	47
SAIC-0220 MS	4-Chloro-3-methylphenol	101	86	94	16.0	26-103	33
SAIC-0220 MS	4-Nitrophenol	78	92	85	16.5	11-114	50
SAIC-0220 MS	Acenaphthene	88	93	91	5.5	31-137	19
SAIC-0220 MS	N-Nitrosodipropylamine	89	76	83	15.8	41-126	38
SAIC-0220 MS	Pentachlorophenol	84	98	91	15.4	17-109	47
SAIC-0220 MS	Phenol	87	84	86	3.5	26-90	35
SAIC-0220 MS	Pyrene	101	112	107	10.3	35-142	36
SAIC-0221 MS	1,2,4-Trichlorobenzene	94	85	90	10.1	38-107	23
SAIC-0221 MS	1,4-Dichlorobenzene	82	70	76	15.8	28-104	27
SAIC-0221 MS	2-Chlorophenol	96	84	90	13.3	25-102	50
SAIC-0221 MS	2,4-Dinitrotoluene	107	105	106	1.9	28-89	47
SAIC-0221 MS	4-Chloro-3-methylphenol	98	99	99	1.0	26-103	33
SAIC-0221 MS	4-Nitrophenol	84	90	87	6.9	11-114	50
SAIC-0221 MS	Acenaphthene	90	89	90	1.1	31-137	19
SAIC-0221 MS	N-Nitrosodipropylamine	86	88	87	2.3	41-126	38
SAIC-0221 MS	Pentachlorophenol	85	87	86	2.3	17-109	47
SAIC-0221 MS	Phenol	88	79	84	10.8	26-90	35
SAIC-0221 MS	Pyrene	100	104	102	3.9	35-142	36

Duplicate samples were collected and analyzed for each test run. Duplicate results for the semivolatile treated soil samples are not presented here because there were no detectable levels of semivolatile target compounds present in the primary or duplicate samples. There were some low level phthalate compounds detected, but they were qualified as laboratory blank contaminants. The core water samples also had some low level phthalate contamination which were suspected to be from laboratory contamination with no other target compounds detected. The core water samples indicate that there was no loss of semivolatile target compounds during the drilling process.

### **Semivolatile Scrubber Water Samples**

Samples of the scrubber water were collected before and after each Demonstration Test. These samples were collected to evaluate any accumulation of semivolatiles in the scrubber liquor and were considered secondary critical measurements. These samples were collected from a tap in the scrubber sump using prescribed procedures. These samples were extracted using SW-846 Method 3520. The extracts were analyzed under instrument conditions outlined later in this section.

Pre-demonstration testing indicated that the generally accepted surrogates for SW-846 Method 8270 were not recovered properly in the scrubber liquor samples. It was postulated that high levels of  $\text{NO}_2$  in the exhaust gas was nitrating the acid surrogates, resulting in poor recoveries of the acid spike compounds. To compensate for these poor recoveries, three additional acid surrogates were selected for spiking in this matrix and in the MM5 resin samples:  $^{13}\text{C}$ -pentachlorophenol, 2,4-dinitrophenol- $\text{d}_3$ , and 4,6-dinitro-2-methylphenol- $\text{d}_2$ . These compounds were selected and pre-approved because they exhibited satisfactory recoveries in pre-demonstration test activities and because they were highly substituted phenolic compounds which were less subject to nitration. Recovery criteria that was placed on these compounds are based on the non-labeled analogs recovery criteria presented in SW-846 Method



8270. The surrogate recoveries are presented in Table 24 for the scrubber water semivolatile samples. It should be noted that the soil matrix originally proposed for this demonstration contained significant quantities of pentachlorophenol, anticipating the use of this compound for determining DREs. Because a different soil matrix was ultimately chosen, and the primary compound of interest was hexachlorobenzene, acid surrogate recoveries were consequently less important.

Reviewing the data presented in this table indicates that the base/neutral surrogates were recovered very well from this matrix. These were critical for project conclusions when evaluating DREs for hexachlorobenzene and 2-Methylnaphthalene. Recoveries for the base/neutral surrogates that were outside control limits exceeded the recovery range. There is, however, a wide scatter of results for the acid surrogates. As anticipated, the routine 8270 surrogates were not recovered well in this matrix, with the exception of 2,4,6-tribromophenol. The most probable reason for this is that the large bromine atoms prevent the  $\text{NO}_x$  from attacking the phenolic ring of this molecule. Other acid surrogates showed marginal to good recoveries. Recovery

Table 24. Scrubber Water Semivolatile Surrogate Recoveries

Compound	No. of Results*	Mean Recovery (%)	Std. Dev. (%)	Min. (%)	Max. (%)	No. of Results Outside Limits		Control Limits (%)
						Low	High	
2-Fluorobiphenyl	12	74	14	52	95	0	0	43 - 116
2-Fluorophenol	12	16	29	0	83	10	0	21 - 100
2,4-Dinitrophenol- $\text{d}_3$	12	120	70	0	223	3	2	D - 191
2,4,6-Tribromophenol	12	38	37	0	103	3	0	10 - 123
4,6-Dinitro-2-methylphenol- $\text{d}_2$	12	85	41	0	117	2	0	D - 181
$^{13}\text{C}$ -Pentachlorophenol	12	56	35	0	95	4	0	47 - 176
Nitrobenzene- $\text{d}_5$	12	91	15	75	130	0	1	35 - 114
Phenol- $\text{d}_5$	12	42	28	0	84	10	0	10 - 94
Terphenyl- $\text{d}_{14}$	12	110	20	92	155	0	1	33 - 141

\* Number of routine field samples and field duplicates, not including blanks, matrix spikes, and recovery checks.  
D - Detected.

check samples and method blanks indicated satisfactory recoveries of the special acid surrogates and the normal acid surrogates. These results indicate that acid compounds may not have been accurately quantitated or detected in the scrubber water matrix. The scrubber sump is maintained at a high pH with sodium hydroxide to neutralize acid gases. The results indicate that the scrubber solution, along with the NO<sub>2</sub> gases produced by the process, have an impact on acid compounds. As indicated by the results there were very few detectable SW-846 Method 8270 compounds found in the scrubber water samples and these data were not used when evaluating critical project conclusions. As with volatile data from the scrubber liquor these data were used in conjunction with emission data to evaluate emission system efficiency.

Matrix spikes were not performed on this matrix because an insufficient amount of sample was sent to the laboratory. This has limited impact on data quality since most of these samples contained no significant levels of semivolatile compounds and were not used in calculating DREs to satisfy the project objectives.

Duplicate samples were collected for each Demonstration Test at the beginning and end of each run. Table 25 summarizes the results of the duplicates. For the pre-test samples, there were very few compounds detected. Most of the values were flagged with a "J" qualifier indicating that they are estimated values below the quantitation limit. Because values were detected below the standard laboratory quantitation limits, agreement among duplicates should not be expected to meet QAPP specifications. However, there is good agreement between some of the detected quantities as indicated by the RPD values.

The duplicate sample for Test 1 (SAIC #106) was lost during the laboratory extraction procedure. Since there were no significant quantities of target compounds detected in the post-test samples, the loss of this sample has no impact on data quality. As with the pre-test samples, the post-test samples contained very few target

compounds. Most of these values were also flagged with the "J" qualifier indicating that these values are estimated. Although some nitrophenolic compounds were detected in one of the two duplicate samples, this does not appear to be a matter of concern because they do not impact project objectives. Since these quantities are close to the detection limits, it is not surprising that they appear in one sample and not the other.

Table 25. Scrubber Water Semivolatile Duplicate Results

Compound	Primary (µg/L)	Duplicate (µg/L)	RPD	Objectives (RPD)
<b>Test 1</b>				
<b>POST-TEST</b>				
bis(2-Ethylhexyl)phthalate	2.6 JB	2.3 J	12	30
Nitrobenzene	9.3 J	6.0 J	43	30
2-Nitrophenol	*	5.9 J	NC	30
<b>Test 2</b>				
<b>PRE-TEST</b>				
bis(2-Ethylhexyl)phthalate	1.8 J	3.0 J	50	30
2-Methylnaphthalene	2.0 J	2.0 J	0	30
Naphthalene	3.0 J	3.2 J	6	30
<b>POST-TEST</b>				
4-Aminobiphenyl	*	3.0 J	NC	30
2,4-Dinitrophenol	25 J	*	NC	30
bis(2-Ethylhexyl)phthalate	2.9 J	*	NC	30
Nitrobenzene	5.0 J	4.8 J	4	30
2-Nitrophenol	9.7 J	*	NC	30
<b>Test 3</b>				
<b>PRE-TEST</b>				
Benzoic Acid	10 J	8.3 J	19	30
<b>POST-TEST</b>				
Naphthalene	1.2 J	1.2 J	0	30

\* Indicates that this compound was not detected.

B Indicates that this compound was detected in a blank.

J Estimated Result. Indicates that this compound was detected below the quantitation limit. The quantitation limit is defined as 5 times the detection limit.

NC Not calculated.

The field blank (SAIC #280) was lost during the extraction procedure. Based upon sample results, which showed no significant concentrations of target analytes, the loss of this sample has no impact on overall data quality.

### Semivolatile Emissions

Samples of the stack gas were collected for all three Demonstration Tests to determine the types and quantities of semivolatile emissions and to provide information for DRE calculations and, therefore, these measurements were considered critical. Samples were collected using an EPA Modified Method 5 (MM5) sampling train with XAD-2 resin. Specifics of this procedure are presented in Section 5. After each Demonstration Test, the sample trains were broken down and thoroughly rinsed to remove any semivolatile analytes from the filter housing, probe, and nozzle (PNR). The solutions were recovered from the impingers and sent to the laboratory for analysis. When the samples arrived in the laboratory they were composited and extracted using SW-846 Method 3540 for the XAD and 3510 for the impinger solutions and PNRs. Samples were spiked with the normal SW-846 Method 8270 surrogates and the special acid surrogates as noted above. These surrogates were added to the MM5 samples because of the high levels of  $\text{NO}_x$  anticipated in the stack. A summary of the surrogate recoveries for the semivolatile emission samples is presented in Table 26. As noted in the table, there were several surrogates that did not meet the specified recovery criteria. Some of the surrogate recovery information was lost because of dilutions that were required to bring target compounds into the calibration range, specifically benzoic acid. However, some samples analyzed without dilutions had surrogates that were not detected (recoveries of 0 percent). As previously noted with the scrubber liquor samples, normal 8270 surrogates were not expected to be detected due to nitration effects caused by high  $\text{NO}_x$  concentrations. This is why other acid surrogates were added to the matrix. Specifically, substituted phenols with large steric hinderences were chosen to prevent nitration. One method blank had poor recovery of 2,4-dinitrophenol- $\text{d}_3$  (3 percent) which may indicate that

Table 26. Semivolatile Emission Sample Surrogate Recoveries

Compound	No. of Results <sup>a</sup>	Mean Rec. (%)	Std. Dev. (%)	Min (%)	Max (%)	No. of Results Outside Limits		Control Limits (%)
						Low	High	
2-Fluorobiphenyl	3 <sup>b</sup>	137	45	103	188	0	2	30 - 115
2-Fluorophenol	1 <sup>b</sup>	0	NC	0	0	1	0	25 - 121
2,4-Dinitrophenol-d <sub>3</sub>	4	106	98	0	234	1	1	D - 191
2,4,6-Tribromophenol	1 <sup>b</sup>	0	NC	0	0	1	0	19 - 122
4,6-Dinitro-2-methylphenol-d <sub>2</sub>	4	115	20	92	140	0	0	D - 181
<sup>13</sup> C-Pentachlorophenol	4	23	29	0	61	2	0	14 - 176
Nitrobenzene-d <sub>5</sub>	4	91	23	63	120	0	0	23 - 120
Phenol-d <sub>5</sub>	1 <sup>b</sup>	2	NC	2	2	1	0	24 - 113
Terphenyl-d <sub>14</sub>	4	105	28	72	140	0	1	18 - 137

<sup>a</sup> Number in parentheses is number of routine field samples, including duplicates analyzed. Blanks, matrix spikes, and recovery checks are not included.

<sup>b</sup> Number of results less than number of samples because surrogates were diluted out in some samples.

NC - Not calculated.

D - Detected.

there was a problem with this surrogate standard. Low recoveries, 27 and 18 percent, were also noted in the recovery check samples. The carbon-labeled pentachlorophenol surrogate was not recovered in the Test 1 or Test 1 duplicate samples, due to a dilution effect. However, this surrogate was recovered within acceptable limits for every other sample. Recovery of acidic compounds in this matrix were, therefore, considered to be acceptable, but were not considered critical for evaluating project objectives. The base/neutral surrogates were recovered reasonably well with the exception of some high and low recoveries of 2-fluorobiphenyl. The specific reason for these QC outliers has not been determined but does not impact project objectives because all other base/neutral surrogates were within QC limits.

A second sample train was installed into the stack for Test 1 to collect a duplicate semivolatile stack sample. The results of this duplicate pair is presented in Table 27. Most of the targeted compounds detected in these two runs are flagged with the "J" qualifier. Results that are above the method quantitation limits show good agreement, with the exception of the phthalate compounds. Phthalates are commonly reported

as laboratory contaminants. Their low level presence, or absence, does not appear to have impacted data quality.

The field blank collected for these samples was clean with the exception of some low level phthalates and 120  $\mu\text{g}$  of naphthalene. It is important to note that this is approximately the same level of naphthalene that was detected in all of the process samples. Therefore, the levels of naphthalene that were detected in the emission samples were a result of field blank contamination and were disregarded when evaluating the semivolatile emission data from these tests.

### Metals

Several parameters were tested for metal content at various sampling locations throughout the system. Metals were denoted as a secondary critical parameter. Samples for metals analysis were collected from the feed soil, the treated soil, the

Table 27. Duplicate Semivolatile Emission Sample Results

Compound	Primary (ppbV)	Duplicate (ppbV)	RPD
Acetophenone	•	2.57 J	NC
Benzoic Acid	231.10	242.06	5
Dibutylphthalate	•	0.51 J	NC
Diethylphthalate	•	49.51	NC
2,4-Dinitrophenol	17.52 J	•	NC
bis(2-Ethylhexyl)phthalate	28.22 B	0.82 JB	189
Naphthalene	10.69	7.90	30
2-Nitrophenol	•	9.24 J	NC
4-Nitrophenol	12.56 J	•	NC

• Indicates that this compound was not detected.

B Indicates that this compound was detected in a blank.

J Estimated Result. Indicates that this compound was detected below the quantitation limit. The quantitation limit is defined as 5 times the detection limit.

NC Not calculated.

scrubber liquor before and after the test, the scrubber makeup and the stack gas. For these matrices, most of metals data was generated using SW-846 Method 6010, ion coupled plasma (ICP). A few metals could not be analyzed using this method. Therefore, mercury analysis was performed using SW-846 Method 7470/7471, cold vapor; arsenic by SW-846 Method 7060, atomic absorption; and selenium by SW-846 Method 7740, atomic absorption. To insure that data of known quality was obtained, the instruments were calibrated in accordance with procedures outlined in the appropriate methods and/or the QAPP.

For the ICP, a mixed standard calibration was performed each day using a multipoint calibration curve. For this calibration to be acceptable, the measured value of the high calibration standard had to be within  $\pm 10\%$  of the true value. Once the calibration met this acceptance criteria, a calibration check was performed at a frequency of 10%. Reagent blanks were also analyzed at a 10% frequency and all target metals had to be less than five times the method detection limit. Matrix spike and matrix spike duplicates were analyzed for each sample matrix. Duplicate samples were also collected in the field and analyzed for metals. ICP interference checks were performed at the beginning, middle, and end of each analysis. These checks required a 80 to 120% recovery of the true value for EPA check sample elements.

For the atomic absorption and the cold vapor analyses, similar analytical constraints were placed on instrument performance. Before analyzing samples, a multipoint calibration was performed. Calibration checks were performed after each multipoint calibration and after every 10 samples. Reagent blanks were also analyzed at a 10% frequency. The results of these blanks required that the target analyte was less than five times the method detection limit. Matrix spikes and matrix spike duplicates were performed for each matrix. As with the other metal parameters, field duplicates were collected and analyzed.

## **Feed Soil Metals**

Samples were collected of the feed soil under similar conditions as described for the feed soil semivolatile organics. At the end of the Demonstration Tests, samples were collected from the composite jar and sent to the laboratory for metals determination using SW-846 procedures specified in the QAPP. Several samples were collected from the composite jar which were randomly collected aliquots of the same composite. Five samples were collected for each test run. As with the semivolatiles, it was not possible to distinguish between these aliquots by specifying random primary and duplicate samples since they were all taken from the same jar. Therefore, the results are presented together for each test. Table 28 presents the results for the feed soil metals. As with the semivolatile samples, the data is presented two ways. The first part presents test-specific information with the appropriate means and standard deviations. The summary portion provides information on all of the test runs since the soil used for each test was from the same lot. As demonstrated by the results presented in Table 28, good agreement was achieved for each test along with good statistical results for all of the tests.

In order to allow a comparison of the feed soil and the treated soil results, the laboratory used a modified digestion procedure which uses a combination of hydrofluoric, hydrochloric, and nitric acids. This was done because of the difficulty encountered with breaking down the treated soil matrix during pre-demonstration activities. Since a combination of both acids was required for the digestion of the treated soil, this procedure was also used for the feed soil so that the results could be evaluated equivocally.

Matrix spikes were performed on the feed soil. Because of the high levels of zinc oxide in the feed soil, special considerations were made for the spiking of zinc in this matrix. Two samples which did not contain any zinc oxide spike (but which had been previously spiked with both diesel and hexachlorobenzene) were sent to the laboratory



**Table 28. Feed Soil Metal Results**

Compound	Sample 1 (mg/kg)	Sample 2 (mg/kg)	Sample 3 (mg/kg)	Sample 4 (mg/kg)	Sample 5 (mg/kg)	Mean	Std. Dev.	RSD
<b>Test 1</b>								
Aluminum	55000	52000	50000	51000	54000	52400	2074	3.96
Arsenic	210	240	200	170	210	206	25	12.18
Barium	610	540	520	500	580	550	45	8.13
Cadmium	7.3	7.0	•	•	•	NC	NC	NC
Calcium	13000	14000	12000	12000	13000	12800	837	6.54
Chromium	23	21	20	28	21	23	3	14.20
Copper	560	630	510	510	570	556	50	8.96
Iron	41000	38000	36000	37000	38000	38000	1871	4.92
Lead	480	470	450	420	460	456	23	5.05
Magnesium	5400	5300	4900	5200	5000	5160	207	4.02
Manganese	720	930	860	680	760	790	103	13.03
Mercury	1.3	1.4	1.3	1.2	1.4	1.32	0.08	6.34
Potassium	21000	19000	18000	18000	20000	19200	1304	6.79
Sodium	11000	10000	9600	9900	10000	10100	529	5.24
Vanadium	86	70	70	78	77	76	7	8.72
Zinc	23000	23000	23000	24000	23000	23200	447	1.93
<b>Test 2</b>								
Aluminum	48000	47000	45000	47000	47000	46800	1095	2.34
Arsenic	190	180	210	170	180	186	15	8.15
Barium	460	470	460	470	490	470	12	2.61
Cadmium	5.2	7.2	•	6.1	•	6	1	16.24
Calcium	12000	13000	12000	13000	12000	12400	548	4.42
Chromium	19	24	22	20	34	24	6	25.28
Copper	550	530	550	580	700	582	68	11.74
Iron	36000	38000	34000	35000	37000	36000	1581	4.39
Lead	400	400	390	430	410	406	15	3.74
Magnesium	4200	4400	4000	4500	4700	4360	270	6.20
Manganese	660	660	680	860	840	740	101	13.65
Mercury	1.4	1.3	1.5	1.3	1.3	1.36	0.09	6.58
Potassium	19000	19000	19000	19000	20000	19200	447	2.33

(cont.)

Table 28. (Continued)

Compound	Sample 1 (mg/kg)	Sample 2 (mg/kg)	Sample 3 (mg/kg)	Sample 4 (mg/kg)	Sample 5 (mg/kg)	Mean	Std. Dev.	RSD
Test 2 (Continued)								
Sodium	10000	9800	10000	10000	10000	9960	89	0.90
Vanadium	83	84	69	73	88	79	8	10.10
Zinc	22000	21000	27000	23000	27000	24000	2828	11.79
Test 3								
Aluminum	49000	50000	49000	48000	52000	49600	1517	3.06
Arsenic	200	200	200	260	220	216	26	12.07
Barium	500	490	480	510	520	500	16	3.16
Cadmium	.	.	5.5	.	.	NC	NC	NC
Calcium	12000	12000	12000	12000	13000	12200	447	3.67
Chromium	29	27	28	24	26	27	2	7.18
Copper	580	560	620	760	600	624	79	12.70
Iron	36000	36000	35000	38000	38000	36600	1342	3.67
Lead	410	430	430	420	410	420	10	2.38
Magnesium	4300	4600	4300	4400	4800	4480	217	4.84
Manganese	700	780	740	800	660	736	57	7.78
Mercury	1.1	1.3	1.2	1.2	1.2	1.2	0.07	5.89
Potassium	19000	19000	19000	20000	19000	19200	447	2.33
Sodium	11000	10000	10000	10000	11000	10400	548	5.27
Vanadium	81	80	79	83	87	82	3	3.86
Zinc	21000	23000	23000	22000	21000	22000	1000	4.55
Summary	Mean		Standard Deviation		RSD			
Aluminum	49600		2798		5.64			
Arsenic	203		25		12.13			
Barium	507		43		8.49			
Cadmium	6		1		14.27			
Calcium	12467		640		5.13			
Chromium	24		4		17.24			
Copper	587		68		11.65			
Iron	36867		1727		4.68			
Lead	427		27		6.28			

(cont.)

**Table 28. (Continued)**

Summary	Mean	Standard Deviation	RSD
Magnesium	4667	424	9.08
Manganese	755	87	11.49
Mercury	1.3	0.1	7.98
Potassium	19200	775	4.03
Sodium	10153	452	4.45
Vanadium	79	6	7.97
Zinc	23067	1831	7.94

specifically for the zinc spiking. The laboratory was also sent a portion of the zinc oxide lot which was used for the spiking of the Demonstration Test soil. The unspiked soil was spiked in the lab for MS and MSD analyses at the same level as the zinc oxide feed soil spike. Results of these spikes are presented in Table 29. The remaining metals were spiked into two actual feed soil samples. These results are also summarized and presented in Table 29. As noted by the data presented in this table, precision and accuracy objectives, as specified in the QAPP, were met for all parameters except silver. Silver was recovered poorly in the MS and MSD. However the precision was excellent for these recoveries. The poor recovery results have no impact on data quality since silver was not detected in any of the feed soil samples and was not a critical analyte.

Field blanks were collected for the feed soil at the time of sampling. The results of the field blanks indicated a small quantity of aluminum contamination for each of the tests. The amount of this contamination was 670, 360, and 340 ppm for Tests 1, 2, and 3, respectively. For the worst case, highest contamination and lowest aluminum feed soil concentration, this represents 1.5 percent of the measured value detected in routine samples. Therefore, field blank contamination for aluminum has little or no impact on data quality. Additionally, a small quantity (76 ppm) of iron was detected in the Test 3 field blank. This has no impact on data quality since reported iron concentrations were several orders of magnitude higher than the field blank.

**Table 29. Feed Soil Metal Matrix Spikes Results (Test 1)**

Compound	% Recoveries			RPD	Objectives	
	MS	MSD	Mean		Accuracy (% Rec.)	Precision (RPD)
Aluminum	70	71 Q	71	1.4	65-140	30
Antimony	98	91	95	7.4	65-140	30
Arsenic	109	108	109	0.9	65-140	30
Barium	94	96	95	2.1	65-140	30
Beryllium	92	92	92	0.0	65-140	30
Cadmium	94	94	94	0.0	65-140	30
Calcium	94	88	91	6.6	65-140	30
Chromium	96	97	97	1.0	65-140	30
Copper	98	102	100	4.0	65-140	30
Iron	70 Q	68 Q	69	2.9	65-140	30
Lead	90	94	92	4.3	65-140	30
Magnesium	94	94	94	0.0	65-140	30
Manganese	97	100	99	3.0	65-140	30
Mercury	102	84	93	19.4	65-140	30
Nickel	98	97	98	1.0	65-140	30
Potassium	90	110	100	20.0	65-140	30
Selenium	83	81	82	2.4	65-140	30
Silver	22 Q	22 Q	22	0.0	65-140	30
Sodium	90	85	88	5.7	65-140	30
Thallium	98	97	98	1.0	65-140	30
Vanadium	94	89	92	5.5	65-140	30
Zinc*	98	92	95	6.3	75-120	30

Q Outside control limits.

- \* Zinc was not actually spiked into the Test 1 feed soil, but rather into a sample of feed soil previously spiked with diesel oil and hexachlorobenzene but no zinc oxide.

Method blanks for metals contained consistent 50 and 220 ppm levels of copper and iron, respectively. The copper contamination is of significance since this level represents approximately 5 to 10 percent of the detected copper in the feed soil samples. Since copper was not present in large enough quantities to effectively track process performance, the blank contamination has little effect on project objectives. Copper was not used in the evaluation of the technology. The iron present in the field

blank and method blank totals approximately 1 to 2 percent of the feed soil content and does not have a significant impact on data quality. All feed soil metals data appears in Appendix D.

#### **Treated Soil Metals**

Samples of the treated soil were collected after the melt had cooled and hardened. Samples were collected in accordance with procedures outlined in Section 5 and previously discussed in this section. To effectively evaluate the metal content of the treated soil, the samples were crushed into a powder form and subjected to microwave digestion. A mixture of hydrofluoric, hydrochloric, and nitric acids was used to break down the glass monolithic matrix. The QAPP specified that a primary and a duplicate sample be collected for each test run. Table 30 presents the results of the treated soil metal duplicates for Tests 1, 2, and 3. As noted in the table, precision objectives met the specified QAPP criteria for all metals with the exception of arsenic which had a RPD of 38 percent in Test 2. Arsenic concentrations were very low (less than 20 ppm) for all of the Demonstration Test results; hence, this QC outlier does not have an impact on data quality.

A matrix spike analysis and a matrix spike duplicate analysis were performed on a treated soil sample from Test 1. The results of these analyses are presented in Table 31. As noted in the table, all of the spiked compounds met precision and accuracy objectives for all of the metals with the exception of silver, and one high recovery for aluminum. The matrix spike duplicate for aluminum was recovered at 143% while both the MS and MSD for silver were recovered at 16 and 17%, respectively. These QC outliers do not have a significant impact on overall data quality for the treated soil metals because neither silver nor aluminum were used in the evaluation of project objectives.

**Table 30. Treated Soil Metal Duplicate Results**

Compound	Primary (mg/kg)	Duplicate (mg/kg)	RPD	Objectives (RPD)
<b>Test 1</b>				
Aluminum	48000	49000	2.06	30
Arsenic	13	14	7.41	30
Barium	530	510	3.85	30
Calcium	27000	27000	0.00	30
Chromium	520	500	3.92	30
Copper	800	820	2.47	30
Iron	160000	160000	0.00	30
Lead	120	95	23.26	30
Magnesium	6000	5500	8.70	30
Manganese	1900	1900	0.00	30
Nickel	290	280	3.51	30
Potassium	16000	15000	6.45	30
Sodium	8200	8600	4.76	30
Vanadium	61	61	0.00	30
Zinc	6600	6900	4.44	30
<b>Test 2</b>				
Aluminum	45000	47000	4.35	30
Arsenic	13	19	37.50	30
Barium	460	500	8.33	30
Calcium	21000	20000	5.00	30
Chromium	520	500	3.85	30
Copper	1700	1300	26.67	30
Iron	150000	150000	0.00	30
Lead	130	100	26.09	30
Magnesium	4200	5000	17.39	30
Manganese	1900	1900	0.00	30
Nickel	260	270	6.06	30
Potassium	16000	16000	0.00	30
Sodium	8700	8600	1.16	30
Vanadium	60	59	1.68	30

(cont.)

**Table 30. (Continued)**

Compound	Primary (mg/kg)	Duplicate (mg/kg)	RPD	Objectives (RPD)
<b>Test 2 (Continued)</b>				
Zinc	9300	8800	5.52	30
<b>Test 3</b>				
Aluminum	45000	41000	9.30	30
Arsenic	11	13	16.67	30
Barium	480	420	13.64	30
Calcium	18000	18000	0.00	30
Chromium	610	610	0.00	30
Copper	820	830	1.21	30
Iron	220000	210000	4.65	30
Lead	120	120	0.00	30
Magnesium	5000	4100	19.78	30
Manganese	2600	2500	3.92	30
Nickel	290	280	3.51	30
Potassium	15000	14000	6.90	30
Sodium	7800	7800	0.00	30
Vanadium	49	52	5.94	30
Zinc	8600	9100	5.65	30

The laboratory method blanks performed along with the analyses of these samples were all clean. Analysis of the drill core cooling water sample, which was collected to measure any losses during sampling, contained very low levels of metals. All metals detected in this sample were less than 10 ppm with the exception of the following: calcium, 18 ppm; potassium, 62 ppm; and sodium, 40 ppm. These results indicate that there were no significant losses of metal compounds from the drilling process because the amounts found in these samples were several orders of magnitude lower than routine analyses of the treated soil samples. All of the treated soil data may be found in Appendix E.

**Table 31. Treated Soil Metal Matrix Spikes Results (Test 1)**

Compound	% Recoveries			RPD	Objectives	
	MS	MSD	Mean		Accuracy (% Rec.)	Precision (RPD)
Aluminum	132	143 Q	138	8.0	65-140	30
Antimony	94	94	94	0.0	65-140	30
Arsenic	107	111	109	3.7	65-140	30
Barium	101	103	102	2.0	65-140	30
Beryllium	90	90	90	0.0	65-140	30
Cadmium	88	90	89	2.2	65-140	30
Calcium	86	96	91	11.0	65-140	30
Chromium	94	95	95	1.1	65-140	30
Copper	94	96	95	2.1	65-140	30
Iron	93	89	91	4.4	65-140	30
Lead	95	98	97	3.1	65-140	30
Magnesium	98	96	97	2.1	65-140	30
Manganese	99	100	100	1.0	65-140	30
Nickel	93	93	93	0.0	65-140	30
Potassium	120	126	123	4.9	65-140	30
Selenium	86	84	85	2.4	65-140	30
Silver	16 Q	17 Q	17	6.1	65-140	30
Sodium	95	96	96	1.0	65-140	30
Thallium	92	90	91	2.2	65-140	30
Vanadium	94	95	95	1.1	65-140	30
Zinc	88	89	89	1.1	75-120	30

Q - Outside control limits.

### Scrubber Water Metal Samples

Samples of the scrubber water were collected before and after each Demonstration Test. The samples were tested for metal content by using procedures outlined in SW-846. Most metal results were obtained by digesting the samples using SW-846 Method 3010 and analyzing the digestate by SW-846 Method 6010, ICP. Other



metals (arsenic, mercury, and selenium) were evaluated using procedures outlined in SW-846 Methods 7060, 7470, and 7740, respectively.

One primary and one duplicate sample were collected at the beginning and the end of each demonstration test from the scrubber sump. Sampling procedures are described in Section 5. Table 32 presents the results of the scrubber water duplicate samples. Values were reported in this table only if they appeared in both samples. In general, most of the QAPP objectives for precision were met. With this much data, it is not surprising that some of the metals do not meet the objectives. The post-test duplicates for Test 2 show the greatest amount of variability and several metals do not meet the objectives. The reason for this is unclear. This may indicate that the contents of the scrubber after this test were not well-mixed when the samples were collected or that the sample line was not purged sufficiently. In general, these QC results are good and do not imply that data quality has been impacted for the scrubber water metal samples. In addition, these data have a minimum impact on the project because scrubber water results are only indirectly used for evaluation of the technology and are not used for DRE or leachability claims.

Matrix spikes were not performed on this matrix for metals with the exception of arsenic. Arsenic was recovered high at values of 152 and 174 percent recovery. The matrix spikes for the additional metals was not performed. Since there were several duplicates collected for this matrix, and since only low levels were detected, the impact of not having spiked sample results is limited and does not seriously affect data quality. These data were not used in determining previously defined numerical objectives and, overall, are not critical in evaluating process performance.

Laboratory method blanks that were analyzed along with the scrubber liquor samples were free of contamination and therefore do not impact data quality.

**Table 32. Scrubber Water Sample Metal Duplicate Results**

Compound	Primary (mg/L)	Duplicate (mg/L)	RPD	Objectives (RPD)
<b>Test 1</b>				
<b>PRE-TEST</b>				
Aluminum	0.45	0.50	10.53	20
Arsenic	0.021	0.024	13.33	20
Barium	0.011	0.011	0.00	20
Calcium	13	12	8.00	20
Chromium	0.45	0.57	23.53	20
Copper	0.91	1.00	9.42	20
Iron	13	16	20.69	20
Lead	0.053	0.054	1.87	20
Magnesium	4.1	4.1	0.00	20
Manganese	0.12	0.14	15.38	20
Nickel	0.13	0.18	32.26	20
Potassium	14	14	0.00	20
Selenium	0.0099	0.0089	10.64	20
Sodium	350	360	2.82	20
Vanadium	0.050	0.053	5.83	20
Zinc	0.61	0.77	23.19	20
<b>POST-TEST</b>				
Aluminum	3.0	2.9	3.39	20
Arsenic	4.9	5.5	11.54	20
Barium	0.17	0.17	0.00	20
Cadmium	0.085	0.093	8.99	20
Calcium	9.8	10.0	2.02	20
Chromium	0.99	1.00	1.01	20
Copper	4.1	4.2	2.41	20
Iron	34	34	0.00	20
Lead	2.6	2.7	3.77	20
Magnesium	3.2	3.3	3.08	20
Manganese	0.61	0.63	3.23	20
Mercury	0.088	0.032	93.33	20

(cont.)

Table 32. (Continued)

Compound	Primary (mg/L)	Duplicate (mg/L)	RPD	Objectives (RPD)
<b>Test 1 (Continued)</b>				
<b>POST-TEST (CONTINUED)</b>				
Nickel	0.41	0.42	2.41	20
Potassium	36	37	2.74	20
Selenium	0.12	0.12	0.00	20
Sodium	4200	4400	4.65	20
Vanadium	0.12	0.12	0.00	20
Zinc	110	120	8.70	20
<b>Test 2</b>				
<b>PRE-TEST</b>				
Aluminum	1.1	1.1	0.00	20
Arsenic	1.5	1.5	0.00	20
Barium	0.045	0.045	0.00	20
Cadmium	0.026	0.024	8.00	20
Calcium	4.6	4.6	0.00	20
Chromium	0.90	0.90	0.00	20
Copper	2.7	2.7	0.00	20
Iron	28	28	0.00	20
Lead	0.78	0.80	2.53	20
Manganese	0.30	0.30	0.00	20
Mercury	0.0040	0.0028	35.29	20
Molybdenum	0.14	0.14	0.00	20
Nickel	0.31	0.31	0.00	20
Potassium	4.8	4.9	2.06	20
Selenium	.014	.012	15.38	20
Silicon	24	24	0.00	20
Silver	0.020	0.020	0.00	20
Sodium	180	190	5.41	20
Vanadium	0.048	0.047	2.11	20
Zinc	27	28	3.64	20

(cont.)

Table 32. (Continued)

Compound	Primary (mg/L)	Duplicate (mg/L)	RPD	Objectives (RPD)
Test 2 (Continued)				
POST-TEST				
Aluminum	2.6	1.7	41.86	20
Arsenic	7.8	3.9	66.67	20
Barium	0.19	0.15	23.53	20
Boron	4.8	4.8	0.00	20
Cadmium	0.140	0.085	48.89	20
Calcium	11	11	0.00	20
Chromium	1.60	0.81	65.56	20
Cobalt	0.020	0.013	42.42	20
Copper	9.0	4.6	64.71	20
Iron	42	22	62.50	20
Lead	4.6	2.3	66.67	20
Magnesium	1.3	1.0	26.09	20
Manganese	0.66	0.45	37.84	20
Mercury	0.13	0.13	0.00	20
Molybdenum	1.5	1.3	14.29	20
Nickel	0.57	0.35	47.83	20
Potassium	25	23	8.33	20
Selenium	0.110	0.091	18.91	20
Silicon	100	68	38.10	20
Silver	0.076	0.073	4.03	20
Sodium	2400	2000	18.18	20
Strontium	0.062	0.058	6.67	20
Vanadium	0.22	0.14	44.44	20
Zinc	240	100	82.35	20
Test 3				
PRE-TEST				
Aluminum	0.49	0.43	13.04	20
Arsenic	0.35	0.32	8.96	20

(cont.)

Table 32. (Continued)

Compound	Primary (mg/L)	Duplicate (mg/L)	RPD	Objectives (RPD)
Test 3 (Continued)				
PRE-TEST (CONTINUED)				
Barium	0.030	0.027	10.53	20
Boron	1.5	1.4	6.90	20
Cadmium	0.025	0.028	11.32	20
Calcium	4.7	4.8	2.11	20
Chromium	1.10	0.96	13.59	20
Copper	1.5	1.4	6.90	20
Iron	41	37	10.26	20
Lead	0.68	0.63	7.63	20
Manganese	0.42	0.38	10.00	20
Mercury	0.0028	0.0027	3.64	20
Molybdenum	0.31	0.28	10.17	20
Nickel	0.28	0.26	7.41	20
Potassium	8.4	7.7	8.70	20
Silicon	29	24	18.87	20
Silver	0.013	0.013	0.00	20
Sodium	650	600	8.00	20
Strontium	0.020	0.020	0.00	20
Vanadium	0.028	0.026	7.41	20
Zinc	26	24	8.00	20
POST-TEST				
Aluminum	1.1	1.1	0.00	20
Arsenic	3.3	7.7	80.00	20
Barium	0.080	0.079	1.26	20
Boron	1.1	1.1	0.00	20
Cadmium	0.090	0.078	14.29	20
Calcium	4.4	4.0	9.52	20
Chromium	1.9	1.9	0.00	20
Cobalt	0.022	0.024	8.70	20

(cont.)

Table 32. (Continued)

Compound	Primary (mg/L)	Duplicate (mg/L)	RPD	Objectives (RPD)
Test 3 (Continued)				
POST-TEST (CONTINUED)				
Copper	3.4	3.3	2.99	20
Iron	46	44	4.44	20
Lead	2.1	2.1	0.00	20
Manganese	0.53	0.52	1.90	20
Mercury	0.018	0.020	10.53	20
Molybdenum	0.40	0.40	0.00	20
Nickel	0.96	0.97	1.04	20
Potassium	13	13	0.00	20
Selenium	0.020	0.017	16.22	20
Silicon	43	42	2.35	20
Silver	0.057	0.048	17.14	20
Sodium	1200	1200	0.00	20
Strontium	0.019	0.018	5.41	20
Vanadium	0.071	0.069	2.86	20
Zinc	65	64	1.55	20

### Metal Emissions Data

For each of the Demonstration Tests, samples of the stack gas were collected to evaluate metal emissions. Samples were collected using the Multiple Metals Train (MMT) which is discussed in Section 5. At the completion of each Demonstration Test, the impinger solutions were collected, the filter was recovered, and the sample train components were rinsed. In the laboratory, the probe and nozzle rinses (PNR) were combined with the filters and digested as single sample. All of the data generated for the metals emissions were obtained from the filter and PNR.

The analyses of the impinger solutions yielded non detectable quantities for most metals, or values at or near the detection limits. Since these results were insignificant, they were not included in the calculation of the total sample train catch. Mercury was the only metal that was found in the impingers at a significant level. This is not surprising given the high volatility of mercury and the operating temperatures of the process. Raw data for these measurements can be found in Appendix C.

For Test 2, a primary and a duplicate MMT were installed into the stack to collect duplicate metal emission samples. The results of these duplicates are presented in Table 33. As seen by the calculated RPD values, the results of the duplicates are poor. Most of the reported values in the duplicate train are a factor of 2 higher than the primary train. A check of raw data and calculations indicated no source of error in the reporting of the results. The results of the duplicates indicate that there is some inconsistency associated with the stack gas flow or sampling procedures. It is possible that one of the trains was not located correctly inside the stack and an isokinetic sample was not collected. There were also several problems noted with the blower during testing activities which may have caused flow irregularities. Several metals were also detected in the field blank which is discussed below. Limitations on these data are discussed later in this section under "CONCLUSIONS AND LIMITATIONS OF DATA."

Field blanks for the metal trains showed detectable levels for most of the target analytes. Aluminum, calcium, and sodium were detected at levels greater than 1,000  $\mu\text{g}$ . The amount of aluminum, calcium, and sodium (combined) in the field blanks represents approximately 25, 10, and 15 percent of the catch for Tests 1, 2, and 3, respectively. Magnesium was detected at 480  $\mu\text{g}$  in the field blank. This represents about 50 percent of the catch for the Demonstration Test samples. All other metals detected in the field blank were insignificant in comparison with the Demonstration Test results. Zinc, which was of critical importance to this demonstration, was

**Table 33. Metals Emissions Duplicate Sample Results (Test 2)**

Compound	Primary (ppm)	Duplicate (ppm)	RPD
Aluminum	1.396 B	2.800 B	66.92
Antimony	0.046	0.136	98.90
Arsenic	3.323 B	9.753 B	98.35
Barium	0.113 B	0.242 B	72.68
Beryllium	0.0005	0.0010	66.67
Cadmium	0.057	0.119	70.45
Calcium	2.525 B	5.089 B	67.35
Chromium	0.930 B	1.951 B	70.88
Copper	6.646 B	13.994 B	71.20
Iron	43.197 B	89.053 B	68.83
Lead	3.256 B	6.785 B	70.29
Magnesium	0.498 B	0.933 B	60.80
Manganese	0.326 B	0.763 B	80.26
Mercury	0.0043 B	0.0080 B	60.16
Nickel	0.236 B	0.509 B	73.29
Potassium	9.969	21.203	72.78
Selenium	0.015	0.034	77.55
Silver	0.009	0.019	71.43
Sodium	5.981 B	12.722 B	72.08
Thallium	0.017	0.042	84.75
Vanadium	0.093	0.195	70.83
Zinc	116.299 B	250.197 B	45.78

B - Indicates that this compound was detected in the field blank.

detected at 160  $\mu\text{g}$ . This is several orders of magnitude lower than values found in test samples; therefore, field blank contamination has little or no effect on the evaluation of the ability of the process to treat zinc. Field blank data may be found in Appendix C. Other metals data was not used in evaluating project objectives other than total emissions. The blank data does not impact total emissions output as zinc is the dominant metal in the gas stream as stated by project conclusions. Method



blanks analyzed along with these samples were clean with the exception of small quantities of iron (5.5  $\mu$ g) being detected in one blank. These levels are insignificant in comparison the levels of iron detected in the Demonstration Test samples.

Matrix spike recovery checks were prepared and analyzed for the multiple metals train by spiking a blank filter and clean impinger solutions with the targeted analytes. Recovery information for these spikes is presented in Table 34. This table shows that the recovery for most of the analytes fell within the accuracy objectives specified by the laboratory. Silver was the only outlier with a 20 percent recovery from the filter media. Since silver was not a metal of great interest to this project, this poor recovery has no impact on data quality.

### TCLP Analyses

Leachability of the treated soil was considered an extremely critical parameter for evaluation in these Demonstration Tests. Samples were subjected to TCLP and then analyzed for semivolatiles and metals. The TCLP extractions were performed in accordance with SW-846 Method 1311. The TCLP leachate was analyzed using SW-846 Method 8270 for semivolatiles and SW-846 Method 6010 for most metals. Other metals were analyzed as previously specified in this section. Detailed discussions of the extraction, digestion, and quality control procedures utilized to ensure proper sample preparation, instrument tuning, and calibration have been outlined previously in this section for both semivolatiles and metals.

### TCLP Metals

Samples of the feed and treated soil were collected to determine the leachability of metals. In general, low levels of metals were detected in the leachate samples except for calcium, zinc, and sodium in the feed soil. Zinc, which was spiked at high levels, leached freely from the feed soil and was effectively contained in the treated material.

Table 34. Recovery Check Results for Multiple Metals Trains

Compound	Filter/Liquid	% Recovery
Arsenic	Filter	91
Mercury	Filter	101
Silver	Filter	20 Q
Antimony	Filter	83
Beryllium	Filter	86
Cadmium	Filter	86
Zinc	Filter	87
Magnesium	Filter	88
Copper	Filter	90
Manganese	Filter	90
Vanadium	Filter	90
Nickel	Filter	92
Barium	Filter	93
Aluminum	Filter	94
Chromium	Filter	94
Lead	Filter	94
Thallium	Filter	95
Calcium	Filter	96
Sodium	Filter	96
Iron	Filter	98
Potassium	Filter	101
Selenium	Filter	85
Arsenic	Liquid	107
Mercury	Liquid	99
Aluminum	Liquid	92
Antimony	Liquid	93
Cadmium	Liquid	94
Copper	Liquid	95
Manganese	Liquid	95
Thallium	Liquid	95
Vanadium	Liquid	95

(cont.)

Table 34. (Continued)

Compound	Filter/Liquid	% Recovery
Barium	Liquid	96
Beryllium	Liquid	96
Chromium	Liquid	96
Magnesium	Liquid	96
Nickel	Liquid	96
Silver	Liquid	97
Sodium	Liquid	97
Zinc	Liquid	97
Iron	Liquid	99
Calcium	Liquid	100
Lead	Liquid	100
Potassium	Liquid	100
Selenium	Liquid	92

Q - Outside laboratory recovery objectives (75-125%).

Calcium was also effectively encapsulated in the treated slag. Additional discussion of metals leachability is presented in Section 6. For Test 1, a duplicate sample of the feed soil was collected and analyzed. For each of the Demonstration Tests, duplicate samples of the treated soil were collected and analyzed. Table 35 presents the results of the duplicate samples. As noted in the table, several of the metals did not meet the precision objectives stated in the QAPP for all of the duplicate pairs in the feed and treated soil. In general, the duplicate results for the feed appear to be satisfactory except for analytes detected at very low levels. There were values that exceeded the limits for iron, copper and mercury in the feed soil, while the treated soil had a variety of elements which exceeded the QAPP objectives. Some metals were detected in one of the duplicate pairs and not the other. In these cases, the detected value was close to the reporting limits and data quality does not appear to have been impacted. These high RPD values indicate that there is some variability associated within the treated soil. This can be expected since the process does not "mix" the soil while it is being treated. The variability of some of these results does not imply that the data is of poor quality, rather it suggests variability in the treated soil matrix. This variability,

Table 35. TCLP Metals Duplicate Sample Results

Compound	Primary (mg/L)	Duplicate (mg/L)	RPD	Objectives (RPD)
Test 1				
FEED SOIL				
Barium	0.16	0.12	28.57	30
Cadmium	0.066	0.064	3.08	30
Calcium	190	170	11.11	30
Copper	6.2	3.7	50.51	30
Iron	0.110	0.055	66.67	30
Magnesium	7.8	7.9	1.27	30
Manganese	5.0	4.6	8.33	30
Mercury	0.0003	0.0008	90.91	30
Potassium	4.9	4.2	15.38	30
Sodium	1600	1400	13.33	30
Vanadium	0.094	0.097	3.14	30
Zinc	1000	1000	0.00	30
TREATED SOIL				
Aluminum	0.29	0.34	15.87	30
Barium	0.087	0.074	16.15	30
Calcium	1.8	2.5	32.56	30
Copper	0.12	0.23	62.86	30
Iron	2.1	3.1	38.46	30
Manganese	0.050	0.061	19.82	30
Sodium	1500	1500	0.00	30
Zinc	0.39	0.51	26.67	30
Test 2				
TREATED SOIL				
Aluminum	0.28	0.55	65.06	30
Barium	0.090	0.080	11.76	30
Calcium	1.3	3.8	98.04	30
Copper	0.17	0.54	104.23	30
Iron	2.4	3.5	37.29	30

(cont.)

Table 35. (Continued)

Compound	Primary (mg/L)	Duplicate (mg/L)	RPD	Objectives (RPD)
<b>Test 2 (Continued)</b>				
<b>TREATED SOIL (CONTINUED)</b>				
Manganese	0.051	0.071	32.79	30
Sodium	1400	1400	0.00	30
Zinc	0.32	0.40	22.22	30
<b>Test 3</b>				
<b>TREATED SOIL</b>				
Aluminum	0.39	0.25	43.75	30
Barium	0.070	0.080	13.33	30
Calcium	2.9	1.2	82.93	30
Copper	0.11	0.50	127.87	30
Iron	4.3	58	172.39	30
Manganese	0.13	0.36	93.88	30
Sodium	1400	1400	0.00	30
Zinc	0.37	0.23	46.67	30

however, does not significantly impact project conclusions because of how this data is evaluated, as explained in Section 6. Data evaluations account for variability associated with this matrix.

Spiked samples were analyzed for the treated and feed soil. These results are summarized in Table 36. The feed soil spikes exhibit good precision and accuracy values for all metals with the exception of calcium, sodium, and zinc. This is most likely due to the high levels of these compounds found in the unspiked samples. It is possible that these metals were not spiked at high enough levels to exceed the native concentration found in the matrix for these metals. Hence, this behavior can be expected and does not imply that routine samples results have been impacted. The treated soil spikes showed excellent precision and accuracy values for all metals with

Table 36. TCLP Metals Spiked Sample Results

Field ID	Element	% Recoveries			RPD	Objectives	
		MS	MSD	Mean		Accuracy (% Rec.)	Precision (RPD)
TEST SOIL							
SAIC-0309 MS	Aluminum	103	96	100	7.0	65-140	30
SAIC-0309 MS	Antimony	91	89	90	2.5	65-140	30
SAIC-0309 MS	Arsenic	88	93	91	5.5	65-140	30
SAIC-0309 MS	Barium	101	101	101	0.0	65-140	30
SAIC-0309 MS	Beryllium	101	101	101	0.0	65-140	30
SAIC-0309 MS	Cadmium	96	96	96	0.0	65-140	30
SAIC-0309 MS	Calcium	48 QX	138 QX	NC	NC	65-140	30
SAIC-0309 MS	Chromium	100	99	100	1.0	65-140	30
SAIC-0309 MS	Copper	86	108	97	22.7	65-140	30
SAIC-0309 MS	Iron	102	102	102	0.0	65-140	30
SAIC-0309 MS	Lead	97	97	97	0.0	65-140	30
SAIC-0309 MS	Magnesium	96	100	98	4.1	65-140	30
SAIC-0309 MS	Manganese	82	107	95	26.5	65-140	30
SAIC-0309 MS	Mercury	117	115	116	1.7	65-140	30
SAIC-0309 MS	Nickel	99	98	99	1.0	65-140	30
SAIC-0309 MS	Potassium	94	106	100	12.0	65-140	30
SAIC-0309 MS	Selenium	75	74 Q	75	1.3	65-140	30
SAIC-0309 MS	Silver	97	96	97	1.0	65-140	30
SAIC-0309 MS	Sodium	0 QX	304 QX	NC	NC	65-140	30
SAIC-0309 MS	Thallium	107	102	105	4.8	65-140	30
SAIC-0309 MS	Vanadium	100	99	100	1.0	65-140	30
SAIC-0309 MS	Zinc	0 QX	1110 QX	NC	NC	75-120	30
TREATED SOIL							
SAIC-0314 MS	Aluminum	100	102	101	2.0	65-140	30
SAIC-0314 MS	Antimony	110	108	109	1.8	65-140	30
SAIC-0314 MS	Arsenic	96	97	97	1.0	65-140	30
SAIC-0314 MS	Barium	102	103	103	1.0	65-140	30
SAIC-0314 MS	Beryllium	102	103	103	1.0	65-140	30
SAIC-0314 MS	Cadmium	97	98	98	1.0	65-140	30

(cont.)

Table 36. (Continued)

Field ID	Element	% Recoveries			RPD	Objectives	
		MS	MSD	Mean		Accuracy (% Rec.)	Precision (RPD)
TREATED SOIL (CONTINUED)							
SAIC-0314 MS	Calcium	105	107	106	1.9	65-140	30
SAIC-0314 MS	Chromium	101	102	100	1.0	65-140	30
SAIC-0314 MS	Copper	99	101	100	2.0	65-140	30
SAIC-0314 MS	Iron	103	104	104	1.0	65-140	30
SAIC-0314 MS	Lead	104	106	105	1.9	65-140	30
SAIC-0314 MS	Magnesium	103	104	104	1.0	65-140	30
SAIC-0314 MS	Manganese	100	101	101	1.0	65-140	30
SAIC-0314 MS	Mercury	99	98	99	1.0	65-140	30
SAIC-0314 MS	Nickel	102	103	103	1.0	65-140	30
SAIC-0314 MS	Potassium	121	118	120	2.5	65-140	30
SAIC-0314 MS	Selenium	64 Q	54 Q	59	16.9	65-140	30
SAIC-0314 MS	Silver	97	98	98	1.0	65-140	30
SAIC-0314 MS	Sodium	430 QX	100	100	NC	65-140	30
SAIC-0314 MS	Thallium	102	108	105	5.7	65-140	30
SAIC-0314 MS	Vanadium	101	102	102	1.0	65-140	30
SAIC-0314 MS	Zinc	101	101	101	0.0	75-120	30

Q Outside control limits.

X Amount of spike added was significantly less than the natural analyte concentration in the sample; recovery not meaningful due to dilution or background interference.

NC Not calculated.

the exception of sodium. Problems associated with the sodium recovery are discussed below and discussed in Section 6.

Method blanks for the TCLP metals samples were free of significant contamination that may have effected results with the exception of sodium. Sodium was detected in a method blank at a level similar to those found in project samples. This could explain the poor precision and accuracy values for sodium in the treated soil spikes. Although sodium contamination was detected in the method blanks, this does not present a significant impact on data quality. Since sodium hydroxide was used in the

scrubber as a neutralizing agent, sodium was not a heavily weighted factor used to evaluate critical project objectives.

### **TCLP Semivolatiles**

Samples of the feed and treated soil were collected for evaluation of leachable semivolatile compounds. There were no target compounds detected in either of these matrices. Duplicates were collected and analyzed for each test as described in the TCLP metals section. Since none of the targeted compounds were detected, it was not possible to evaluate the results of the sample duplicates from a precision standpoint. The investigators of this project were surprised to find that none of the hexachlorobenzene leached from the feed soil. To confirm these results, a special study, which is later discussed under "SPECIAL STUDIES," was conducted.

Matrix spikes were performed on both the treated soil and feed soil leachates. These results are summarized in Table 37. The feed soil show one poor recovery for phenol and some high-RPD values for 2-chlorophenol, 4-chloro-3-methylphenol and phenol. Recoveries for the treated soil were all acceptable and the RPD values were within the control limits. Since no target compounds were detected in the feed soil, the poor QC associated with a few of the spike compounds has no impact on TCLP data quality.

Surrogates spiked into the TCLP semivolatile leachate samples are summarized in Table 38. As noted in the table, there are some recoveries that are outside the control limits. Based upon the sample results, these surrogate recoveries have little impact on the data because there were no semivolatile compounds detected in the leachates and these data were not used to evaluate numerical project objectives. The project conclusions simply state that no semivolatiles were found to leach from the treated soil.



Table 37. TCLP Semivolatile Matrix Spike Results (Test 3)

Field ID	Compound	% Recoveries			RPD	Objectives	
		MS	MSD	Mean		Accuracy (% Rec.)	Precision (RPD)
FEED SOIL							
SAIC-0309 MS	1,2,4-Trichlorobenzene	103	94	99	9.1	38-107	23
SAIC-0309 MS	1,4-Dichlorobenzene	82	74	78	10.3	28-104	27
SAIC-0309 MS	2-Chlorophenol	69	36	53	62.9	25-102	50
SAIC-0309 MS	2,4-Dinitrotoluene	90	89	90	1.1	28-89	47
SAIC-0309 MS	4-Chloro-3-methylphenol	71	30	51	81.2	26-102	33
SAIC-0309 MS	4-Nitrophenol	84	54	69	43.5	11-114	50
SAIC-0309 MS	Acenaphthene	108	96	102	11.8	31-137	19
SAIC-0309 MS	N-Nitrosodipropylamine	100	93	97	7.3	41-126	38
SAIC-0309 MS	Pentachlorophenol	65	42	54	43.0	17-109	47
SAIC-0309 MS	Phenol	46	20	33	78.8	26-90	35
SAIC-0309 MS	Pyrene	105	102	104	2.9	35-142	36
TREATED SOIL							
SAIC-0314 MS	1,2,4-Trichlorobenzene	82	91	87	10.4	38-107	23
SAIC-0314 MS	1,4-Dichlorobenzene	71	74	73	4.1	28-104	27
SAIC-0314 MS	2-Chlorophenol	74	83	79	11.5	25-102	50
SAIC-0314 MS	2,4-Dinitrotoluene	89	84	87	5.8	28-89	47
SAIC-0314 MS	4-Chloro-3-methylphenol	85	82	84	3.6	26-103	33
SAIC-0314 MS	4-Nitrophenol	61	78	70	24.5	11-114	50
SAIC-0314 MS	Acenaphthene	89	92	91	3.3	31-137	19
SAIC-0314 MS	N-Nitrosodipropylamine	92	87	90	5.6	41-126	38
SAIC-0314 MS	Pentachlorophenol	59	75	67	23.9	17-109	47
SAIC-0314 MS	Phenol	61	72	67	16.5	26-90	35
SAIC-0314 MS	Pyrene	108	102	105	5.7	35-142	36

Table 38. TCLP Semivolatile Surrogate Spike Recovery Data

Compound	No. of Results*	Mean Recovery (%)	Std. Dev. (%)	Min (%)	Max (%)	No. of Results Outside Limits		Control Limits (%)
						Low	High	
FEED SOIL								
2-Fluorobiphenyl	4	58	32	33	105	2	0	43-116
2-Fluorophenol	4	53	29	10	70	1	0	21-100
2,4,6-Tribromophenol	4	71	27	31	89	0	0	10-123
Nitrobenzene-d <sub>5</sub>	4	83	5	78	88	0	0	35-114
Phenol-d <sub>5</sub>	4	70	38	14	92	0	0	10-94
Terphenyl-d <sub>14</sub>	4	110	10	101	124	0	0	33-141
TREATED SOIL								
2-Fluorobiphenyl	7	76	27	39	115	1	0	43-116
2-Fluorophenol	7	70	19	37	94	0	0	21-100
2,4,6-Tribromophenol	7	77	12	58	95	0	0	10-123
Nitrobenzene-d <sub>5</sub>	7	93	14	76	109	0	0	35-114
Phenol-d <sub>5</sub>	7	84	25	43	104	0	4	10-94
Terphenyl-d <sub>14</sub>	7	105	19	77	128	0	0	33-141

\* Number of routine field samples and field duplicates, not including blanks, matrix spikes, and recovery checks.

## Dioxin and Furans

Samples were collected during each of the Demonstration Tests to determine the presence of polychlorinated dibenzodioxins and polychlorinated dibenzofurans (PCDDs/PCDFs). PCDD/PCDF samples were collected because hexachlorobenzene, which is a potential PCDD/PCDF-forming compound under the thermal processing conditions encountered for these tests, was spiked into the feed soil. The sampling strategy was to analyze the stack and treated soil samples first to determine if these compounds were present. If PCDDs/PCDFs were detected in these matrices, then all of remaining samples would be analyzed. Since the samples that were initially analyzed only contained trace quantities of PCDDs/PCDFs, the remaining samples

were not analyzed. The results of all of the PCDD/PCDF samples for the stack gas and the treated soil are presented in Appendices C and E, respectively.

All solid and stack samples were analyzed following the procedures outlined in SW-846 Method 8290. Method 8290 is a high resolution mass spectrometry method for the detection of PCDDs/PCDFs. Method 8290 details the acceptance criteria and procedures used to determine the validity of the results.

For this project, quality control and calibration procedures were followed as outlined in the QAPP. Prior to the analysis of samples, the mass scale was calibrated using perfluorokerosene. An initial 5-point calibration was performed and passed the acceptance criteria of less than 25% RSD for the targeted compounds. Each day the calibration was verified by analyzing a check standard which agreed within 25 % of the initial calibration curve. Additional QC parameters included chromatography checks, retention time checks, and sensitivity checks. The results of these checks were satisfactory for all samples analyzed for these Demonstration Tests.

Blank samples were analyzed as part of the quality control efforts associated with PCDD/PCDF analyses of the solid and stack gas samples. Field blank samples indicated no significant quantities of target analytes. All values were below 150 pg with the exception of OCDD and OCDF detected at 1.2 and 0.29 ng, respectively. All of these compounds were detected in the laboratory blank except for 2,3,4,7,8-PeCDF which was detected at 7 pg. Method blanks associated with the soil samples showed targets below the acceptable level of one-third the calculated theoretical method quantification limit (TMQL), except for OCDD which is not required to follow the blank contamination criteria. Blanks analyzed with the stack gas samples showed 1,2,3,6,7,8-HxCDF, OCDD, 1,2,3,4,7,8-HxCDF and 1,2,3,4,6,7,8-HpCDF analytes detected above one-third the TMQL but below the target detection limit. The 2,3,4,6,7,8-HxCDF analyte was found in the blank at a level less than one-third the

TMQL. Blank results were, nonetheless, deemed acceptable by the analytical laboratory and indicate no significant impact on sample results.

Each sample was spiked with surrogate standards, alternate standards, and internal standards to monitor the quality of the results generated. For each sample extracted, nine carbon-labeled PCDD/PCDF congeners representing the tetra- through octa-chlorinated PCDDs/PCDFs were added to the sample. These standards measured the overall method efficiency and provided a correction for the unlabeled analog (isotope dilution mass spectrometry). Five surrogates and two alternate compounds were added during the sample preparation to measure efficiencies of extract preparation steps.

The summarized results of the surrogate and internal standards are presented in Table 39 for the treated soil samples and Table 40 for the stack gas samples. As noted in these tables, most of the recoveries were within the acceptance ranges. Recoveries of an internal standard that are not within the control does not necessarily indicate a problem since the corresponding target analyte is corrected for the recovery. Only erroneous recoveries would indicate a problem with the quantitations. Internal standard recoveries were satisfactory. Poor surrogate recoveries were noted for only a few analytes and were usually only slightly below or above acceptance limits. Based upon the sample results, the poor recoveries have little or no effect on overall data quality.

Duplicate samples for the stack emission were collected during Test 2. Duplicate samples for the treated soil were also collected and analyzed for each of the Demonstration Tests. Table 41 summarizes the results of the duplicates for the treated soil samples and Table 42 presents the results of the stack gas duplicates. These tables indicate that the majority of the duplicate results do not meet the QAPP objectives. Compounds that were beyond the control limits were most likely due to sample interferences since most of the hits were very low and close to the TMQL.

Table 39. PCDD/PCDF Internal and Surrogate Treated Soil Recoveries<sup>a</sup>

Compound	No. of Results <sup>b</sup>	Mean Recovery (%)	Std. Dev. (%)	Min (%)	Max (%)	No. of Results Outside Limits		Control Limits (%)		
						Low	High			
Surrogate Standards										
<sup>13</sup> C-HpCDF-789	8	80	20	46	107	0	0	40	-	130
<sup>13</sup> C-HxCDD-478	8	94	16	64	119	0	0	40	-	130
<sup>13</sup> C-HxCDF-478	8	79	17	50	106	0	0	40	-	130
<sup>13</sup> C-PeCDF-234	8	92	18	71	118	0	0	40	-	130
<sup>37</sup> Cl-TCDD	9 <sup>c</sup>	56	23	28	97	3	0	40	-	130
Alternate Standards										
<sup>13</sup> C-HxCDF-234	8	84	18	49	111	0	0	25	-	130
<sup>13</sup> C-HxCDF-789	8	81	18	49	106	0	0	25	-	130
Internal Standards										
<sup>13</sup> C-2378-TCDD	9 <sup>c</sup>	59	24	29	102	2	0	40	-	130
<sup>13</sup> C-2378-TCDF	9 <sup>c</sup>	53	21	24	85	3	0	40	-	130
<sup>13</sup> C-PeCDF-123	8	80	29	45	125	0	0	40	-	130
<sup>13</sup> C-PeCDD-123	8	98	32	67	149	0	2	40	-	130
<sup>13</sup> C-HxCDF-678	8	79	24	48	127	0	0	40	-	130
<sup>13</sup> C-HxCDD-678	8	84	22	49	124	0	0	40	-	130
<sup>13</sup> C-HpCDF-678	8	74	16	44	89	0	0	25	-	130
<sup>13</sup> C-HpCDD-678	8	87	20	51	112	0	0	25	-	130
<sup>13</sup> C-OCDD	8	94	24	54	118	0	0	25	-	130

<sup>a</sup> Includes one treated soil core water sample.

<sup>b</sup> Number of routine field samples and field duplicates analyzed. Does not include blanks, matrix spikes, or recovery checks.

<sup>c</sup> Includes confirmation as well as full screen analysis results.

Table 40. PCDD/PCDF Internal and Surrogate Gas Recoveries

Compound	No. of Results <sup>a</sup>	Mean Recovery (%)	Std. Dev. (%)	Min (%)	Max (%)	No. of Results Outside Limits		Control Limits (%)		
						Low	High			
Stack Gas										
Surrogate Standard										
13C-HpCDF-789	4	97	14	82	111	0	0	70	-	130
13C-HxCDD-478	4	167	78	108	279	0	2	70	-	130
13C-HxCDF-478	4	118	7	111	126	0	0	70	-	130
13C-PeCDF-234	4	108	8	98	118	0	0	70	-	130
27Cl-TCDD	8 <sup>b</sup>	104	8	95	116	0	0	70	-	130
Alternate Standards										
13C-HxCDF-234	4	102	19	79	120	0	0	40	-	130
13C-HxCDF-789	4	72	16	52	88	0	0	40	-	130
Internal Standards										
13C-2378-TCDD	8 <sup>b</sup>	77	39	34	151	2	1	40	-	130
13C-2378-TCDF	8 <sup>b</sup>	74	33	35	131	2	1	40	-	130
13C-PeCDF-123	4	65	16	42	77	0	0	40	-	130
13C-PeCDD-123	4	73	32	53	120	0	0	40	-	130
13C-HxCDF-678	4	82	8	75	94	0	0	40	-	130
13C-HxCDD-678	4	84	41	36	135	0	1	40	-	130
13C-HpCDF-678	4	76	7	71	86	0	0	25	-	130
13C-HpCDD-678	4	74	17	56	94	0	0	25	-	130
13C-OCDD	4	59	30	28	86	0	0	25	-	130

<sup>a</sup> Number of routine field samples and duplicates analyzed. Does not include blanks, matrix spikes, or recovery checks.

<sup>b</sup> Includes confirmation as well as full screen analysis results.

Table 41. PCDD/PCDF Duplicate Soil Sample Results

Compound	Primary (ppt)	Duplicate (ppt)	RPD	Objectives (RPD)
Treated Soil - Test 1				
1234678-HpCDD	0.41 B	0.37 B	10.26	25
OCDD	15.00 B	5.30 B	95.57	25
2378-TCDF	0.21 B	.	NC	25
234678-HxCDF	0.27 B	0.31 B	13.79	25
1234678-HpCDF	0.33	.	NC	25
OCDF	2.00 B	0.49 B	121.29	25
Total PeCDD	0.86	.	NC	25
Total HxCDD	.	0.35	NC	25
Total HpCDD	0.41	0.37	10.26	25
Total TCDF	0.61	1.00	48.45	25
Total HxCDF	0.27	0.31	13.79	25
Total HpCDF	1.30	.	NC	25
Treated Soil - Test 2				
1234678-HpCDD	1.30 B	.	NC	25
OCDD	21.10 B	2.60 B	156.12	25
2378-TCDF	0.23 B	0.27 B	16.00	25
234678-HxCDF	.	0.32 B	NC	25
OCDF	1.90 B	.	NC	25
Total PeCDD	7.30	3.50	70.37	25
Total HxCDD	.	0.57	NC	25
Total HpCDD	2.40	.	NC	25
Total TCDF	0.23	0.27	16.00	25
Total PeCDF	0.34	0.15	77.55	25
Total HxCDF	6.80	0.32	182.02	25
Treated Soil - Test 3				
123678-HxCDD	0.22	0.11	66.67	25
123789-HxCDD	0.23	.	NC	25
1234678-HpCDD	2.90 B	0.77 B	116.08	25

(cont.)

Table 41. (Continued)

Compound	Primary (ppt)	Duplicate (ppt)	RPD	Objectives (RPD)
Treated Soil - Test 3				
OCDD	14.20 B	12.50 B	12.73	25
2378-TCDF	0.20 B	0.35 B	54.55	25
23478-PeCDF	0.16		NC	25
123478-HxCDF	0.28	0.08	111.11	25
123678-HxCDF	0.10	0.04	85.71	25
234678-HxCDF	0.81	0.33 Q	84.21	25
1234678-HpCDF	1.00	0.18	138.98	25
1234789-HpCDF	0.34	0.06	140.00	25
OCDF	2.20	1.10	66.67	25
Total TCDD		0.17	NC	25
Total PeCDD	0.21	0.28	28.57	25
Total HxCDD	0.50	0.15	107.69	25
Total HpCDD	2.90	1.30	76.19	25
Total TCDF	0.20	0.35	54.55	25
Total PeCDF	0.47	0.08	139.29	25
Total HxCDF	1.50	0.38	119.15	25
Total HpCDF	2.30	0.50	128.57	25

\* - Indicates that this compound was not detected.

B - Indicates that this compound was detected in a blank.

Q - Indicates that this is an estimated value.

NC -Not calculated.



Table 42. PCDD/PCDF Duplicate Soil Sample Results

Compound	Primary (pptV)	Duplicate (pptV)	RPD	Objectives (RPD)
Stack Gas - Test 2				
2378-TCDD	2.89E-04	2.98E-04	3.07	25
12378-PeCDD	3.26E-04	2.69E-04	19.16	25
123478-HxCDD	1.49E-03 Q	2.45E-04	152.29	25
123678-HxCDD	5.95E-04	4.91E-04	19.48	25
123789-HxCDD	2.68E-03 Q	7.36E-04	113.82	25
1234678-HpCDD	4.65E-03 B	3.61E-03 B	25.18	25
OCDD	2.10E-02 B	1.61E-02 B	26.42	25
2378-TCDF	6.46E-03	5.64E-03	13.55	25
12378-PeCDF	2.39E-03	1.69E-03	34.31	25
23478-PeCDF	3.76E-03	2.82E-03	38.57	25
123478-HxCDF	9.31E-03	7.16E-03	26.11	25
123678-HxCDF	3.72E-03	2.81E-03	27.87	25
234678-HxCDF	4.97E-03 B	4.35E-03 B	13.30	25
123789-HxCDF	6.21E-04	2.05E-04	100.73	25
1234678-HpCDF	1.17E-02 B	9.37E-03 B	22.12	25
1234789-HpCDF	1.71E-03	1.17E-03	37.50	25
OCDF	6.29E-03 B	6.48E-03 B	2.98	25
Total TCDD	3.61E-04 Q	2.98E-04	19.12	25
Total PeCDD	1.96E-03 Q	1.08E-03 Q	57.89	25
Total HxCDD	1.49E-02 Q	5.15E-03	97.26	25
Total HpCDD	9.57E-03	7.44E-03	25.04	25
Total TCDF	2.89E-02 Q	2.07E-02	33.06	25
Total PeCDF	3.08E-02	2.48E-02 Q	21.58	25
Total HxCDF	3.41E-02	2.38E-02	35.58	25
Total HpCDF	1.96E-02	1.55E-02	23.36	25

B - Indicates that this compound was detected in a blank.

Q - Indicates that this value is an estimated value.

Several of these compounds were also found in method blanks where indicated. Based upon the low sample results, no significant impacts on data quality arise from the duplicate results.

### Stack Gas Particulates

Particulate data was collected for each of the Demonstration Tests. Particulate loading was determined using a gravimetric procedure. For Test 1, a primary and a duplicate sample were collected. Precision, as indicated by the RPD of the duplicates, was measured at 35 percent. This exceeded QAPP control limits. This is an indication that the particulates within the stack were not well-mixed or that there was a problem in the collection of an isokinetic sample. This is in agreement with the high RPDs associated with the MMT duplicate samples which indicated the particulate distribution was not uniform within the stack. As previously mentioned, this may have been a result of problems associated with the blower which may have caused irregular flow patterns in the stack. Data users should be cautioned that the values associated with the particulate measurements may have an error range of  $\pm 35$  percent of the measured value. As noted by the particulate summary results, the potential error associated with these data has no effect on conclusions pertaining to particulate emission since the reported values were well above the regulatory limits. This is discussed in further detail in Section 6.

One field blank (filter) for particulates showed 2.3 mg of loading; the second blank showed no particulate loading. These values are very small in comparison to the catch on actual sample filters and, therefore, have no significant impact on sample results.

## Chloride

Chloride content in the stack gas was measured for each test. Chloride samples were analyzed using ion chromatography. Results of multipoint calibrations and calibration checks met laboratory specifications. Three samples were spiked and analyzed. Spike recoveries were at 95, 89, and 83 percent with a control range of 75 to 125 percent. Preparation blanks prepared for and analyzed with these samples were free of contamination. A laboratory duplicate sample was analyzed, these results were compared against the original analysis and yielded an 8 percent RPD. Quality control parameters for these measurements indicate no limitations on measured results.

## Continuous Emission Monitoring

Flue gas was monitored continuously for O<sub>2</sub> by EPA Method 3A, NO<sub>x</sub> by EPA Method 7E, SO<sub>2</sub> by EPA Method 66, CO by EPA Method 10, CO<sub>2</sub> by EPA Method 3A, and THC by EPA Method 25A. Samples were collected from the stack through stainless steel tubing and passed through a series of impingers for gas conditioning. Sample gas then passed to a manifold for distribution to each of the monitors.

Each analyzer was calibrated at the beginning and end of each Demonstration Test. The calibration consisted of three points which bracketed the average concentration of interest for most compounds. The NO<sub>x</sub> calibration only went up to 888 ppm, which was lower than the average concentration detected for each Demonstration Test. For Tests 2 and 3, a cylinder that contained 5,070 ppm was used to check the upper limit of the calibration (it should be noted that this is close to reported NO<sub>x</sub> concentrations). Results of these checks indicated that the analyzer measurements were within an average of 5% of the known cylinder value. Calibration checks, zero, and drift checks were acceptable for all of the gases for each demonstration test.

After calibrating the analyzers for Test 1, audit cylinders were analyzed to check the calibrations for NO<sub>x</sub> and CO. Audit cylinders were EPA Protocol No. 1 certified. Cylinder concentrations were certified at 2,351 ppm for NO<sub>x</sub> and 40.4 ppm for CO. Results of these checks showed a RPD of 2 to 3 percent for the NO<sub>x</sub> and a RPD of 5 percent for the CO. The results of the QC checks associated with the CEMs indicate that the data obtained are of acceptable quality.

### **Physical Property Measurements**

Demonstration Test soil samples were analyzed for higher heating value (HHV), bulk density, and moisture (all non-critical), as well as chloride content. Quality control efforts for the HHV consisted of the analysis of known standards three times. Recoveries of these checks were 99, 99, and 100 percent of the theoretical values. Three duplicate sample to measure the precision of the measurements all yielded RPD of 0 percent. Four duplicates were analyzed to provide QC for the density measurements. These results yielded identical results for two samples and a RPD of 0.4 and 2.26 percent for the other two. Moisture measurements were performed twice on a sample and yielded identical results. Quality control for the chloride measurements consisted of a spiked sample and the analyses of a reference material. The spike recovery was 85 percent and the reference material was 106 percent of the theoretical value. These results imply that there are no limitations associated with the physical properties measurements.

### **AUDIT FINDINGS**

At the start of the Demonstration Test, a field Technical Systems Review (TSR) was conducted by an EPA RREL QA contractor. The results of this audit were satisfactory with only minor concerns which were corrected in the field. Project organization and QA management were reviewed and deemed satisfactory for this project. The

auditors also reviewed the sample custody and sample shipping. All procedures were being performed in accordance with the QAPP and pertinent DOT regulations.

A review of the solid and liquid sampling revealed some concerns with the compositing of the solid samples. Unequal portions of the feed soil were being collected for compositing and placed in 1-gallon glass jar. Since it was necessary to collect a minimum of 3.5 liters of sample for all of the required analytical parameters, there was some concern that the sample could not be mixed as specified in the QAPP. To remedy these problems, a graduated sampling device was obtained and equal quantities of sample were collected for each sampling episode. To mix the samples properly, at the end of the test, the composite sample was divided into two 1-gallon jars. Each jar was thoroughly mixed and equal quantities of sample were obtained from the jars to fill the individual containers required for each analysis. Instructions were sent to the laboratory to ensure that the samples were mixed thoroughly to avoid sampling only one of the stratified layers.

The audit also consisted of a check of the stack sampling methods. All of the sampling trains were set-up according to the referenced methods and the QAPP. Some slight modifications that would not significantly impact sample results were noted with the gas canister samples. One calibration form was missing for one of the Method 5 consoles; the form was received by fax later that day. A performance evaluation audit was conducted for the NO<sub>x</sub> and CO analyzers. The results of these audits were acceptable for each gas. There was some moisture, which may have biased the results, noted in the sample line for the THC analyzer. It was suggested that THC data be obtained from MSE since they had recently passed a previous RREL TSR. Since THC was not a critical parameter for this demonstration, no major corrective actions were taken. Some concern was also expressed regarding the data storage of the CEMs since there was no hard copy or data backup if the CEM computer crashed. This was remedied by supplying a printer and backing up the data on a floppy disk.

The auditors held a debriefing meeting to discuss their findings with project management and completed a corrective action recommendations (CAR) form.

The laboratory audit was conducted on August 15, 1991. The results of this audit were judged by RREL's auditor as satisfactory. This was approximately two weeks after the last Demonstration Test sample was received. The audit reviewed QAPP specific procedures relating to the analysis of these samples. The auditor reviewed procedures, documentation, and sample data in several areas of the laboratory. At the completion of the audit, a CAR identified only two items of concern: (1) it was not apparent that the concentration of the special spiking solutions used for the SW-846 Method 8270 samples had been verified; and (2) while in the ion chromatography lab, where the chloride analyses were being performed, chloride duplicates were not run at the frequency specified in the QAPP. Additional duplicates were performed on the chloride samples as per the QAPP to correct the problem identified in the audit. Chloride data quality was not affected.

An additional laboratory audit, specifically for the VOST analyses was conducted on August 8, 1991. This audit was performed separately because another subcontractor laboratory was analyzing these samples. the results of the audit were also judged to be "satisfactory". Changes in procedures which resulted from auditor's recommendations included 1) using a different standard for calibration to improve the response factor precision, and 2) performing a method detection limit study. These items were addressed and appropriate corrective action was taken as necessary. Because of this response, data quality was not affected and the data was deemed satisfactory for accomplishing project objectives. Copies of this and all other audit reports can be found in Appendix L.

## **MODIFICATIONS AND DEVIATIONS FROM THE QAPP**

Because a substantial amount of time passed between the original approval of the QAPP and the Demonstration Test, it was necessary to make some modifications to reflect current laboratory practices. Changes were also made based upon experience gained through pre-demonstration activities. Below is a summary discussion of each of these changes and how they have impacted the overall data quality.

- The target lists for SW-846 Methods 8240 and 8270 were modified to reflect the compounds that were currently being analyzed by the laboratory on a routine basis. Since the compounds of interest were still included, and the detection limits were not affected, semivolatile data quality was not impacted.
- Microwave digestion was used for metal samples rather than standard SW-846 procedures. During pre-demonstration activities, it was noted that the SW-846 procedures were not capable of completely digesting the treated soil samples. A microwave digestion that incorporated the use of hydrofluoric acid to better digest the treated soil was used on both the feed and treated soil to provide consistency. The basic procedure was a modification of Method 3051 which has not yet been EPA-approved. This procedure adequately fulfilled the objectives of the Demonstration Test.
- Lead and thallium were analyzed using ICP rather than the graphite furnace method. The quality of these data were not impacted by this change.
- For SW-846 Methods 8240 and 8270, the top 20 Tentatively Identified Compounds (TICs) were tentatively identified and semi-quantitated for each sample.

- Sixty grams of the feed soil were analyzed using SW-846 Method 3550. This was done to obtain a more representative sample of the feed. The hexachlorobenzene was spiked in the mixture as a solid crystal and may have been subjected to high variability if a small extract aliquot was taken. Because a large sample size was used, these samples required large dilutions and surrogate spiking at levels similar to that of the hexachlorobenzene spike. Three surrogates, d<sub>5</sub>-nitrobenzene, 2-fluorobiphenyl, and d<sub>3</sub>-methylnaphthalene were chosen for surrogate spiking because of their ability to represent the compounds of interest in the feed and their availability. These modifications were made to improve the quality of data obtained from the feed samples.
- Matrix spikes for metals were modified for zinc. Soil that did not contain the zinc oxide spike was spiked at the feed concentration (28,000 ppm) to provide matrix spike information. The unspiked feed (no zinc) was also spiked with arsenic, cadmium, chromium, and lead at levels approximately five times the concentrations of the feed samples.
- Aluminum, calcium, iron, magnesium, and potassium were added to the target list for all metal parameters.
- The draft Method 5041 was used in place of SW-846 Method 5040 for VOST analyses. Method 5041 incorporates the use of a capillary column. VOST data was not impacted by this change.
- The scrubber and stack samples were spiked with three additional surrogates because of the anticipated matrix recovery problems associated with the routine SW-846 Method 8270 acid surrogates. The three surrogates (C<sup>13</sup>-pentachlorophenol, 2,4-dinitrophenol-d<sub>3</sub>, and 4,6-dinitro-2-methylphenol-d<sub>2</sub>) were selected based upon studies prompted by pre-demonstration results. Adding these surrogates enhanced recovery information for these matrices.



- Matrix spikes for the feed soil were modified. It was not feasible to spike the routine spiking compounds at levels which would be detectable after diluting the extracts to bring the hexachlorobenzene into the calibration range. Soil that did not contain the hexachlorobenzene spike was spiked at the feed concentration, 1,000 ppm of hexachlorobenzene, to provide matrix spike information. This provided an assessment of the recoverability of the hexachlorobenzene in the feed soil. These samples were to be re-extracted with fresh solvent to assist in the evaluation of the extraction efficiencies of the feed samples.
- Composite samples of the feed soil for semivolatile and metal analyses were collected instead of discrete samples as specified in the QAPP. This was done to ensure representativeness of the feed soil samples.
- Scrubber solid samples were not collected and analyzed as specified in the QAPP. Insufficient quantities of solids were generated during the demonstration to provide enough samples mass for the specified analyses.
- Gas canister samples of the stack gas that were collected for volatile organics compound analysis were not analyzed. This was an option provided in the QAPP in the event that the VOST cartridges were overloaded. VOST data was determined to be of sufficient quality such that the analysis of these samples was not warranted.
- The only samples analyzed for PCDDs/PCDFs were the stack gas and the treated soil samples. This was an option provided by the QAPP in the event that no significant PCDD/PCDF compounds were detected in these samples. Since no significant contamination was detected, the remaining PCDD/PCDF samples were not analyzed.

- Duplicate sample analysis for each test series, as outlined in the QAPP would not be required for volatiles, semivolatiles, metals, and TCLP. During the laboratory audit, it was noted that this requirement was redundant since these methods required matrix spike analysis for each matrix.

## SPECIAL STUDIES

There were only a few special studies performed in support of this demonstration. Studies were conducted to improve procedures used to measure analytical parameters and ensure that the analytes were being properly quantitated. Some of these studies were prompted by the results of the pre-demonstration test. The first study related to the recovery of surrogates in the scrubber solution.

It was noted during the pre-demonstration activities that the routine acid surrogates used from SW-846 Method 8270 (phenol- $d_5$ , 2-fluorophenol, and 2,4,6-tribromophenol) were not recovered in the XAD resin and scrubber liquor extracts. It was postulated that these surrogates were being nitrated from the  $NO_x$  generated by the process. Additionally, the scrubber solutions contained sodium hydroxide which may have also caused chemical reactions to occur with the acid surrogates. Four experiments were conducted to evaluate the reason for poor surrogate recovery in these samples and to recommend a course of action for the demonstration test. The four experiments are summarized below:

- 1) A solution of nitric acid was formulated to attain a pH of less than 2, (similar to the scrubber pH). This solution was then slowly passed through XAD resin spiked with a base/neutral/acid (BNA) matrix spike solution. The spike included pentachlorophenol (PCP) and 2-methylnaphthalene, which were two compounds that may have been present in these matrices during demonstration testing. After spiking, the XAD was collected and analyzed. Spiked XAD resin

and nitric acid solution were analyzed separately along with an XAD resin blank to assure proper QA/QC.

**RESULTS:** Nitric acid appeared to have very little effect on the spiked XAD. Very small quantities of any of the compounds of interest were transferred to the eluent and, while recoveries of all BNA spikes from the XAD resin were low, all were within acceptable ranges. Surrogate spike recoveries were also acceptable. Matrix compounds spiked separately on the resin and in the nitric acid solution indicated that extraction of these compounds was not a problem. A separate analysis of blank XAD resin showed that the resin was free of any contamination.

- 2) Scrubber liquor, obtained from the developer, was spiked with the BNA matrix spike. This was performed in duplicate to determine the precision of the data. Extraction and analysis were conducted by SW-846 Method 3520 and SW-846 Method 8270, respectively.

**RESULTS:** Recovery results of the matrix spike for phenol, 2-chlorophenol, and 4-chlorophenol and the acid surrogate spikes were 0 percent. Pentachlorophenol (PCP) recovery averaged around 25 percent. Although this is a generally low recovery, it is still considered acceptable. Surprisingly, 2-nitrophenol, and 2,4-dinitrophenol were detected by SW-846 Method 8270. These compounds were not spiked in the study. It was concluded that the disappearance of the spike compounds and the appearance of the nitrated compounds was caused by the presence of  $\text{NO}_x$  in the scrubber solution which apparently reacted with the spikes.

- 3) Additional scrubber liquor was spiked with the BNA matrix solution and two types of analyses were performed on both unspiked and spiked scrubber liquor. The two analytical methods were SW-846 Method 8270 and HPLC for PCP

analysis only. In addition, a GC/MS search was performed on both spiked and unspiked scrubber liquor to tentatively identify and quantify the next 20 highest peaks which were not part of the 8270 target list.

RESULTS: Results of Study (2) suggested that the nitrification phenomena required further investigation. Results of this study were similar to that of the second experiment. Several nitro-phenolic compounds were detected in the spiked solution while no significant amounts of these compounds were detected in the unspiked samples. HPLC analysis for PCP showed that this compound remained stable, as would be expected, since it has no open sites for nitrification. The purpose of HPLC analysis was to eliminate the question of extraction efficiency; verification that all of the PCP remained was required. The results of the TICs found other nitrated phenolic compounds which were not present in the unspiked liquor. The presence of the nitrated TICs suggested that the matrix spikes were being nitrated.

- 4) The final experiment was similar to the first experiment, except instead of using nitric acid, scrubber liquor was used in its place.

RESULTS: After determining that nitrification had occurred in solution it was necessary to determine if this sample effect could take place on XAD resin. To test this, scrubber liquor was poured through spiked resin, then the resin and eluent were collected and analyzed. While this is not exactly the same conditions as collecting a sample with the MM5 train on site, it was expected to give an indication of possible reactions. Carbon-labeled PCP was also spiked into these samples to track PCP reactions. (Note that PCP was originally a target analyte when these experiments were performed.) Results of these samples indicated that nitrification could occur on the XAD. The experiments suggested that PCP probably remained stable and therefore would be detected in both the scrubber solution and gas.

These studies concluded that the routine acid surrogates for SW-846 Method 8270 would not be detected in the scrubber liquors of the XAD resin samples. Therefore it was necessary to select surrogates that would be stable in these matrices. Surrogates were selected for spiking and modifications to the QAPP were made accordingly before the demonstration test began. Additional surrogates used for the demonstration have been discussed in earlier portions of this section. As previously noted, pentachlorophenol was originally a target analyte but was later not considered critical to accomplishing project objectives when a different soil was used for the demonstration. Hence, the recovery of acid compounds became less critical

The second study related to the digestibility of the treated soil. It was noted for analyses conducted during pre-demonstration that the normal SW-846 digestion procedures were only able to leach, and not completely digest the treated soil. This indicated that these procedures were inadequate for the digestion of this matrix. This was obvious from the sample results and from the appearance of the remaining sample in the digestate.

Experiments were conducted using a lithium-borate digestion which was better able to breakdown the glassified soil matrix. Since these earlier experiments, microwave digestion techniques were developed and appeared to be a superior digestion procedure. At the time of the Demonstration Tests, the microwave digestion procedure was not an EPA-approved method. The microwave digestion was a modification of SW-846 method 3051. The modification to the method required the use of hydrofluoric acid in combination with hydrochloric acid and nitric acid to completely dissolve the sample rather than to leach the sample. A total digestion was necessary for the analyses of the treated soil due to the limited leachability of metals in this matrix. The method was used for the feed soil samples so values could be comparable when performing a material balance.

One final study was conducted to determine the effectiveness of the feed soil spiking. After spiking was completed, samples were collected and analyzed for semivolatiles, TCLP semivolatiles, and TCLP metals. The results of the semivolatile analyses assisted in identifying special sample requirements for surrogate spiking, sample dilution, and extract clean-up to remove hydrocarbon interference from the diesel fuel.

Results of the semivolatile analysis (with no extract cleanup) found hexachlorobenzene at 294 ppm, naphthalene at 71 ppm, and 2-methylnaphthalene at 134 ppm. TCLP semivolatile results identified four leachable constituents: hexachlorobenzene (34 ppm), 2-methylnaphthalene (21 ppm), naphthalene (10 ppm), and phenanthrene (5 ppm). The TCLP metals analysis identified only three metals: barium at 54 ppb, cadmium at 80 ppb, and zinc at 960 ppm. Results of these studies indicated that the spiking levels were sufficient to evaluate DRE for hexachlorobenzene and leachability of zinc.

Upon learning of the preliminary results of the semivolatile leachate samples for the Demonstration Tests, the investigators were surprised to learn that hexachlorobenzene was not detected in the feed soil leachate samples. To verify these results fresh samples were collected of the remaining feed material, before it was shipped for disposal. These samples were subjected to TCLP and analyzed for semivolatiles by an independent laboratory. The results of this second analyses confirmed that the hexachlorobenzene was no longer leachable, hence no further investigations were conducted.

## **SAMPLE HOLDING TIMES**

Holding times were specified in the QAPP for each analytical parameter. Most samples were analyzed within the required holding time with the following exceptions:

**Description:** Semivolatile feed soils SW-846 Method 8270, extract holding time violated by 2 days.

<b>Samples:</b>	#113 - Test 1 Feed	#243 - Test 2 Feed
	#116 - Test 1 Feed	#206 - Test 2 Feed
	#117 - Test 1 Feed	#205 - Test 2 Feed
	#127 - Test 1 Feed Blank	#207 - Test 2 Feed
	#166 - Test 1 Feed	#208 - Test 2 Feed
	#167 - Test 1 Feed	#204 - Test 2 Feed
	#159 - Feed Matrix Spike	
	#160 - Feed Matrix Spike Dup	

**Cause:** Due to the high levels of hexachlorobenzene in the feed samples and the potential interference of the diesel, the feed sample extracts went through extract clean-up. Upon analyzing these samples, it was discovered that the hexachlorobenzene was not detectable. Investigations within the laboratory determined that the neutral alumina removed the hexachlorobenzene from the extract. The fraction of the extract that remained, which went through GPC and not the neutral alumina, was analyzed and the hexachlorobenzene was detected. Several shots on the mass spectrometer were required to bring the results within the calibration range and obtain surrogate recovery information. These efforts took some time within the laboratory and valid results were not obtained until two days past the extract holding time.

**Impact:** In viewing the results of the Test 3 feed soil samples, which were analyzed within the required extract holding time, these results agree very well. It appears that the integrity of the sample extract was not

impacted and the results for these samples are useable for calculating DREs.

## CONCLUSIONS AND LIMITATIONS OF DATA

Upon review of all data related to this project, along with the QC associated with these results, the data appears to be of sufficient quality to provide proper evaluation of the Demonstration Test objectives. Overall, most of the QAPP requirements were met with regard to precision, accuracy and completeness. There were very few samples that were lost due to holding time difficulties, laboratory preparation, and poor quality control results.

Among parameters of secondary critical concern were the semivolatile analyses of the scrubber samples. As noted during the discussion of these data, poor and highly variable recoveries were noted for the 8270 and special acid surrogates for some of these samples. These results imply that acid compounds (phenols) may have been chemically attacked causing new compounds to be formed which are not part of the 8270 target list. In addition, there may have been analytical problems in the laboratory. Certainly phenol- $d_5$  and 2-Fluorophenol were most likely nitrated and therefore not detected as suggested by pre-demonstration studies. Whatever the reason for some samples showing low recoveries the associated semivolatile scrubber samples contained a very limited amount of low level target hits, hence these poor recoveries have limited impact on this project.

One of the limitations that may be associated with these results relates to the apparent variability between the duplicate samples collected of the stack gases. It was noted in the particulate and metal duplicate samples that there was some variability associated with these measurements. As mentioned in the discussion of these results, this variability may be due to low stack flowrates which were caused



by a faulty blower during Tests 1 and 2. The blower was noted as having flow problems and trouble maintaining the required negative pressure in the reactor chamber. This could have caused interruptions in stack flow patterns that may have influenced these samples. Other sources of error in the stack duplicate samples may include sample train installation, sample train maintenance or filter changes, and the differences in sampling intervals. If these variables were not exactly the same for the primary and duplicate trains, some differences can be expected in the results. Although there were problems identified with these duplicate samples, the overall quality of the data is adequate for the objectives of this project based upon data used for evaluating furnace performance.

The stack data for all parameters appear to be satisfactory. Stack flowrate measurement for all of the sample trains during each test were in close agreement with each other. No significant problems were noted during the review of the data. VOST samples that were collected were provided adequate data such that the gas canisters did not require analysis. There were only a few target compounds in the VOST tubes that exceeded the calibration range of the GC/MS. These compounds were benzene and chloromethane. It is important to note that VOST data that exceeded the calibration range was very rare. These few data points that exceeded the calibration range did not saturate the GC/MS, hence the quantitations associated with these values appear to be reasonable. Due to the limited volatile emissions for this process, these few data points do not limit the use of these data.

All data presented in this report have **NOT** been corrected for field or laboratory blank contamination. Where appropriate, the raw data have been flagged when laboratory method blank contamination is suspected as interfering with sample results. Field blank contamination is not denoted on the raw data. Field blank contamination of naphthalene was, however, significant for the stack gas samples. Similar values of naphthalene were detected in the field blank as in routine samples. Naphthalene data in the semivolatile emission samples is highly suspected as field contamination and,

therefore, was not used in evaluation of project objectives. Small quantities of several metals were found in the field blank for the MMT. The source of this contamination has not been identified. Most of the contaminant concentrations were small in respect to routine samples and therefore has a limited impact on data quality. Other blank contamination was limited in scope and has no direct impact on data associated with this project.

As noted in the Appendices and the Report, all data has been converted to a lbs/100 lbs feed basis where appropriate. This was done to facilitate the engineering review of these data. Feed rates for each test were targeted at 120 lbs/hr. Because of process interruptions and unforeseen events, this feed rate was not precisely achieved for each test. Feed rates were determined by summing the total feed material fed to the unit divided by the total time required to feed this material minus any process interruptions. These parameters were obtained by the evaluation of field notes, process logs, and field observation. There may be a limited amount of error associated with this evaluation which does not significantly affect the calculated results.

No information was obtained for the second extraction of a feed sample to determine if hexachlorobenzene was completely extracted from the soil during normal extraction procedures. This procedure was placed in the QAPP as a precautionary measure to evaluate extraction efficiency if the hexachlorobenzene was recovered poorly from feed samples. The laboratory did extract a sample twice, but accidentally combined the two extracts and analyzed them as a single sample. Fortunately, hexachlorobenzene was recovered in the routine feed samples at anticipated pre-spiked levels and the results of this study were not necessary. Therefore data quality has not suffered from the loss of this study.

In closing, data generated for this project is rated as satisfactory and is of sufficient quality to provide for the proper evaluation of test objectives.