HARBAUER SOIL WASHING/VACUUM-DISTILLATION SYSTEM

HARBAUER GmbH & COMPANY KG FACILITY

MARKTREDWITZ, GERMANY

EPA - BMBF BILATERAL SITE DEMONSTRATION

INNOVATIVE TECHNOLOGY EVALUATION REPORT

AUGUST 1996

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Notice

The information in this document has been prepared for the U.S. Environmental Protection Agency's (EPA) Superfund Innovative Technology Evaluation (SITE) program in accordance with a bilateral agreement between EPA and the Federal Republic of German Ministry for Research and Technology (BMBF), and under Contract No. 68-C5-0037 to PRC Environmental Management, Inc. This document has been subjected to EPA's peer and administrative reviews, and has been approved for publication as an 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 Superfund Amendments and Reauthorization Act (SARA) of 1986. The program is administered by the U.S. Environmental Protection Agency (EPA) Office of Research and Development (ORD). The purpose of the SITE program is to accelerate the development and use of innovative remediation technologies applicable to Superfund and other hazardous waste sites. This purpose is accomplished through technology demonstrations designed to provide performance and cost data on selected technologies.

This technology demonstration, conducted under the SITE program, evaluated the Harbauer soil washing/vacuum-distillation technology developed by Harbauer GmbH & Co. KG, Berlin, Germany. The demonstration was conducted at the Harbauer facility located in Wölsau near Marktredwitz, Landkreis (County of) Wunsiedel, State of Bavaria, Germany. The Harbauer technology demonstration evaluated the treatment of soils from the Chemische Fabrik Marktredwitz (CFM) site, a former chemical formulation plant, located in Marktredwitz, Germany. Soils from the CFM site are contaminated with high levels of mercury, as well as other metals and organic contaminants. Based on an agreement between Harbauer and the Federal Republic of German Ministry for Research and Technology (BMBF), and due to budgetary limitations, this demonstration focused only on the technology's ability to remove mercury from soil. This Innovative Technology Evaluation Report (ITER) provides an interpretation of the data collected during the Harbauer SITE demonstration and discusses the potential applicability of the technology to other contaminated sites.

A limited number of copies of this report will be available at no charge from EPA's Center for Environmental Research Information, 26 Martin Luther King Drive, Cincinnati, Ohio 45268, (513) 569-7562. Requests should include the EPA document number found on the report's cover. When the limited supply is exhausted, additional copies can be purchased from the National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, Virginia 22161, (703) 487-4600. Reference copies will be available at EPA libraries in the Hazardous Waste Collection.

E. Timothy Oppelt, Director
National Risk Management Research Laboratory

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Acronyms And Abbreviations

ARARs	Applicable or relevant and appropriate requirements
ArGe	Arbeitgemeinschaft focon-Probiotec
ASTM	American Society for Testing and Materials
BMBF	Federal Republic of German Ministry for Research and Technology
°C	Degrees Celsius
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFM	Chemische Fabrik Marktredwitz
CFR	Code of Federal Regulations
DM	Deutsche Mark
m^3	Cubic meter
EPA	United States Environmental Protection Agency
g/g	Grams per gram
hPa	Hectopascals
ITER	Innovative Technology Evaluation Report
m	Meter
m^3	Cubic meter
m^3/hr	Cubic meters per hour
MCL	Maximum contaminant level
mg/kg	Milligrams per kilogram
mg/L	Milligrams per liter
mL	Milliliter
mm	Millimeter
mt/hr	Metric tons per hour
NPL	National Priorities List
NRMRL	National Risk Management Research Laboratory
ORD	U.S. EPA Office of Research and Development
PRC	PRC Environmental Management, Inc.
PSD	Particle size distribution
QA/QC	Quality assurance and quality control
QAPP	Quality Assurance Project Plan
RCRA	Resource Conservation and Recovery Act
RPD	Relative percent difference
SARA	Superfund Amendments and Reauthorization Act (of 1986)
SITE	Superfund Innovative Technology Evaluation
TCLP	Toxicity Characteristic Leaching Procedure
æg/L	Micrograms per liter
æg/NCM	Microgram per normal cubic meter
æm	Micron
UCL	Upper confidence limit

Conversion Table
(Metric to English Units)

To Convert	Into	Multiply By
Centimeters	Feet	3.28×10^{-2}
Centimeters	Inches	0.39
Cubic meters	Cubic feet	35
Cubic meters	Gallons	264
Cubic meters	Cubic yards	1.31
Degrees Celsius	Degrees Fahrenheit	add 17.8 and multiply by 1.80
Hectopascals	Atmosphere	9.86×10^{-4}
Kilograms per square meter	Pounds per square inch, absolute	1.42×10^{-3}
Kilograms	Pounds	2.21
Kilograms per liter	Pounds per cubic foot	12.8
Kilometers	Miles (statute)	0.62
Liters	Gallons	0.26
Liters per second	Cubic feet (standard) per minute	2.12
Meters	Feet	3.28
Millimeters	Inches	0.039
Square meters	Square feet	10.8

Acknowledgments

This report was prepared under the direction of Mr. Donald Sanning, the EPA SITE project manager at the National Risk Management Research Laboratory (NRMRL) in Cincinnati, Ohio. This report was prepared by Mr. Roger Argus and Mr. John Menatti of PRC Environmental Management, Inc. (PRC). Contributors to, and reviewers of, this report were Ms. Ann Leitzinger of NRMRL, Mr. Burkhard Heuel-Fabianek and Mr. Kai Steffens of Probiotec, Dr. Reiner Kurz of Institut Fresenius, and Ms. Lisa Scola of PRC.

Executive Summary

This report summarizes the findings of an evaluation of the Harbauer soil washing/vacuum-distillation technology developed by Harbauer GmbH & Co. KG, Berlin, Germany. This evaluation was conducted under a bilateral agreement between the U.S. Environmental Protection Agency (EPA) Superfund Innovative Technology Evaluation (SITE) program and the Federal Republic of German Ministry for Research and Technology (BMBF). The Harbauer technology demonstration was conducted on November 23 and 24, 1994 (the stack gas sampling portion of the demonstration was performed on July 13 and 14, 1995) at the Harbauer facility, located in Wölsau near Marktredwitz, Landkreis (County of) Wunsiedel, State of Bavaria, Germany. Elemental mercury-contaminated soil from the Chemische Fabrik Marktredwitz (CFM) site was used for this demonstration.

The Harbauer Technology

The Harbauer technology is a soil washing/vacuum-distillation soil treatment system consisting of two primary processes:

Soil Washing Process

The soil washing process separates the contaminated feed soil into a coarse-grained fraction and a fine-grained fraction. An intermediate-grained fraction (100 microns (æm) - 2 millimeters (mm) is also included as an optional step, however for this demonstration no intermediate fraction was used). The coarse soil fraction (2 to 60 millimeters [mm]) is analyzed and, if the mercury concentration is less than 50 milligrams per kilogram (mg/kg), can be disposed of in the adjacent Chemische Fabrik Marktredwitz (CFM) landfill. If the mercury concentration in the coarse soil is greater than or equal to 50 mg/kg, it is crushed and treated in the vacuum-distillation process. The fine soil fraction (< 2 mm), which can contain mercury at concentrations up to 5,000 mg/kg, is treated in the vacuum-distillation process.

Vacuum-Distillation Process

The vacuum-distillation process heats the soil under a vacuum to volatilize and remove mercury. Contaminated soil is heated to a temperature of approximately 380°C under a reduced pressure (vacuum) of approximately 100 to 200 hectopascals (hPa). The boiling point of elemental mercury is approximately 350 °C at 1014 hPa. Mercury vapors removed from the vacuum-distillation unit enter a water-cooled multistep condenser unit that liquefies the vapors into elemental mercury. The treated fine soil fraction, which contains residual mercury at concentrations ranging from 11 to 31 mg/kg, is mixed with the coarse soil fraction containing less than 50 mg/kg mercury and disposed of in the CFM landfill.

Waste Applicability

The Harbauer technology demonstrated near Marktredwitz, Germany, effectively removed mercury from sandy loam and loam textured soils. The developer claims that in addition to mercury, the technology can also remove some other volatile metals, and volatile and semivolatile organic compounds from soil.

Demonstration Objectives and Approach

This bilateral SITE demonstration of the Harbauer technology was designed with five primary and three secondary objectives. The objectives were chosen to provide potential users of the technology with the information necessary to assess the applicability of the Harbauer technology for treatment of soil from other contaminated sites. In addition, the information acquired from this demonstration may encourage a company in the U.S. to consider the technology for use at Superfund sites. The following primary and secondary objectives were selected to evaluate the technology:

Primary Objectives:

P-1 Document the mercury concentration in the treated soil at a confidence level of 95 percent.

P-2 Determine the mercury removal efficiency achieved by the Harbauer treatment system.

P-3 Document the levels of Toxicity Characteristic Leaching Procedure (TCLP)-leachable mercury in the untreated and the treated soil.

P-4 Document stack gas and process water treatment effluent mercury concentrations.

P-5 Determine the mercury removal efficiency of the vacuum-distillation process.

Secondary Objectives:

S-1 Document the moisture content and the particle size distribution of untreated feed soil and treated soil from the soil washing process and the vacuum-distillation process.

S-2 Document key, nonproprietary system operating parameters.

S-3 Document remediation costs per metric ton of soil.

The demonstration project objectives were achieved by collecting and analyzing soil, process water, and stack gas samples. Soil and process water samples were collected on November 23 and 24, 1994, in accordance with a quality assurance project plan (QAPP) dated November 1994; however, stack gas samples could not be collected at that time. An amendment to the QAPP dated March 20, 1995 was prepared and the stack gas samples were collected on July 13 and 14, 1995 in accordance with the QAPP amendment.

Demonstration Conclusions

Based on the Harbauer bilateral SITE demonstration, the following conclusions may be drawn about the Harbauer soil washing/vacuum-distillation soil treatment technology:

* The 95 percent upper confidence limits of mercury concentrations in treated soil for the three test runs were 22.6 mg/kg, 24.2 mg/kg, and 21.7 mg/kg. Average total mercury concentrations in the sandy loam and loam soils treated were reduced from 875 mg/kg to less than 20 mg/kg.

* Average total mercury removal efficiencies for the soils ranged from 97.6 to 98.0 percent.

* Average TCLP-leachable mercury concentrations in the soils were reduced from 82 micrograms per liter (μg/L) to less than 6 μg/L.

* The average mercury concentration in treated process water discharged to the municipal sewer was 5 μg/L.

* The average mercury concentration in the treated stack gas discharged to the atmosphere was 2.92 micrograms per normal cubic meter (these measurements were not made during the same test runs that demonstration soil samples were collected).

* The mercury removal efficiencies achieved by the vacuum-distillation process for the three test runs were 98.7 percent, 98.4 percent, and 96.6 percent.

* The estimated treatment cost is 480 Deutsche Marks (DM) per metric ton, which is

approximately $320 per metric ton (assuming a 1.5 DM to $1 exchange rate).

In Appendix A, the developer has provided case studies from Germany and the developer's interpretation of the data collected during this bilateral SITE demonstration.

Technology Applicability

The Harbauer technology was evaluated to identify its advantages, disadvantages, and limitations. The evaluation was based on the nine criteria used for decision making in the Superfund feasibility study process. Table ES-1 summarizes the evaluation. The overall effectiveness of the system depends upon the soil type, contaminant type and concentration, the residence time of soil in the system, the temperature of the system, and the contaminant soil-vapor partitioning (mass transfer coefficient). The mass transfer coefficient depends on the contaminant's vapor pressure, Henry's Law Constant, and the soil's physical-chemical properties. This demonstration was limited to an evaluation of the technology's ability to remove mercury from soil.

TABLE ES-1

FEASIBILITY STUDY EVALUATION CRITERIA FOR THE HARBAUER TECHNOLOGY
HARBAUER BILATERAL SITE DEMONSTRATION
MARKTREDWITZ, GERMANY

CRITERION		HARBAUER TECHNOLOGY ASSESSMENT
1	Overall Protection of Human Health and the Environment	Provides both short-term and long-term protection by reducing the contaminant concentrations in soil and reducing the leachability of the contaminants from the soil. Harbauer also claims that the technology treats waste mixtures that include volatile and semivolatile organics, and some metals. Requires measures to protect workers and the public during excavation, handling, and transportation of contaminated soil. Treated soil can be disposed of in a landfill or potentially reused for fill soil onsite. During the demonstration, mercury concentrations in soil were reduced to levels below the 1995 risk-based Preliminary Remediation Goals (PRGs) established by EPA. Some contaminants, such as mercury, can be recycled and reused. Process water effluent is treated via precipitation, activated carbon adsorption, and ion exchange allowing discharge to the municipal sewer system. Air emissions are treated with a wet scrubber and activated carbon adsorption units.
2	Compliance with Federal Applicable or Relevant and Appropriate Requirements (ARARs)	Requires compliance with RCRA treatment, storage, and land disposal regulations (for a hazardous waste). Because the contaminants (mercury) can be recycled, permit requirements may be less stringent. Excavation of contaminated soil and operation of an on-site treatment unit may require compliance with location-specific ARARs. Stack gas emission controls are needed to ensure compliance with air quality standards if volatile compound or particulate emissions occur during excavation, handling, or transportation prior to treatment. Corrective actions must also be performed in accordance with Occupational Safety and Health Administration requirements. Treated waste may be delisted or handled as a nonhazardous waste. Compliance with chemical-, location-, and action-specific ARARs must be determined on a site-specific basis. Compliance with chemical-specific ARARs depends on the treatment efficiency of the Harbauer system and the contaminant concentrations in the feed soil. Treated process water may be discharged to a municipal sewer; however, discharge to a surface water body would require compliance with Clean Water Act regulations.

TABLE ES-1 (Continued)

FEASIBILITY STUDY EVALUATION CRITERIA FOR THE HARBAUER TECHNOLOGY
HARBAUER BILATERAL SITE DEMONSTRATION
MARKTREDWITZ, GERMANY

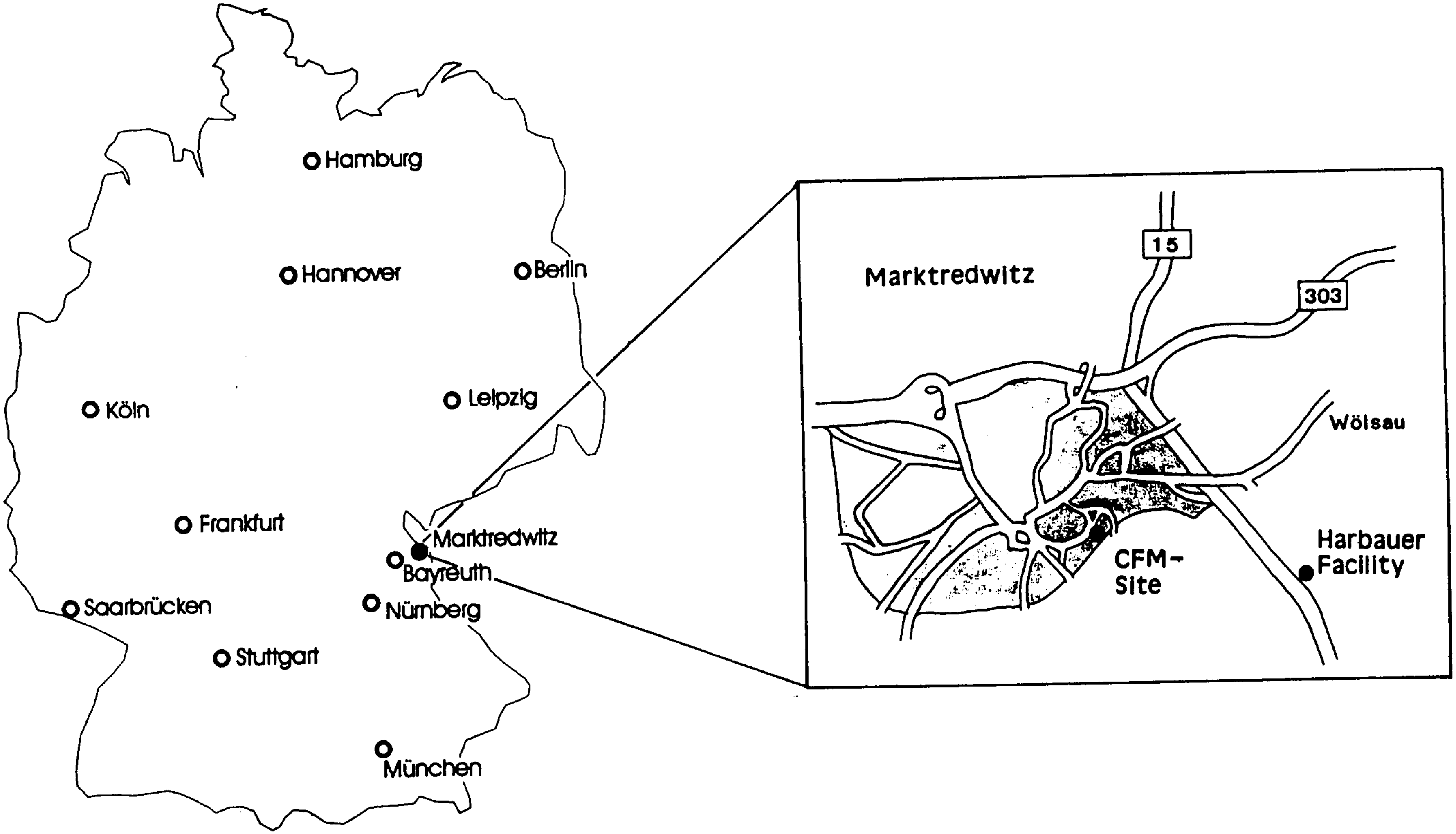
CRITERION		HARBAUER TECHNOLOGY ASSESSMENT
3	Long-Term Effectiveness and Permanence	Contaminants are permanently removed from the soil. The leaching potential of the contaminants from the soil is also reduced. Treatment residuals such as elemental mercury can be recycled and activated carbon can be regenerated on site.
4	Reduction of Toxicity, Mobility, or Volume Through Treatment	Long-term remediation of the contaminated soil is achieved by reduction of contaminant mass and mobility (leachability). Contaminant volume is reduced because contaminants are removed from the soil and concentrated (condensed).
5	Short-Term Effectiveness	Appropriate measures must be taken to prevent fugitive emissions and dust during excavation, handling, and transportation of contaminated soil for treatment. Adverse impacts from the stack gas are mitigated by passing the emissions through activated carbon adsorption units before discharge to the atmosphere.
6	Implementability	Contaminated soils must be characterized prior to transport to the Harbauer facility for treatment. In some cases, such as soils containing a significant clay or organic carbon fraction or soils contaminated with mixtures of compounds, a treatability test is recommended. A land area of 2,500 square meters is required to set up the treatment plant equipment.
7	Cost	Capital costs for the facility are 45,000,000 DM ($30,000,000) and annual operation and maintenance costs are $4,800,000 DM ($3,200,000). Treatment costs are approximately 480 DM/metric ton ($320/metric ton, assuming a 1.5 DM per $ exchange rate)
8	State Acceptance	State acceptance is likely because the Harbauer system does not use incineration and contaminants are condensed and recycled. Because the contaminants are recycled, permit requirements may be less stringent. State regulatory agencies will require permits to operate the treatment system, for air emissions, and to store contaminated soil and treatment residuals.
9	Community Acceptance	The small risks presented to the community from facility air emissions, the fact that the technology does not use incineration, and the permanent removal of the contaminants from soil may make public acceptance of the technology more likely. However, community acceptance of any type of permanent hazardous waste treatment facility in an area is difficult to predict.

Chapter 1.0

Introduction

This report documents the findings of an evaluation of the Harbauer soil washing/vacuum-distillation technology developed by Harbauer GmbH & Co. KG, Berlin, Germany. This evaluation was conducted under a bilateral agreement between the U.S. Environmental Protection Agency (EPA) Superfund Innovative Technology Evaluation (SITE) program and the Federal Republic of German Ministry for Research and Technology (BMBF). The Harbauer technology demonstration was conducted on November 23 and 24, 1994 (stack gas sampling was performed on July 13 and 14, 1995) at the Harbauer facility located in Wölsau near Marktredwitz, Landkreis (County of) Wunsiedel, State of Bavaria, Germany (see Figure 1). The demonstration evaluated the technology's effectiveness to remediate mercury contaminated soil from the Chemische Fabrik Marktredwitz (CFM) site. Soil, process water, and stack gas sampling was conducted by Arbeitgemeinschaft (ArGe) focon-Probiotec with assistance from PRC Environmental Management, Inc. (PRC). System operating parameters were monitored and recorded by Harbauer. All sample analyses were performed by Institut Fresenius. All demonstration activities were conducted in accordance with the November 1994 quality assurance project plan (QAPP) (PRC 1994) and the March 20, 1995 QAPP amendment (PRC 1995). ArGe focon-Probiotec, Harbauer, and Institut Fresenius contributed extensively to the development of this document.

This report provides information from the bilateral SITE demonstration of the Harbauer technology that is useful for remedial managers, environmental consultants, and other potential technology users in implementing the technology at contaminated sites. Section 1.0 presents an overview of the SITE program and the bilateral agreement, describes the Harbauer technology, and lists key contacts. Section 2.0 presents information relevant to the technology's effectiveness, including CFM site background, demonstration procedures, and the results and conclusions of the demonstration. Section 3.0 presents information on the costs associated with applying the technology. Section 4.0 presents information relevant to the technology's application, including an assessment of the technology related to the nine feasibility study evaluation criteria used for decision making in the Superfund process, applicable wastes/contaminants, key features of the technology, materials handling requirements, site support



HARBAUER DEMONSTRATION
WÖLSAU, GERMANY

FIGURE 1

HARBAUER FACILITY AND CFM SITE

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requirements, and limitations of the technology. Section 5.0 summarizes the technology status, and Section 6.0 lists references used in preparing this report. Appendices A and B present case studies provided by the developer and the results of field and laboratory quality assurance reviews.

1.1 Superfund Innovative Technology Evaluation Program

This section provides background information about the EPA SITE program. Additional information about the SITE program, the Harbauer technology, and the demonstration can be obtained by contacting the key individuals listed in Section 1.4.

EPA established the SITE program to accelerate development, demonstration, and use of innovative technologies to remediate hazardous waste sites. The demonstration portion of the SITE program focuses on technologies in the pilot-scale or full-scale stage of development. The demonstrations are intended to collect performance data of known quality. Therefore, sampling and analysis procedures are critical. Approved quality assurance/quality control (QA/QC) procedures must be stringently applied throughout the demonstration.

Past hazardous waste disposal practices and their human health and environmental impacts prompted the U.S. Congress to enact the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) of 1980 (PL96-510). CERCLA established a Hazardous Substance Response Trust Fund (Superfund) to pay for handling emergencies at uncontrolled hazardous waste sites and for cleaning up the sites. Under CERCLA, EPA has investigated these hazardous waste sites and established national priorities for site remediation. The ultimate objective of the investigations is to develop plans for permanent, long-term site cleanups, although EPA initiates short-term removal actions when necessary. EPA's list of the nation's top-priority hazardous waste sites that are eligible to receive federal cleanup assistance under the Superfund program is known as the National Priorities List (NPL).

Congress expressed concern over the use of land-based disposal and containment technologies to mitigate problems caused by releases of hazardous substances at hazardous waste sites. As a result of this concern, the 1986 reauthorization of CERCLA, called the Superfund Amendments and Reauthorization Act (SARA), mandates that EPA "select a remedial action that is protective of human health and the environment, that is cost effective, and that utilizes permanent solutions and alternative treatment technologies or resource recovery technologies to the maximum extent practicable." In response to this requirement, EPA established the SITE program to accelerate development, demonstration, and use of innovative technologies for site cleanups. The SITE program has four goals:

- Identify and, where possible, remove impediments to development and commercial use of innovative technologies
- Conduct demonstrations of the more promising innovative technologies to establish reliable performance and cost information for site characterization and cleanup decision-making
- Develop procedures and policies that encourage selection of effective innovative treatment technologies at uncontrolled hazardous waste sites
- Structure a development program that nurtures emerging technologies

Each year EPA selects the best available innovative technologies for demonstration. The screening and selection process for these technologies is based on four factors: (1) the technology's capability to treat Superfund wastes, (2) expectations regarding the technology's performance and cost, (3) the technology's readiness for full-scale demonstrations and applicability to sites or problems needing remedy, and (4) the developer's capability of and approach to testing. SITE program demonstrations are administered by EPA's Office of Research and Development (ORD) through the National Risk Management Research Laboratory (NRMRL) in Cincinnati, Ohio.

SITE demonstrations are usually conducted at uncontrolled hazardous waste sites such as EPA removal and remedial action sites, sites under the regulatory jurisdiction of other federal agencies, state sites, EPA testing and evaluation facilities, sites undergoing private cleanup, the technology developer's site, or privately owned facilities. In the case of the Harbauer technology demonstration, the site was selected cooperatively by EPA and BMBF. The EPA - BMBF bilateral agreement is discussed in Section 1.2.

SITE and bilateral SITE demonstrations should provide detailed data on the performance, cost-effectiveness, and reliability of innovative technologies. These data will allow potential users of a technology to have sufficient information to make sound judgments about the applicability of the technology to a specific site (waste) and to allow comparisons of the technology to other treatment technology alternatives.

1.2 United States And German Bilateral Agreement On Remediation Of Hazardous Waste Sites

In April 1990, EPA and the BMBF entered into a bilateral agreement to gain a better understanding of each country's efforts in developing and demonstrating remedial technologies. The bilateral agreement has the following goals:

- Facilitate an understanding of each country's approach to the remediation of contaminated sites
- Demonstrate innovative remedial technologies as if the demonstrations had taken place in their native country
- Facilitate international technology exchange

Technologies at 12 sites, six in the U.S. and six in Germany, will be evaluated under the bilateral agreement. Individual, or in some cases, multiple remediation technologies will be demonstrated at each site. Technology evaluations occurring in the U.S. correspond to SITE demonstrations; those occurring in Germany correspond to full-scale site remediation activities (and are referred to as bilateral SITE demonstrations). In the case of the U.S. evaluations, demonstration plans are prepared following routine SITE procedures. Additional monitoring and evaluation measurements required for evaluation of the technology under German regulations will be specified by the German partners. For the demonstrations occurring in Germany, the German partners will provide all required information to allow the U.S. partners to develop an EPA Category II QAPP. An EPA Category II QAPP titled "Quality Assurance Project Plan for the Harbauer Soil Washing/Vacuum-Distillation System Demonstration at the Chemische Fabrik Marktredwitz Site, Marktredwitz, Germany" dated November 1994 was prepared for this demonstration (PRC 1994).

ArGe focon-Probiotec (a partnership of two German environmental consulting firms) was commissioned by BMBF to compile summary reports for the German technologies and sites, to evaluate the U.S.

demonstration plans, and to facilitate the bilateral agreement on behalf of the BMBF. The ArGe focon-Probiotec technical consulting partnership is not directly involved in the German remedial actions and the partnership does not influence actual site remediation activities. The bilateral project organization is presented in Figure 2.

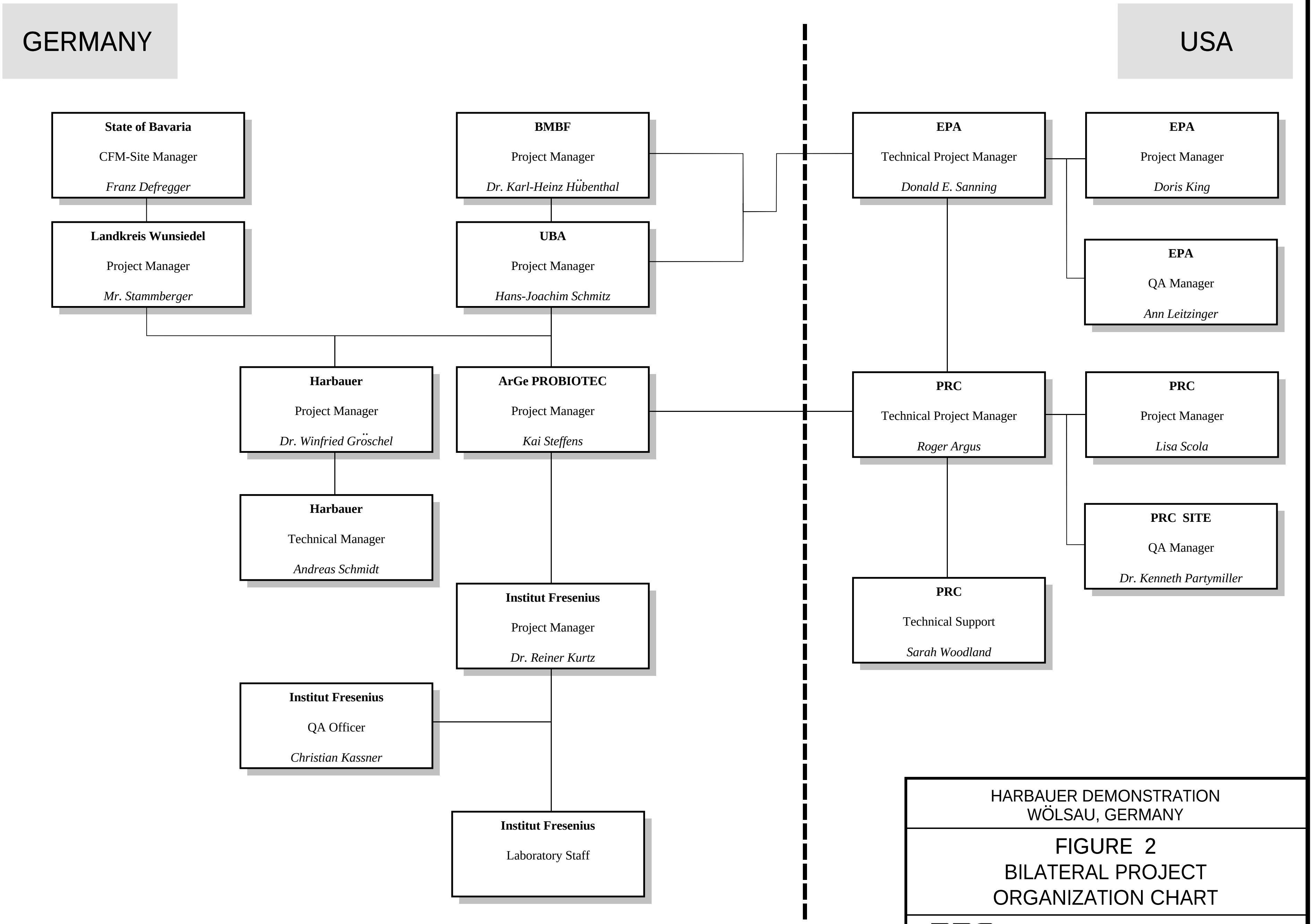
1.3 Harbauer Technology Description

This section describes the process equipment and system operations of the Harbauer soil treatment facility. The information provided in this section is limited due to Harbauer's proprietary claims on much of the process design and system operating data. For example, detailed descriptions of system components are not available as Harbauer considers this information proprietary. However, an effort has been made to present enough information to understand the technology's concept of operation and the results of the demonstration.

1.3.1 Process Equipment

The Harbauer treatment system consists of two main processes, soil washing and vacuum distillation. The soil washing process separates the contaminated feed soil into a coarse-grained fraction and a fine-grained fraction. The coarse soil fraction (2 to 60 millimeters [mm]) is analyzed and, if the mercury concentration is less than 50 milligrams per kilogram (mg/kg), disposed of in the adjacent CFM landfill. If the mercury concentration in the coarse soil is greater than or equal to 50 mg/kg, it is crushed and treated in the vacuum-distillation unit. The fine soil fraction (< 2 mm),which contains mercury at concentrations up to 5,000 mg/kg, is treated in the vacuum-distillation unit.

The vacuum-distillation process heats contaminated soil under a vacuum to volatilize and remove mercury from the soil. Contaminated soil is heated to a temperature of approximately 380°C under a reduced pressure (vacuum) of approximately 100 to 200 hectopascals (hPa). The boiling point of



HARBAUER DEMONSTRATION
WÖLSAU, GERMANY

FIGURE 2
BILATERAL PROJECT
ORGANIZATION CHART

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elemental mercury is approximately 350°C at 1014 hPa (1 atmosphere); therefore, the reduced pressure. causes mercury to be volatilized more rapidly and removed from the soil at the system temperature Mercury volatilizes from the soil and the mercury vapors are removed from the vacuum-distillation unit. The mercury vapors enter a water-cooled multistep condenser unit that liquefies the vapors into elemental mercury. The treated fine soil fraction, which contains residual mercury at concentrations ranging from 11 to 31 mg/kg, is mixed with the coarse soil fraction (containing mercury at less than 50 mg/kg) and disposed of in the CFM landfill.

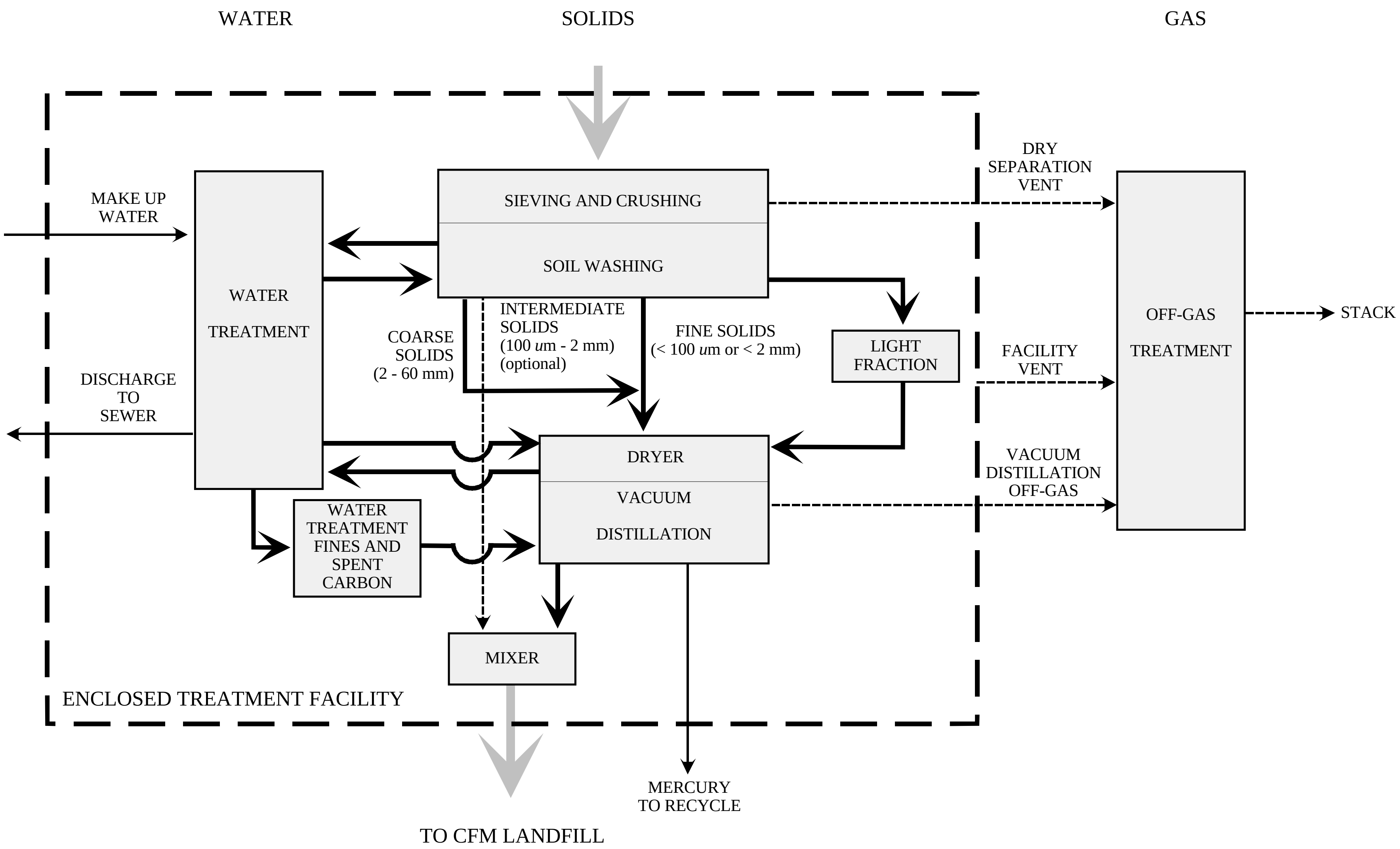
The Harbauer soil washing/vacuum-distillation unit is a full-scale, commercial, transportable plant configured as shown in Figure 3. In addition to soil washing and vacuum-distillation processes, the unit incorporates air and process water treatment processes. These processes are described in the following sections.

1.3.1.1 Soil Washing Process

The Harbauer soil washing process is a continuous, water-based (no detergents or surfactants are used), volumetric reduction process that is based on two mechanisms:

1. Removal of mercury from coarse-grained soils (soils with a particle diameter greater than 2 mm).
2. Separation of the coarse-grained soils from fine-grained soils (soils with a particle diameter less than 2 mm).

The soil washing process results in a coarse-grained fraction that contains residual mercury at concentrations less then 50 mg/kg, and a fine-grained fraction that contains mercury at concentrations greater than 50 mg/kg. The optional intermediate-grained fraction was not segregated during this demonstration.



HARBAUER DEMONSTRATION
WÖLSAU, GERMANY

FIGURE 3

SYSTEM FLOW DIAGRAM

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Contaminants tend to chemically and physically adhere to the fine-grained soil fraction (silts and clays) due to its greater surface area and higher natural organic matter content. The silts and clays, in turn, tend to adhere to coarser sand and gravel particles. The soil washing process removes the more highly contaminated silt and clay from the coarser soil fractions and scrubs the coarser fractions, resulting in clean or only slightly contaminated sand and gravel.

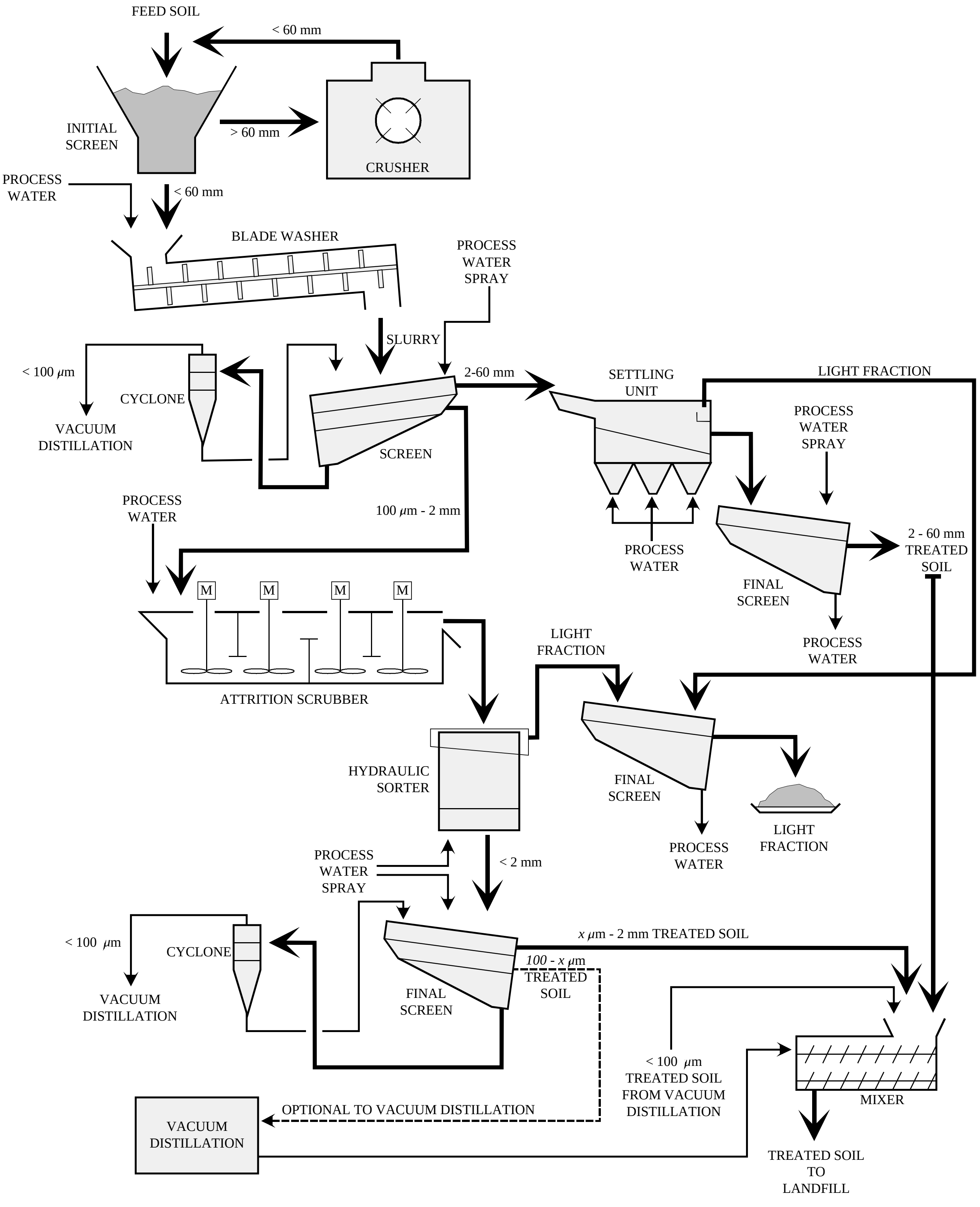
The soil washing process involves the sieving and crushing of soil to a particle diameter less than 60 mm; mixing the soil with recycled process water in a double-axis blade washer; and separating the soil into a relatively clean coarse fraction (particle diameter between 2 and 60 mm), a highly contaminated fine fraction (particle diameter smaller than 2 mm), and a lightweight coarse fraction. A system flow diagram of the soil washing plant is shown in Figure 4.

The soil washing process involves the following steps:

1. Initial Screening
2. Crushing (if necessary)
3. Blade washing
4. Screening
5. Attrition scrubbing
6. Hydraulic classification via settling or hydraulic sorting
7. Final screening

Initially, the soil is mechanically sieved in a vibratory screen to less than 60 mm in diameter. Materials greater than 60 mm in diameter are crushed by a mechanical crusher and refed to the screening device.

After the initial screening and crushing, the soil is transported via an encapsulated conveyor belt to the highest point of the treatment facility and is transferred downward by gravity through the various washing and separation steps. First, the soil is fed continuously to a double-axis blade washer unit where it is mixed with water at a rate of one cubic meter of water per metric ton of soil. Feed water to the blade



HARBAUER DEMONSTRATION
WÖLSAU, GERMANY

FIGURE 4

SOIL WASHING PROCESS

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washer is recycled process water. A constant feed rate of contaminated soil is maintained by a belt-scale feed-back control system.

The soil/water slurry exiting the blade washer unit is transferred to a screening unit consisting of a double screen and a hydrocyclone. The soil particles with a diameter less than 100 microns (æm) pass through the screens and cyclone and are separated as part of a process water slurry. This slurry is pumped to the vacuum-distillation unit that is described in the next section. Soil particles with a diameter of 100 æm to 2 mm are transferred to an attrition scrubber, where additional recycled process water is added and the slurry is intensely mixed (this step was not used during the demonstration). During mixing, surface contamination is scrubbed from the coarser grained soil by abrasion of particles against each other.

After passing the attrition scrubber, the soil-water mixture enters a hydraulic sorter, where the lightweight particles are removed. After being separated from the water stream by screening and washed with a process water spray, the lightweight fraction is collected in bins. The remaining soil-water mixture that passed the hydraulic sorter is fed to a cyclone, where the grains with a size less than 100 æm, newly generated from breakdown of larger grains or clumps during processing in the attrition scrubber, are separated and pumped to the vacuum-distillation process.

Soil particles larger than 100 æm are separated from the process water by screening, washed with a process water spray, and brought out of the process as treated soil via a vertical discharge pipe directly into a mixer. The mixer blends the washed fraction with the treated fines from the vacuum-distillation process.

The coarse-grained solids from the initial screening step are conveyed to a settling unit in which the lightweight fraction is removed. This coarse lightweight fraction is added to the 100 æm to 2 mm lightweight fraction for final screening and collection in bins.

The heavier coarse solids are drawn from the settling unit into a final screening/spray washing step before they are blended with the (optional) intermediate 100 æm to 2 mm washed soil and the treated fines.

The separated process water is treated and then recycled except for a slipstream that is discharged to the municipal sewer after a final polishing treatment step. Section 1.3.1.4 provides a description of the water treatment process.

Soil processing time of the entire soil washing process is approximately 25 minutes. The maximum throughput of the soil washing unit is approximately 20 metric tons per hour.

1.3.1.2 Vacuum-Distillation Process

The fine grained (< 2 mm diameter), highly contaminated fraction of soil that is generated by the soil washing process is transferred as a slurry to the vacuum-distillation unit. The vacuum-distillation process involves heating the soil to a temperature high enough to volatilize mercury. At 1 atmosphere (1014 hPa), the boiling point of elemental mercury is approximately 350øC (662øF). The Harbauer vacuum-distillation process heats the soil to approximately 380øC (716øF) under reduced pressure (vacuum) conditions. The reduced pressure (100 - 200 hPa) lowers the boiling point of mercury, resulting in more efficient removal of mercury from the soil and lower energy consumption.

The vacuum-distillation process consists of four primary units:

- Mechanical dewatering
- Drying
- Vacuum distillation
- Cooling

The dryer, the vacuum-distillation unit, and the cooler each have an outside diameter of 3 meters (m), an overall length of 10 m, and rotate at 10 revolutions per minute.

A process flow diagram for the vacuum-distillation process is provided in Figure 5. Prior to mechanical dewatering, Ferrogranul (an iron chloride sulfate) and a polymeric flocculating agent are added to the soil slurry, causing heavy metals to flocculate. The slurry is then allowed to settle in six clarifiers operated in parallel. The flocculating agent causes the fine particles to aggregate, settle, and separate from the process water. The concentrated solids are then mechanically dewatered in a filter press to minimize the energy consumption of the subsequent drying process. The fine particle filter cake from the filter press is transported via a conveyor to the top-most point of the vacuum-distillation process and passes downward, by gravity, through the various process units. The process water from the concentrating and dewatering is sent to water treatment for purification prior to reuse.

The filter press solids are fed to a rotating drum dryer that is heated indirectly by steam to approximately 100°C. The steam is produced in a noncontact heat exchanger using excess thermal energy from the off-gas of the vacuum-distillation unit. The dryer is also equipped with additional stand-by burners that may be used during process start-up or nonroutine operation. The residence time of the soil in the dryer is approximately 30 minutes. Soil exiting the dryer has a residual moisture content of less than 1 percent by weight. The off-gas from this drying step is treated in the off-gas treatment process. Solids exiting the dryer are fed through a double chamber vacuum lock via a screw-conveyor to the vacuum-distillation unit, a cylindrical rotating processor heated indirectly by propane burners. Under reduced pressure conditions of 100 - 200 hPa and an average temperature of 380°C, the mercury and other compounds are volatilized from the soil.

Predemonstration trial runs using contaminated soil from the CFM site showed the best removal efficiencies at temperatures of 380°C with residence times of approximately 30 minutes in the vacuum-distillation unit. The maximum throughput of the vacuum-distillation unit is approximately four metric tons per hour.

After passing the vacuum-distillation unit, the soil is transported via a screw conveyor to a cooler. The cooler is a water-cooled rotating drum. The cooled soil (temperature less than 50°C) passes a dual-chamber vacuum lock and enters the mixer via a conveyor belt and a vertical discharge pipe. The treated

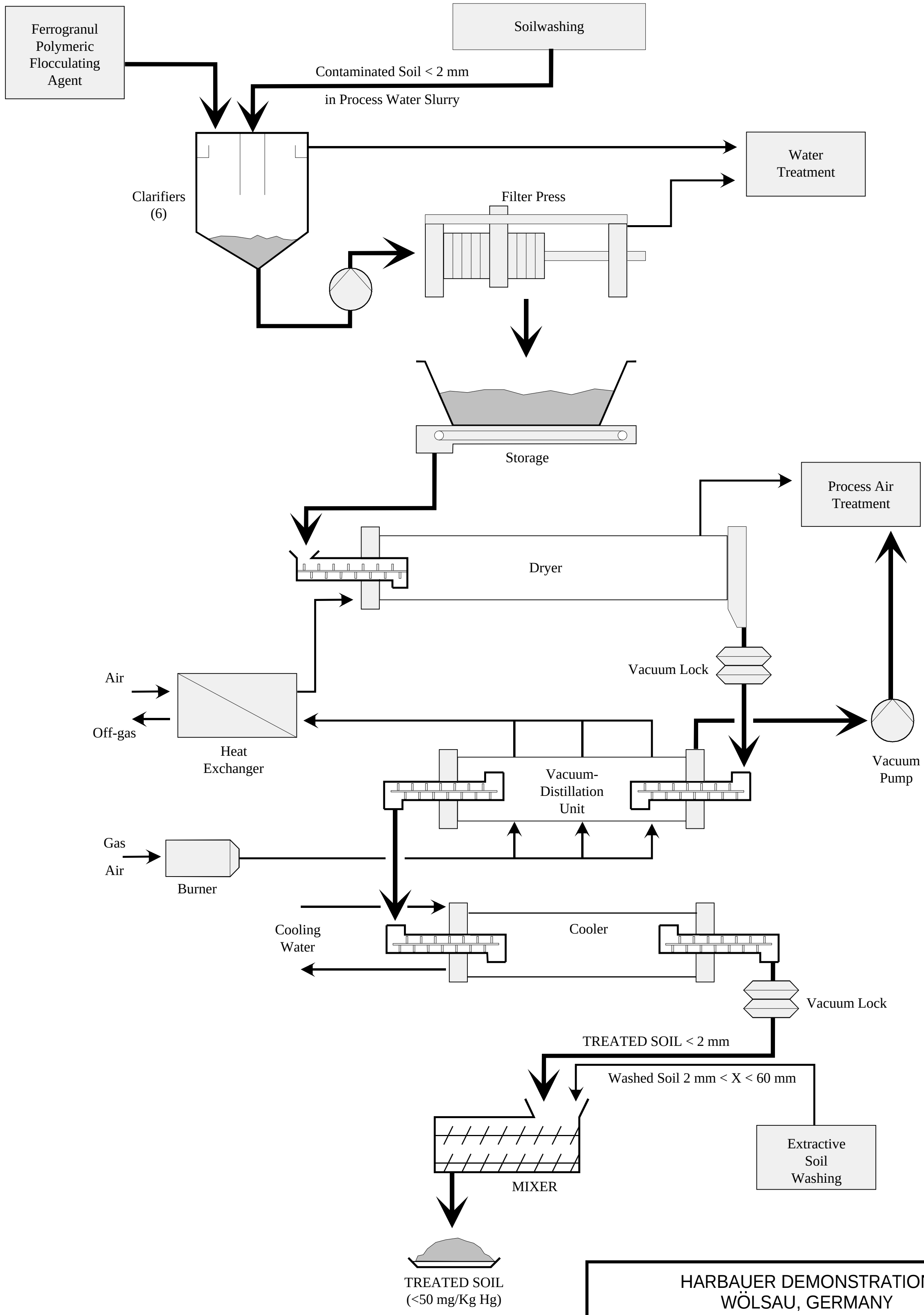

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FIGURE 5

VACUUM-DISTILLATION PROCESS

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soil (containing between 5 and 30 mg/kg of mercury) is mixed with washed coarse-grained soil and is disposed of in the CFM landfill adjacent to the treatment facility.

1.3.1.3 Air Treatment Process

The entire Harbauer system is encapsulated and maintained under a slight vacuum. The off-gas from the facility is captured and treated before release to the atmosphere. A schematic flow diagram of the air treatment process is shown in Figure 6. The air treatment process has a capacity of approximately 40,000 cubic meters per hour. The dry (mechanical) separation vent, which contains emissions from the crusher, is passed through a dust filter before joining the plant vent gases. Stack gas treatment consists of two activated carbon beds (iodinized and non-iodinized) arranged in series. Water vapor and mercury vapor are removed from the vacuum-distillation unit by vacuum and enter the off-gas treatment process. Mercury volatilizes primarily in the vacuum-distillation unit and enters a water-cooled, multistep condenser unit. Harbauer estimates that approximately 90 percent of the mercury is condensed and recovered as liquid elemental mercury. The relatively small amounts of off-gas exiting the condenser unit (approximately 500 cubic meters per hour) are treated in a conventional wet gas scrubber and are then blended with the larger amount of off-gas from the facility ventilation system. The individual performance of these air treatment process units was not evaluated during this demonstration. Therefore, a detailed description of these units is not provided here.

1.3.1.4 Process Water Treatment Process

Process water from the entire facility is treated and reused. Before being reused, process water is sent to an on-site treatment process, which uses sedimentation with a polymeric flocculating agent, precipitation with an alkaline solution of anhydrite, flocculation of heavy metals using Ferrogranul, pH adjustment using hydrochloric acid addition, and activated carbon polishing. The entire process contains approximately 60 cubic meters of process water. A schematic flow diagram of the water treatment process is shown in Figure 7. A slipstream of treated process water is discharged to the municipal sewer, and municipal make-up water is added to the recycled stream at rates of between 10 to 15 cubic meters per hour. Prior to discharge, this wastewater slip-stream is treated by ion exchange for mercury removal. Spent activated carbon is regenerated in the vacuum-distillation unit.

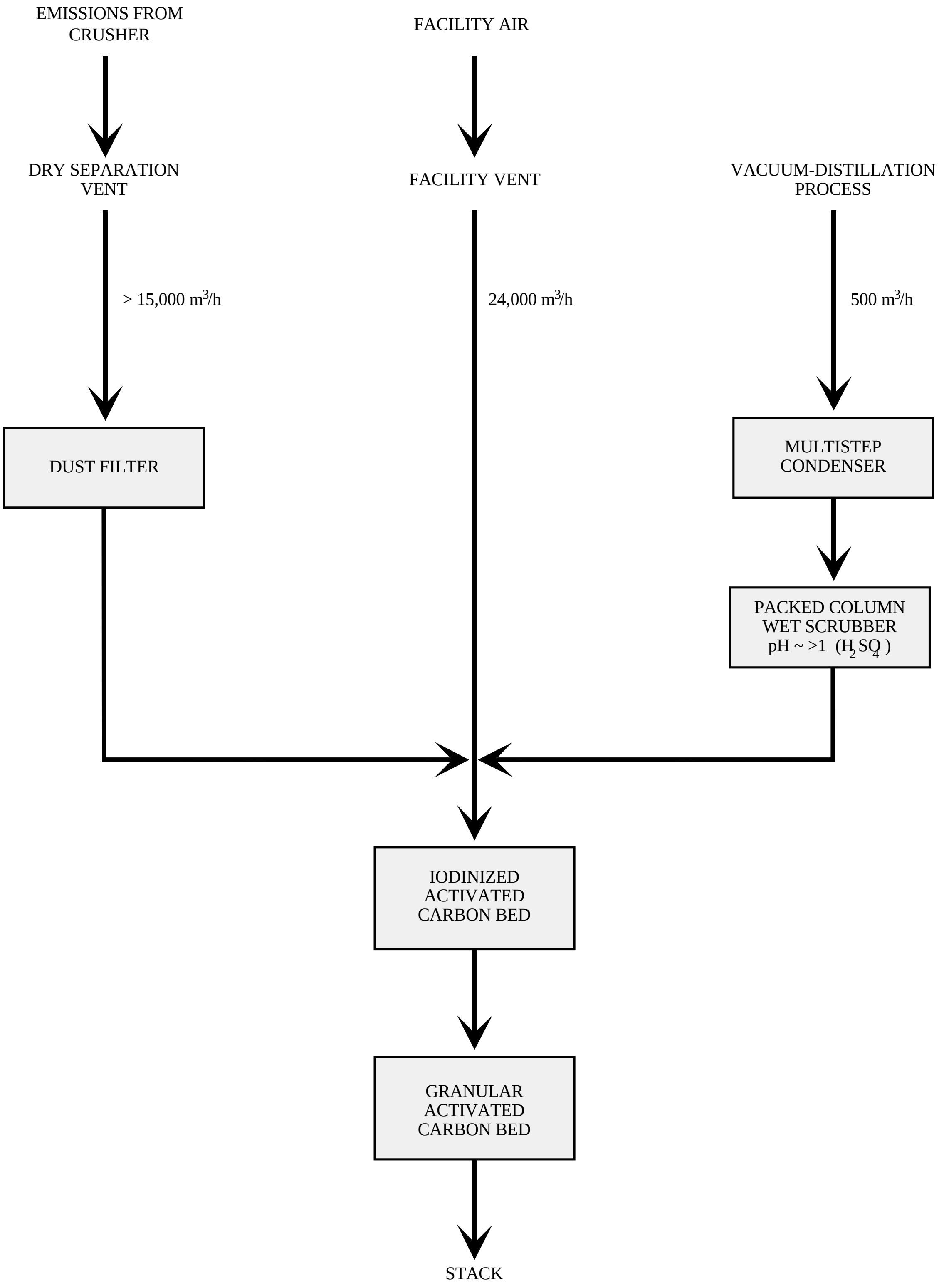

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FIGURE 6

AIR TREATMENT PROCESS

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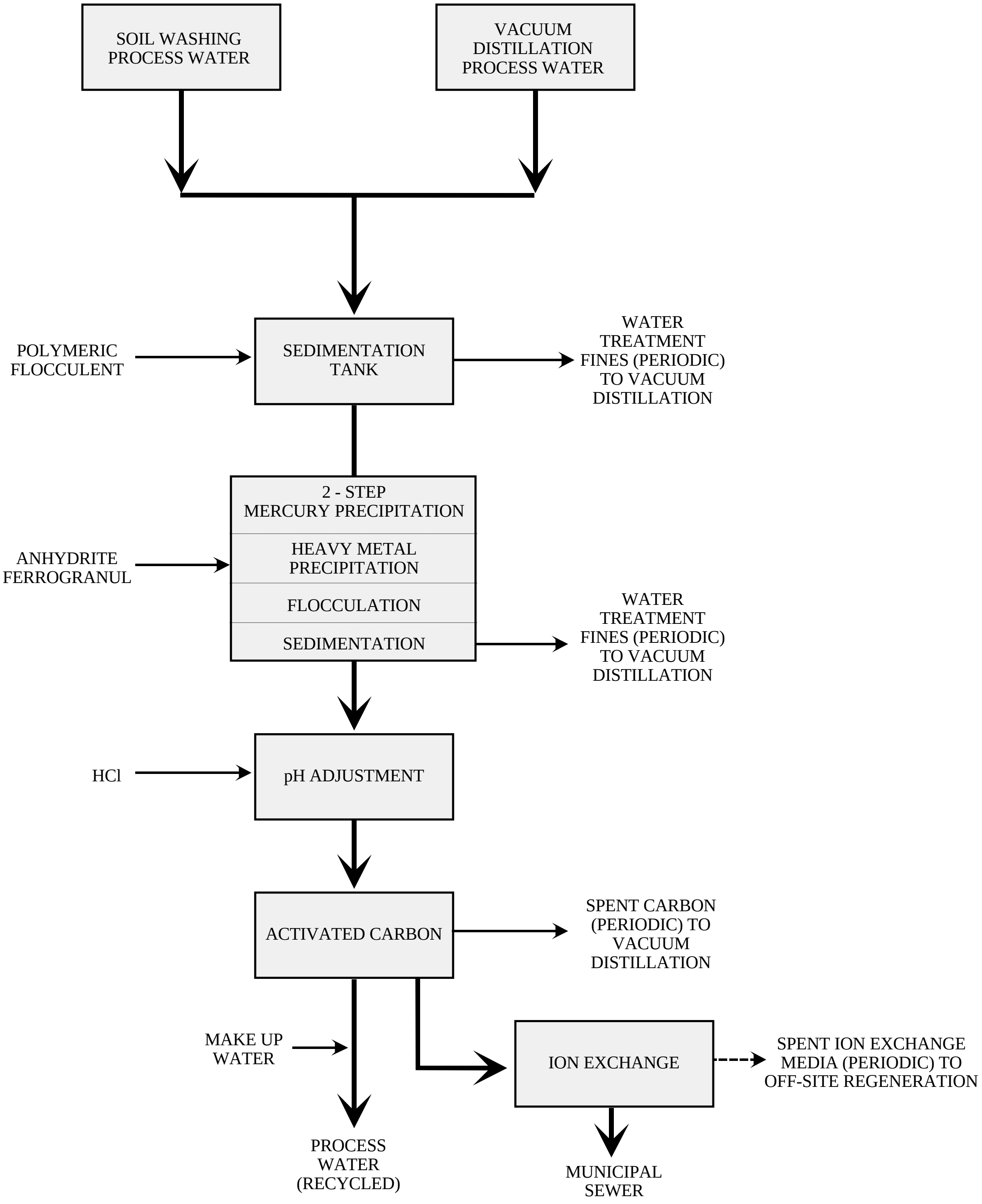

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FIGURE 7
PROCESS WATER
TREATMENT PROCESS

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1.3.2 System Operation

Approximately 4 to 6 weeks before the demonstration was scheduled to begin, the source area of the contaminated soil to be treated from the CFM site changed. The soil to be treated included large amounts of coarse construction debris (concrete and brick, for example) that was more porous than the natural gravel found at the site. Because of the relatively high concentrations of mercury present in the porous construction debris, all of the construction debris and soil was crushed and subsequently treated in the vacuum-distillation unit for the demonstration.

Excavated soil from the CFM site was transported to the Harbauer facility in Wölsau. For regulatory and health and safety reasons, the soil was transported in air-tight containers of 15 metric ton capacity. The container trucks were equipped with a self-contained breathing apparatus for the driver, which allowed the truck to enter the Harbauer plant via a truck lock. The air from the truck lock and soil feed area, where the containers are unloaded, was captured and treated in the plant off-gas treatment process.

The soil processing rate of the system depends on the mercury concentration in the feed soil. Maximum soil processing rates of the soil washing and the vacuum-distillation processes are approximately 20 and 4 metric tons per hour, respectively. Therefore, the soil processing rate of the system is limited by the vacuum-distillation process. Mercury concentrations in the soil at the CFM site ranged from 500 to 5,000 mg/kg and the soils used for this demonstration had average mercury concentrations ranging from 780 to 1,080 mg/kg. Soil processing rates during the demonstration ranged from 2.4 to 3.7 metric tons per hour. Approximately 63 metric tons of mercury contaminated soil were treated during the three demonstrations test runs (22 total hours).

The soil washing process can be configured to obtain a separation diameter (maximum grain size that is separated to be fed to the vacuum-distillation process) of between 100 µm and 2 mm.

The Harbauer system is fully automated to allow for 24-hour operation. For the CFM site remedial project, the soil washing process operated 8 to 14 hours per day. The vacuum-distillation process operated 24 hours a day. The amount of feed soil exiting the soil washing process that could not be processed immediately by the vacuum-distillation process was stored in a buffer container. The buffer container stored enough soil for 2 days operation of the vacuum-distillation unit and allowed for 24-hour operation of the vacuum-distillation unit.

Highly contaminated residuals (solids) from the water treatment process and separated lightweight materials from the soil washing process are fed periodically (once per week or once per month) to the vacuum-distillation process when the collection bins (capacity approximately 1 cubic meter) are full. The lightweight fraction consists of wood chips, coal particles, plastics, and other debris. The treated residuals are part of the solid stream exiting the vacuum-distillation unit. During the technology demonstration, these internal process residuals were not treated by the vacuum-distillation process because they represented a small fraction of the total throughput of the process and would introduce an additional variable that could unnecessarily complicate achieving the demonstration objectives.

1.4 Key Contacts

Additional information on the Harbauer technology and the EPA-BMBF bilateral technology evaluation program can be obtained from the following sources:

Harbauer Soil Washing/Vacuum-Distillation Technology

Dr. Winfried Gröschel and Mr. Andreas Schmidt
Harbauer GmbH & Co KG
Flughafenstrasse 21
D-12053 Berlin
(030) 613730 0

EPA-BMBF Bilateral Technology Evaluation Program

Doris King
Project Manager
U.S. Environmental Protection Agency
Office of Research and Development
26 West Martin Luther King Drive
Cincinnati, OH 45268
513-569-7491

Donald Sanning
Technical Project Manager
U.S. Environmental Protection Agency
Office of Research and Development
26 West Martin Luther King Drive
Cincinnati, OH 45268
513-569-7875

Information on the SITE program is available through the following on-line information clearinghouses:

- The Alternative Treatment Technology Information Center (ATTIC) System (operator: 703-908-2137) is a comprehensive, automated information retrieval system that integrates data on hazardous waste treatment technologies into a centralized, searchable source. This data base provides summarized information on innovative treatment technologies.

- The Vendor Information System for Innovative Treatment Technologies (VISITT) (Hotline: 800-245-4505) data base contains current information on nearly 300 technologies submitted by nearly 200 developers, manufacturers, and suppliers of innovative treatment technology equipment and services.

- The OSWER CLU-IN electronic bulletin board contains information on the status of SITE technology demonstrations. The system operator can be reached at 301-589-8268.

Technical reports may be obtained by contacting the Center for Environmental Research Information (CERI), 26 West Martin Luther King Drive in Cincinnati, OH 45268 at 513-569-7562.

Chapter 2.0

Harbauer Technology Effectiveness

This section documents the background, field and analytical procedures, results, and conclusions used to assess the ability of the Harbauer technology to remove mercury from contaminated soil. This assessment is based on data collected during the Harbauer bilateral SITE demonstration conducted at the Harbauer facility at Wölsau, Germany and on case studies supplied by the technology developer. Because the results of the bilateral SITE demonstration are of known quality, conclusions are primarily drawn from the demonstration results.

2.1 Background

The bilateral SITE demonstration of the Harbauer technology was conducted at the Harbauer facility located in Wölsau near Marktredwitz, Landkreis (County of) Wunsiedel, State of Bavaria, Germany (Figure 1). Contaminated soil for the demonstration was excavated from the CFM site located in Marktredwitz, Bavaria, Germany (Figure 1). The CFM site background is described in the following section. An overview of the demonstration objectives and approach is presented in Section 2.1.2

2.1.1 Chemische Fabrik Marktredwitz (CFM) Site Background

The CFM site is a 0.5 square kilometer area formerly used as a chemical production facility. It is located in the city-center of Marktredwitz, Bavaria, Germany. Founded in the year 1788, CFM was one of the oldest chemical manufacturing facilities in the world. After nearly 200 years of operation, the facility was closed in 1985 because of severe soil and groundwater contamination. The location of the site is shown in Figure 1.

Mercury was processed at the CFM site for the production of pesticides, herbicides, and other mercury-containing products. Additionally, spent solvents, chemical wastes, and treatment residuals were stored and accidentally spilled on the site. The primary contaminant of concern at the site, however, is mercury. Initial site investigations conducted in 1986 indicated that high concentrations of mercury were present in the concrete and brick portions of the buildings and in the soil. In 1988, the State of Bavaria decided to fund remedial action on the site. The Landkreis Wunsiedel, a co-funder of the project, was commissioned to manage the remedial action project.

2.1.1.1 Site Contamination

A comprehensive site characterization effort has been completed that indicates extensive contamination of on-site building debris, subsurface soil, and groundwater. To provide background information, this section briefly discusses each contaminated matrix.

Mercury, as well as arsenic, antimony, and lead, have been found in samples of the concrete and brick structures at the site. Although various metals were detected in most samples, mercury was the main contaminant present. In the surface layers of the brick walls, between 400 mg/kg and 3,300 mg/kg mercury was detected. Mercury contamination in the range of several thousand milligrams per kilogram was also present in underground duct tunnels and processing pits.

Based on an evaluation of the potential hazards of the individual chemical substances found on the site, mercury has been identified as the primary contaminant of concern. Extensive subsurface investigations have been conducted; however, limited data were available at the time of the demonstration. Table 2-1 presents soil characterization data from the CFM site. For this demonstration, mercury contaminant levels in the soil were verified before initiating the planned test runs. Groundwater at the CFM site is also contaminated with mercury and other metals; however, characterization data are not presented here, since this demonstration will focus only on treatment of contaminated soil from the site.

2.1.1.2 Site Remediation Plan

In 1988, a plan for the comprehensive remediation of the CFM site was initiated with the objective to allow development of the site as a housing and shopping area concurrent with remediation. The

Table 2-1. Soil Characterization Data From The CFM Site

PARAMETER	VALUE
Mercury	500 - 5,000 mg/kg[a]
Antimony	50 - 500 mg/kg
Total Phenols	~ 10 mg/kg
Total Recoverable Petroleum Hydrocarbons	~ 30 mg/kg
Total Benzene, Toluene, Ethylbenzene, and Xylenes	~ 20 mg/kg
Total Chlorinated Hydrocarbons	~ 25 mg/kg
Moisture Content	15-20 percent
Percentage of soil with particle size less than 2 mm	~70 percent

a Milligrams per kilogram

Harbauer technology was selected to treat the soil and contaminated debris to a level that allows landfilling of the treated solids. The remediation plan incorporates the following elements:

- Demolition of technical facilities and ducts in the buildings. Removal of the highly contaminated surface layer from the brick walls and disposal of this highly contaminated material (and CFM production residuals) in a hazardous waste landfill.

- Protection of the nearby creek, "Kösseine." To prevent groundwater flow to the Kösseine, a subsurface groundwater barrier was installed along the creek. This 200 m-long vertical barrier reaches depths of 7 to 20 m and is connected to the underlying gneiss bedrock. Contaminated groundwater is recovered along the length of the barrier and is treated in an on-site facility.

- Demolition of buildings. In some cases, this was carried out in steel enclosures that prevented high mercury emissions during demolition. The demolition was completed at the end of 1992.

- Soil excavation and backfilling. The soil on the site must be excavated to an average depth of 4 m below the original ground surface. The excavation pit will be backfilled either with clean site soil or with clean imported fill soil, according to the criteria shown in Table 2-2.

Table 2-2. CFM Remedial Project Treatment And Landfill Criteria

PARAMETER	CRITERIA FOR BACKFILLING OR LOCAL DEBRIS LANDFILL (Reinigungsanforderung A)	CRITERIA FOR CFM LANDFILL (Reinigungsanforderung B)[a]
Soil Parameters		
Mercury	≤ 10 mg/kg[b]	≤ 50 mg/kg
Elutriate[c] Parameters		
pH	Not Specified	6.5 - 9.5
Mercury	0.004 mg/L[d]	0.1 mg/L
Lead	0.16 mg/L	2.0 mg/L
Arsenic	0.04 mg/L	Not Specified
Antimony	0.04 mg/L	Not Specified
Arsenic plus Antimony	Not Specified	1.0 mg/L
Cadmium	0.02 mg/L	0.5 mg/L
Nickel	0.2 mg/L	2.0 mg/L
Chromium (VI)	0.2 mg/L	0.5 mg/L
Copper	Not Specified	1.0 mg/L
Dissolved Organic Carbon	Not Specified	< 200 mg/L

[a] Solids exceeding these criteria are to be treated with the Harbauer system
[b] Milligrams/kilogram
[c] Elutriate from the German DIN 38414-S4 Leachability Test
[d] Milligrams per liter

- Soil and debris treatment. Excavated soil and debris that is contaminated at concentrations higher than those listed in Table 2-2 (right column) will be treated at the nearby Harbauer facility.

- The treated soil will be landfilled near the Harbauer facility at the CFM landfill. Excavated soil from the CFM site containing less than 50 mg/kg mercury is landfilled directly. Soil with less than 10 mg/kg mercury may be used to backfill excavations on the site (see Table 2-2 for criteria).

Due to the heavy mercury contamination at the CFM site, selected areas of the site were used to demolish buildings in emissions-free enclosures. To accomplish this, an enclosure was built over the most highly contaminated buildings and an off-gas control system was installed to collect and decontaminate the dusts and vapor emissions from the enclosure. The demolition activities, most of which were accomplished manually, started in 1990 and were completed in the end of 1992. Approximately 5,000 metric tons of debris, consisting of the surface layers of brick walls and CFM production residuals, were found to be highly contaminated.

Contaminated debris from demolition activities exceeding a mercury concentration of 50 mg/kg is to be treated at the Harbauer facility. The overall objective of debris treatment is to reduce the concentration of mercury to less than 50 mg/kg so that the treated debris can be disposed of in the CFM landfill. Soil from the site is to be excavated to a depth of 4 m below ground surface in batches of 250 to 300 metric tons per day. Excavated soil, which consists of alternating layers of sand and clay, will either containing less than 10 mg/kg mercury can be used as backfill soil on the CFM site or disposed of in aundergo soil washing and vacuum-distillation or will be landfilled without treatment. Excavated soil municipal landfill. Excavated soil containing mercury at concentrations greater than 10 mg/kg but less than 50 mg/kg can be landfilled in the CFM landfill that was designed for this project. Excavated soil containing mercury at concentrations greater than 50 mg/kg is treated at the Harbauer soil washing/vacuum-distillation facility in Wölsau. The cleanup standard for mercury in the soil is 50 mg/kg (see Table 2-2 for cleanup criteria for other contaminants).

During excavation operations, different materials (for example, foundation debris, pavement debris, fill soil, loamy soil, and gravel) are separated. The excavated materials are stored in stockpiles for characterization. After determining the level of contamination, the material is transported to the destinations mentioned above according to the concentration of mercury present. A total mass of approximately 100,000 metric tons will be excavated during the remedial action. Approximately 70,000 metric tons of contaminated soil and debris are expected to be treated at the Harbauer facility.

Groundwater at the CFM site is contaminated with mercury, and aromatic, chlorinated, and petroleum hydrocarbons. The extent of groundwater contamination was investigated in 1992 and the results used to design a conventional on-site pump and treat system. Treatment consists of precipitation, flocculation, sedimentation, ion exchange, and activated carbon polishing. Groundwater remediation activities are still under way.

2.1.2 Demonstration Objectives and Approach

Demonstration objectives were selected to provide potential users of the Harbauer technology with the necessary technical information to assess the applicability of the treatment system to other contaminated sites. This bilateral SITE demonstration selected five primary objectives and three secondary objectives to evaluate the Harbauer technology. These demonstration objectives are summarized below:

<u>Primary Objectives:</u>

P-1 Document the mercury concentration in the treated soil at a confidence level of 95 percent.

P-2 Determine the mercury removal efficiency achieved by the Harbauer treatment system.

P-3 Document the levels of Toxicity Characteristic Leaching Procedure (TCLP)-leachable mercury in the untreated and the treated soil.

P-4 Document stack gas and process water treatment effluent mercury concentrations.

P-5 Determine the mercury removal efficiency of the vacuum-distillation process.

The primary objectives were achieved by collecting representative samples of contaminated feed soil, treated soil, treated process water, and stack gas during three test runs.

Secondary Objectives:

S-1 Document the moisture content and the particle size distribution of the contaminated feed soil and the treated soil from the soil washing process and the vacuum-distillation process.

S-2 Document key nonproprietary system operating parameters.

S-3 Document remediation costs per metric ton of soil.

The secondary project objectives and the associated noncritical measurement parameters required to achieve those objectives are listed in Table 2-3.

To meet demonstration objectives, data were collected and analyzed using the methods and procedures summarized in the following section.

2.2 Demonstration Procedures

This section describes the methods and procedures used to collect and analyze samples for the bilateral SITE demonstration of the Harbauer technology. The activities associated with the Harbauer SITE

Table 2-3. Noncritical Measurement Parameters

Secondary Objective	**Measurement Parameter**
Document moisture content and particle size distribution of feed soil entering, and treated soil exiting, the soil washing process and the vacuum-distillation process	Moisture content and particle size distribution in feed soil, treated soil, filter-pressed fine soil fraction, and treated fine soil fraction.
Document key nonproprietary system operating parameters.	Temperature of dryer, temperature of vacuum-distillation unit, feed soil flow rate, fresh water usage, stack gas flow rate.
Document remediation costs per metric ton of soil.	Commercial treatment costs[a]

[a] Commercial treatment costs will be obtained from Harbauer. Capital and operating costs will not be assessed in detail since Harbauer considers this information proprietary.

demonstration included (1) demonstration design, (2) soil, process water, and stack gas sample collection and analysis, and (3) field and laboratory QA/QC. The methods used to collect and analyze samples were conducted in accordance with the procedures outlined in Sections 2.2.1 and 2.2.2. Field and laboratory QA/QC procedures are described in Section 2.2.3.

2.2.1 Demonstration Design

This section describes the demonstration design, sampling and analysis program, and sample collection frequency and locations. The purpose of the demonstration was to collect and analyze samples of known and acceptable quality to achieve the objectives stated in Section 2.1.2.

2.2.1.1 Sampling and Analysis Program

Specific sampling objectives for the demonstration of the Harbauer technology are given below:

- Collect representative samples. Samples were collected in a manner and frequency to ensure that the samples were representative of the medium being sampled.
- Conduct appropriate and necessary physical and chemical characterizations of the representative samples. Samples were collected and analyzed for the necessary target compounds to achieve demonstration project objectives.
- Maintain proper chain-of-custody control of all samples, from collection to analysis.
- Follow quality control and quality assurance procedures appropriate for U.S. EPA ORD Category II projects.
- Samples (soil, process water, and air) were collected by ArGe focon-Probiotec to evaluate technology performance.

At the demonstration kick-off meeting on November 22, 1994, Harbauer informed the demonstration team that during the preceding 4 to 6 weeks there had been a change in the nature of the contaminated feed soil excavated from the CFM site. The feed soil was still highly contaminated, but now contained a large amount of coarse construction debris. The construction debris was a much more porous material than the coarse natural gravel found at the site. The porous nature of the construction debris meant that the contaminants were not just located on the outer surface as they occur on the coarse natural gravel, but were also present within the matrix of the construction debris. This change in the nature of the feed material required Harbauer to modify the soil washing process to treat the coarse (2 to 60 mm) material. An additional crushing/screening step was added to the system after the final screening/spray washing step for the 2 to 60 mm material. This crusher broke down the construction debris into particles that were then fed into the vacuum-distillation process. Oversize material from this additional crushing/screening step was then fed into the mixer for blending with the treated fines. This change in the nature of the feed material and the resulting process adjustments meant that 90 percent of the feed material was planned to be treated by the vacuum-distillation process during the demonstration, rather than the usual 70 percent.

However, during the first planned test run on November 22, 1994, the gearbox for the motor that runs the mixing unit broke. Therefore, treated coarse material from the soil washing process could no longer be blended with the treated fine material from the vacuum-distillation process and discharged for disposal in the CFM landfill. As such, all coarse material from the soil washing process had to be crushed in the additional crushing/screening step described above and fed into the vacuum-distillation process. Any oversize material from the crushing/screening step was fed back into the crusher until it was sufficiently fine-grained for treatment in the vacuum-distillation process. These changes to the process configuration and a problem that developed with the air sampling equipment caused the team to cancel the first run on November 22, 1994 and reschedule it for the next day.

During the three test runs for the Harbauer demonstration, all contaminated material was treated by the soil washing process and the vacuum-distillation process. As stated previously, the limiting factor for the overall system is the vacuum-distillation process throughput. Due to the operational configuration changes described above, the vacuum-distillation process throughput during the demonstration was lower than during routine operations. Two 8-hour and one 6-hour test runs were conducted.

The changes discussed above affected the demonstration soil sampling program. The breakdown of the mixer unit and the resulting change in process operations meant that one of the planned sampling points for the demonstration was not part of the modified process configuration. No samples could be taken of the treated soil exiting the mixer. All samples that were to be taken at this location were instead collected at sampling location S3 (see Figure 8), the treated fines exiting the vacuum-distillation process cooler. Sampling location S3 was located outside the facility building at a new sampling port that had been constructed on the conveyor belt that transports the treated fines to the mixer unit.

The presence of significant amounts of construction debris in the contaminated feed soil necessitated a change in the sample compositing scheme for contaminated soil samples collected at location S1. The QAPP stated that for samples collected at location S1, all material larger than 20 mm in diameter would be removed from the hourly composite sample, weighed and then returned to the process. Because of the porous nature of the construction debris, this material was more highly contaminated than similarly sized, naturally occurring gravels and cobbles at the CFM site. Removal of the coarse construction debris would have greatly influenced the representativeness of the samples. Therefore, it was decided to crush all material greater than 20 mm to a finer size so that it could be included in the sample for analysis. A small portable crusher was provided by Harbauer for this purpose. The crusher was cleaned by processing clean sand through the mechanism.

During the second 8-hour test run on November 24, 1994, Harbauer notified the demonstration team of a potential problem with the third test run scheduled for the following day. Harbauer expressed concern that the entire operation might have to be shut down the following day before the scheduled completion of the third test run, due to difficulties in repairing the mixer unit and the storage capacity of the facility. Taking these concerns into account, it was decided to conduct the third test run immediately after the second test run was completed. Due to facility staffing limitations, the third test was reduced to a 6-hour run. In order to insure comparable sample representativeness in the composite samples, the grab sample frequency was reduced to 7.5 minutes from 10 minutes. Therefore, a complete set of samples could be taken during this reduced test run.

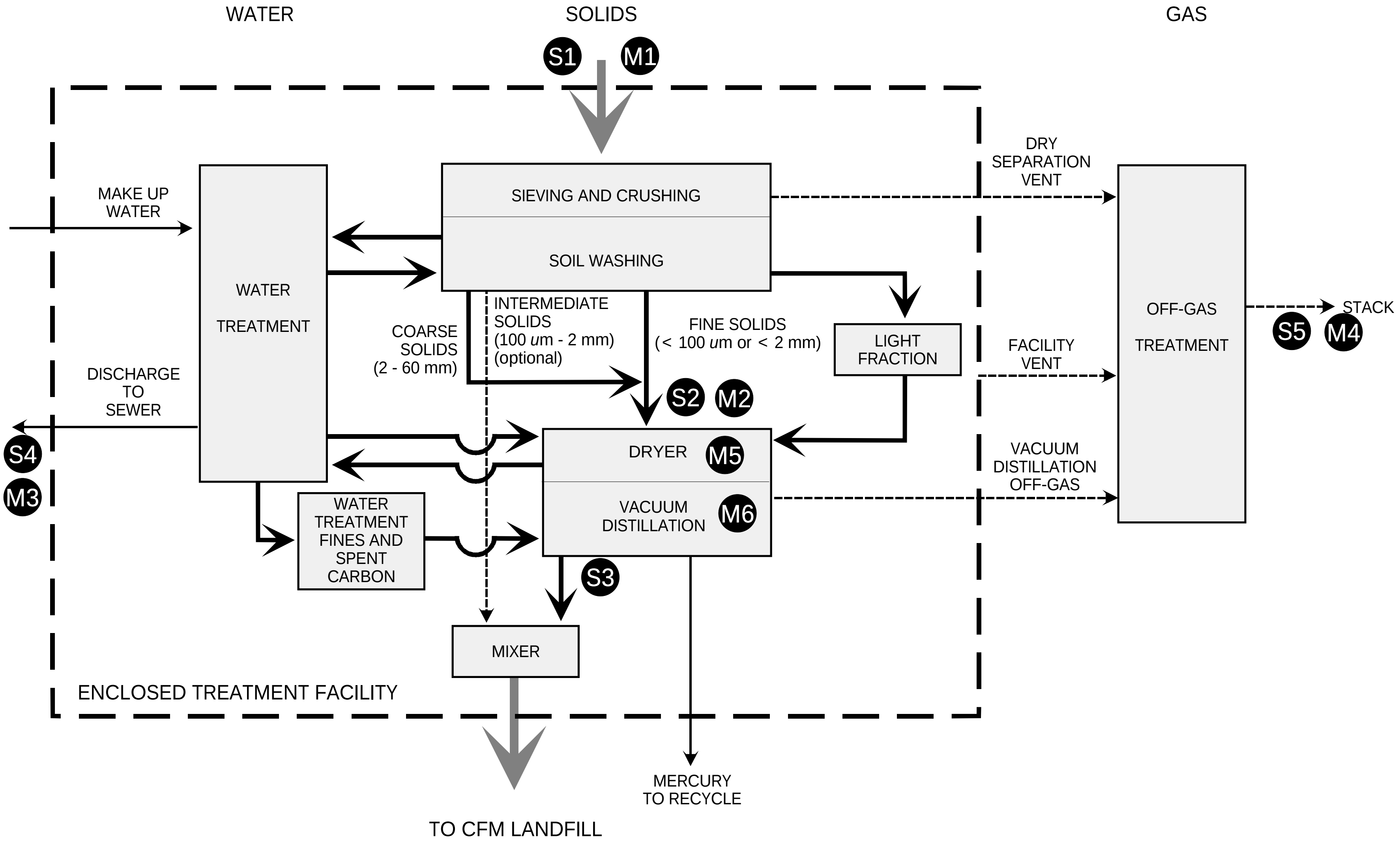

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FIGURE 8
DEMONSTRATION
SAMPLING LOCATIONS

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Not all of the data required to accomplish one of the primary objectives (P-4) were collected during the demonstration. Primary objective P-4 is to document the stack gas and process water mercury concentrations during system operation. Process water was collected and analyzed; however, stack gas was not sampled or analyzed during the November 23-24, 1994 test run.

Stack gas samples, as well as additional soil samples, were collected on July 13 and 14, 1995, while the system was operated in the same manner using similarly contaminated soil as during the November 23 and 24, 1994 demonstration. Stack gas, contaminated soil, and treated soil samples were collected and analyzed in accordance with the QAPP; however, the sampling frequency was modified as described below.

During the stack sampling, soil samples were collected from three sampling location (S1, S3, and S4). Soil samples were collected according to the methodology described in the QAPP except that one grab sample was collected at each 30-minute interval during 4 hours of system operation. These eight soil grab samples were homogenized into one composite sample for laboratory analysis. The composite soil sample represented 4 hours of system operation. During the 4 hours of system operation, one stack gas sample was collected. This sampling methodology was repeated during three 4-hour periods of system operation. The soil samples were analyzed for total mercury and moisture content only.

2.2.1.2 Sampling and Measurement Locations

Sampling locations were selected based on the configuration of the treatment system and project objectives; analytical parameters were selected based on the contaminant to be treated and project objectives. The locations at which samples were collected and measurements taken during the demonstration are shown on Figure 8.

Samples were collected from five sampling locations and measurements were taken at six measurement points in the treatment system to achieve the project objectives established in Section 2.1.2. Specific sampling and measurement procedures are described in Section 2.2.2. Sampling and measurement activities were conducted over two 8-hour test runs and one 6-hour test run, as described above. Grab and composite sampling techniques were employed throughout the demonstration. Figure 8 shows sampling locations for solids, liquids, and gases. These sampling locations are:

- Sampling Location S1: Contaminated feed soil. Contaminated soil and construction debris were introduced into the crusher and fed into the blade washer unit via a conveyor belt. Samples of the contaminated soil were collected from the convey or belt.
- Sampling Location S2: Dewatered contaminated fines from the soil washing process. Contaminated fine soil from the soil washing process was pumped in a suspension to the vacuum-distillation process. The slurry is thickened by adding flocculants and is subsequently dewatered in a filter press to form a filter cake. After storage in a buffer container, the fines are fed to the dryer and vacuum-distillation unit via a conveyor belt. Samples of the contaminated fines were collected from the conveyor belt.
- Sampling Location S3: Treated fines exiting the vacuum-distillation process cooler. After passing the vacuum-distillation process, the treated fines are cooled and pass the outlet vacuum lock. Samples of the treated fines were collected from a sample port on the enclosed conveyor belt that transports treated soil to the mixer unit.
- Sampling Location S4: Treated process water. Samples were collected from an in-line sampling tap before the treated water was discharged to the municipal sewer
- Sampling Location S5: Stack gas. Samples were collected from sampling ports located on the exhaust
- Measurement Location M1: Contaminated feed soil. The mass of the contaminated soil fed through the crusher was measured using a certified truck scale. Measurement Location M2: Dewatered contaminated fines. The mass of the dewatered contaminated fines was measured using a belt scale the belt conveyor to the vacuum-distillation unit.
- Measurement Location M3: Treated process water. The amount of treated process water discharged to the municipal sewer was measured using rotameters prior to discharge.
- Measurement Location M4: Stack gas. Flow rate in the exhaust stack was measured using EPA Method 101A**.**
- Measurement Location M5: Temperature of the dryer unit. The temperature was monitored continuously by Harbauer using a temperature probe (thermocouple) that reaches into the soil stream exiting the unit.
- Measurement Location M6: Temperature of the vacuum-distillation unit. The temperature is monitored continuously by Harbauer using a temperature probe (thermocouple) that reaches into the soil stream within the vacuum-distillation unit.

2.2.2 Sampling and Analytical Methods

This section describes the procedures for collecting representative samples at each sampling location and analyzing collected samples. Samples were collected at five locations. These locations include three soil sampling points, one liquid sampling point, and one gas sampling point, as described in the previous section. System operating parameters were monitored continuously by Harbauer. Sampling began after Harbauer judged that the system was operating at steady state.

2.2.2.1 Soil Samples

Contaminated feed soil, dewatered contaminated fines, and treated soil are transported by dedicated conveyor belts in the Harbauer facility. Samples collected to characterize the three process streams were obtained from conveyor belts. Grab samples were obtained from the conveyor belt position closest to the process (for example, feed soil was sampled just before it entered the soil washing unit). The treated soil was sampled using a sampling port located directly after the outlet vacuum lock.

Composite soil samples were collected at sampling locations S1, S2, and S3 for analysis of critical and noncritical parameters. Grab soil samples were collected from the conveyor belts using a plastic scoop containing approximately 1 liter of soil at a frequency described in Section 2.2.1.1 and summarized in Table 2-4. The soil was immediately transferred to precleaned 4-gallon plastic bowls, resulting in about 6 liters of sample for each of the one-hour composites.

The sample material was transferred into the appropriate precleaned sample containers with minimal headspace for leachable and total mercury analyses. A measured quantity of preservative (zinc powder) was then added to each sample container for total mercury analysis. Archive samples for total and TCLP-leachable determinations were also collected (zinc powder was not added to the samples collected for TCLP analysis). Any soil that was not needed for samples was returned to the specific conveyor belt from which it was collected. Table 2-5 lists the analytical procedures used for samples collected during the demonstration and Figure 9 shows the sample preparation scheme.

Table 2-4. Demonstration Sampling Schedule And Compositing Scheme For Sampling Conducted On November 23 and 24, 1994[a]

Sampling Locations	Hours	1	2	3	4	5	6	7	8
S1 (Feed Soil) and S2 (Feed Fines)	Minutes	0 10 20 30 40 50	0 10 20 30 40 50	0 10 20 30 40 50	0 10 20 30 40 50	0 10 20 30 40 50	0 10 20 30 40 50	0 10 20 30 40 50	0 10 20 30 40 50
	Grab	1 1 1 1 1 1		1 1 1 1 1 1		1 1 1 1 1 1		1 1 1 1 1 1	
	Composite	1		1		1		1	
	Daily Composite	1							
S3 (Treated Soil)	Grab		1 1 1 1 1 1		1 1 1 1 1 1		1 1 1 1 1 1		1 1 1 1 1 1
	Composite		1		1		1		1
	Daily Composite	1							
S4 (Treated Process Water)	Grab	1							
S5[b] (Stack gas)		1							

a Sampling schedule for Runs 1 and 2. Run 3 was completed in 6 hours instead of 8 hours; therefore, soil samples were collected at intervals of 7.5 minutes instead of 10 minutes so that the same number of soil samples were generated during the test run.

b Stack gas was sampled on July 13 and 14, 1995 over three 4-hour test runs.

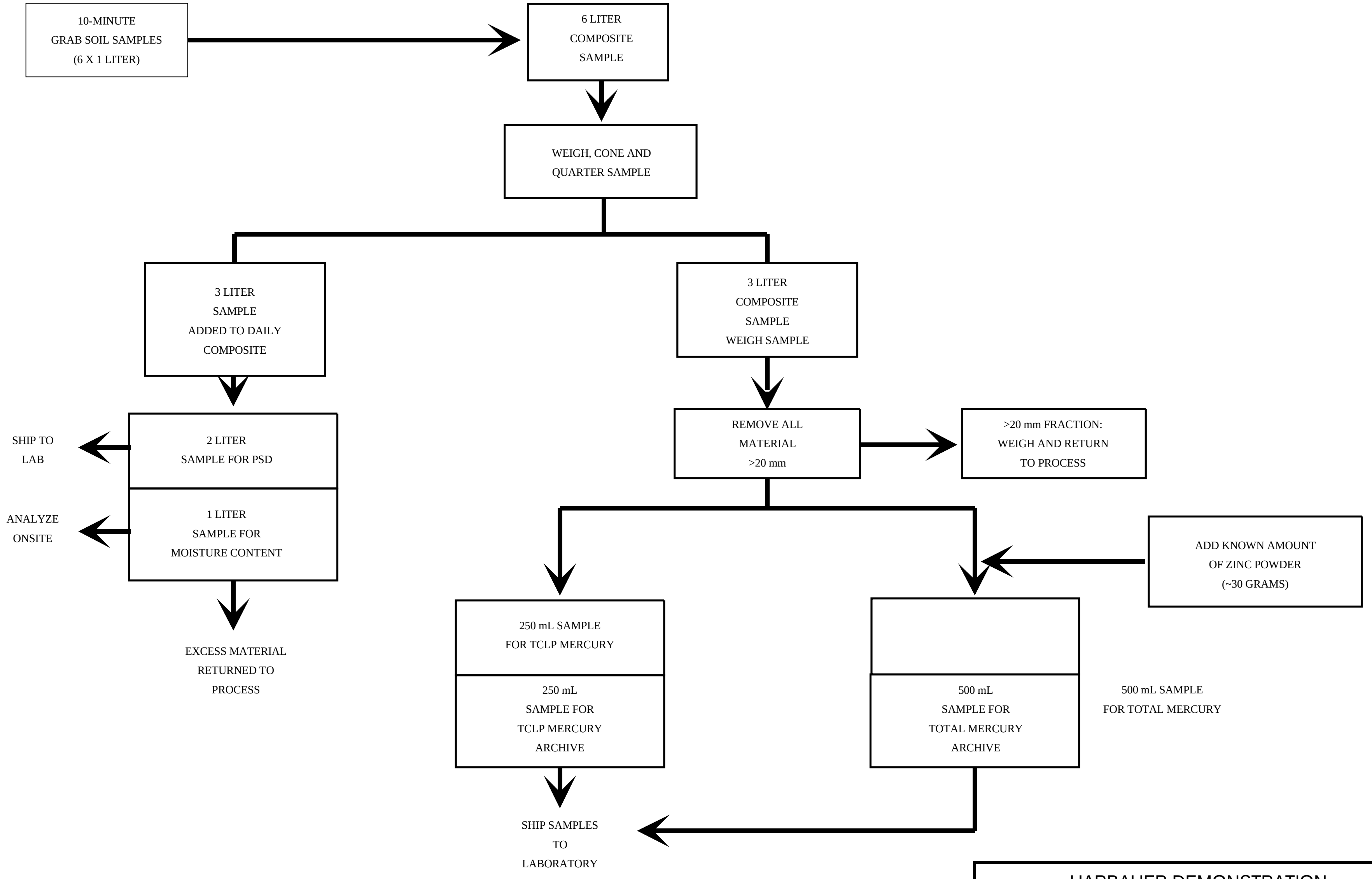
Table 2-5. Analytical Methods

Matrix	Parameter	Method	Reference
Soil	Total Mercury	7471	EPA 1987
	TCLP-leachable Mercury	1311/7470	EPA 1987
	Percent Moisture	D2216	ASTM 1990
	Particle Size	Standard Operating Procedure	Institut Fresenius
Aqueous	Mercury	7470	EPA 1987
Gaseous	Mercury	101A/7470	40 CFR 61/ EPA 1987

The particle size distribution was determined using an Institute Fresenius Standard Operating Procedure, which is based on the German Standard Method DIN 19 683 Parts 1 and 2. This method incorporates sampling a soil suspension in a settling cylinder at timed intervals and determining the concentration of solids in suspension at each interval.

2.2.2.2 Process Water Samples

Process water samples were collected from a sample tap, at the location identified in Section 2.2.1 and shown on Figure 8, directly into precleaned sample containers (provided by a commercial suppler). The samples collected for laboratory analysis were preserved appropriately for the tests to be performed. Sample container types and preservatives were specified in the QAPP. A grab sample was collected midway through each test run. The containers were filled as full as possible to eliminate any head space.



HARBAUER DEMONSTRATION
WÖLSAU, GERMANY

FIGURE 9
DEMONSTRATION SAMPLE
PREPARATION SCHEME

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When liquid samples for determination of mercury were collected, the sample was introduced into the vials gently to reduce agitation that might drive off mercury. The samples were collected directly into the vial without introducing any air bubbles. Each vial was filled until a meniscus appeared over the top.The screw-top lid with the septum (Teflon side toward the sample) was then screwed onto the vial. After tightening the lid, the vial was inverted and tapped to check for air bubbles. If any air bubbles were present, another sample was collected by filling a new, preserved vial. Table 2-5 lists the analytical procedures used for samples collected during the demonstration.

2.2.2.3 Stack Gas Samples

Stack gas samples were collected from sampling ports in the process air stack. One stack gas sample was collected during each of three 4-hour test runs on July 13 and 14, 1995. Samples were collected according to standard U.S. EPA stack sampling methods as published in 40 Code of Federal Regulations (CFR) Part 60 (Standards of Performance for New Stationary Sources, Appendix A, Reference Methods). The sampling point consisted of two port entries at a 90 degree angle to each other on a horizontal plane. The stack internal diameter at the sampling point was 1.2 m. Traverse point locations were determined based on the criteria stated in the sampling method.

Samples for analyses of mercury in the gas stream were collected by a modified Method 5 sampling train, as described in Method 101A of 40 CFR Part 61 (Appendix B, Test Methods). This sampling method was implemented without a particulate trap, because particulates were removed by the activated carbon filter. The entire sampling train was thoroughly rinsed according to method specifications and the rinsate was analyzed along with the collected sample. Table 2-5 lists the analytical procedure used for samples collected during the demonstration.

2.2.3 Quality Assurance and Quality Control Program

Quality control checks and procedures were an integral part of the Harbauer bilateral SITE demonstration to ensure that the QA objectives were met. These checks and procedures focused on the collection of representative samples absent of external contamination and on the generation of comparable data. The QC checks and procedures conducted during the demonstration were of two kinds: (1) checks controlling field activities, such as sample collection and shipping, and (2) checks controlling laboratory activities, such as extraction and analysis. The results of the field and laboratory quality control checks are summarized below and in Section 2.3.3.

2.2.3.1 Field Quality Control Checks

As a check on the quality of field activities including sample collection, shipment, and handling, three types of field QC checks (field blanks, trip blanks, and equipment blanks) were collected. In general, these QC checks assess the representativeness of the samples, and ensure that the degree to which the analytical data represent actual site conditions is known and documented. The field QC results are reported in Section 2.3.3 and were all within acceptable limits.

2.2.3.2 Laboratory Quality Control Checks

Laboratory QC checks are designed to determine the precision and accuracy of the analyses, to demonstrate the absence of interferences and contamination from glassware and reagents, and to ensure the comparability of data. Laboratory-based QC checks consisted of method blanks, matrix spikes/matrix spike duplicates, surrogate spikes, blank spikes/blank spike duplicates, and other checks specified in the analytical methods. The laboratory also performed initial calibrations and continuing calibration checks according to the specified analytical methods. The results of the laboratory internal QC checks for critical parameters were all within acceptable limits and are summarized in Section 2.3.3.

2.2.3.3 Field and Laboratory Audits

EPA technical systems reviews of field and laboratory activities were conducted during the weeks of November 21 and November 28, 1994 respectively. During these reviews, observations and suggestions were noted in the areas of (1) project organization and quality assurance management, (2) field sampling operations and field measurements, (3) sample log-in and custody, and (4) laboratory procedures. Appendix B provides a copy of the observations and recommendations report for these reviews. The review concluded that field sampling activities likely produced representative samples that will meet project objectives. The report further concluded that, assuming effective preparation is completed for the TCLP, laboratory analysis activities should produce data that are of known and adequate quality to meet project objectives. Results of soil samples collected during the July 1995 test runs indicate that mercury concentrations in untreated soil samples were much lower (less than half) than during the November 1994 test runs. This difference may indicate that mercury concentrations measured in the stack gas may not have been indicative of mercury concentrations during the November 1994 test runs. Mercury concentrations in the treated soil were comparable during all test runs.

2.3 Demonstration Results And Conclusions

This section presents the operating conditions, results and discussion, data quality, and conclusions of the bilateral SITE demonstration of the Harbauer soil washing/vacuum-distillation technology. This bilateral SITE demonstration provides the most extensive Harbauer treatment system performance data to date and serves as the foundation for conclusions on the system's effectiveness and applicability to other remediation projects.

2.3.1 Operating Conditions

This section summarizes the configuration of the Harbauer system and operating parameters during the SITE demonstration. During this bilateral SITE demonstration, the Harbauer treatment system was operated at conditions determined by the developer. To document the Harbauer system's operating conditions, soil, water, and air were periodically monitored and sampled. The system operated continually for 8 hours (0800 to 1600) on November 23, 1994 and for 14 hours (0800 to 2200) in two runs on November 24, 1994 over the demonstration period. The demonstration consisted of two 8-hour test runs and one 6-hour test run. The Harbauer technology is presented by the developer as a highly efficient system for the removal of mercury from soil and construction debris (concrete and brick). The Harbauer facility at Marktredwitz, Germany, was designed to remove mercury from contaminated soil, and this demonstration did not evaluate the removal of any other contaminants.

2.3.1.1 Treatment System Configuration

The Harbauer treatment system includes a soil crusher, a blade soil washing unit, an attrition scrubber, hydrocyclones, soil dryer, vacuum-distillation unit, mixer, process water piping, stack gas ducting, and carbon adsorption units. For this demonstration, all of the contaminated soil and construction debris was crushed and then treated using the vacuum-distillation unit. This demonstration was a more stringent test of the system because removal of mercury from the construction debris (concrete and bricks, for example) matrix is more difficult than from the usual sandy and loamy soil. The soil mixer was not used for reasons stated in Section 2.2.1.1. The configuration of the Harbauer treatment system components is shown in Figures 4 and 8.

2.3.1.2 Operating Parameters

The developer monitored the Harbauer soil treatment system throughout the demonstration. System operating parameters monitored included temperature of the soil dryer and vacuum-distillation unit, throughput rate of soil through the soil washing process and the vacuum-distillation unit, flow rate of process water discharged to the sewer, and stack gas flow rate. A summary of the operating parameters measured during the demonstration is presented in Table 2-18.

2.3.2 Results and Discussion

This section presents the results of the bilateral SITE demonstration of the Harbauer technology at Marktredwitz, Germany. The results are presented by and have been interpreted in relation to project objective. The specific primary and secondary objectives are shown at the top of each section in italics followed by a discussion of the objective-specific results. The data used to evaluate the primary objectives are presented in Tables 2-6, 2-7, and 2-8. Data quality and conclusions based on these results are presented in Sections 2.3.3 and 2.3.4, respectively.

Table 2-6. Mercury Concentrations In Feed Soil (Sampling Location S1)

Run No.	Date	Time	Mercury Concentration (mg/kg)[a]	Average Mercury Concentration for Test Run (mg/kg)	ASTM Moisture Content (Percent)
1	11-23-94	0800-0900	800	780	17.1
		1000-1100	568		
		1200-1300	808		
		1400-1500	943		
2	11-24-94	0800-0900	965	766	17.4
		1000-1100	571		
		1200-1300	880		
		1400-1500	647		
3	11-24-94	1600-1645	1,270	1,080	19.5
		1730-1815	731		
		1900-1945	944		
		2030-2115	1,370		

[a] Milligrams per kilogram

Table 2-7. Mercury Concentrations In Dewatered Fines From The Soil Washing Process (Sampling Location S2)

Run No.	Date	Time	Mercury Concentration (mg/kg)[a]	ASTM Moisture Content (Percent)	Moisture Content Correction Factor	Normalized Mercury Concentration[b] (mg/kg)	Average Normalized Mercury Concentration[b] (mg/kg)
1	11-23-94	0800-0900	1,500	33.8	1.25	1,880	1,450
		1000-1100	1,320			1,650	
		1200-1300	1,490			1,870	
		1400-1500	339			424	
2	11-24-94	0800-0900	889	37.4	1.32	1,170	1,070
		1000-1100	1,060			1,410	
		1200-1300	629			830	
		1400-1500	652			861	
3	11-24-94	1600-1645	742	19.0	0.994	738	483
		1730-1815	465			462	
		1900-1945	405			403	
		2030-2115	330			328	

a Milligrams per kilogram
b Mercury concentrations normalized to moisture content of feed soil.

Table 2-8. Mercury Concentrations In Treated Soil From The Vacuum-Distillation Process (Sampling Location S3)

Run No.	Date	Time	Mercury Concentration (mg/kg)[a]	ASTM Moisture Content (Percent)	Moisture Content Correction Factor	Normalized Mercury Concentration[b] (mg/kg)	Average Normalized Mercury Concentration[b] (mg/kg)
1	11-23-94	0900-1000	25.8	0.03	0.82	21.2	18.7
		1100-1200	26.3			21.6	
		1300-1400	20.9			17.1	
		1500-1600	17.9			14.7	
2	11-24-94	0900-1000	15.9	0.00	0.82	13.0	17.5
		1100-1200	15.6			12.8	
		1300-1400	23.8			19.5	
		1500-1600	30.1			24.7	
3	11-24-94	1645-1730	26.7	0.02	0.82	21.9	16.2
		1815-1900	16.6			13.6	
		1945-2030	13.9			11.4	
		2115-2200	21.9			18.0	

[a] Milligrams per kilogram
[b] Mercury concentrations normalized to moisture content in feed soil.

2.3.2.1 Primary Objectives

Primary objectives were considered critical for the evaluation of the Harbauer treatment system. The results for each primary objective are discussed in the following subsections.

2.3.2.1.1 Primary Objective P-1

Document the mercury concentration in the treated soil at a confidence level of 95 percent.

This objective was achieved by collecting samples of the treated soil that was discharged from the vacuum-distillation unit and analyzing the samples for mercury.

The 95 percent upper confidence limit (UCL) for mercury in the treated soil was calculated using the mercury concentration data presented in Table 2-8 and the following equations:

$$UCL_{t,95\%} = x + \frac{ts}{\sqrt{n}}$$

where:

x = Treated soil arithmetic mean mercury concentration normalized to the feed soil moisture content (see equation below)
t = Student's t-test value for a one-tail test at the 95 percent confidence level
s = Sample standard deviation
n = Sample size (number of measurements)

$$C_{tn} = (C_t)\ (Z)$$

where:

C_{tn} = Mercury concentration in treated soil normalized to the arithmetic mean feed soil moisture content (mg/kg)
C_t = Mercury concentration in treated soil (mg/kg)
Z = Normalization factor

The normalization factor was calculated by using the following equation:

$$Z = \frac{1 - M_u}{1 - M_t}$$

where:

Z = Normalization factor
M_u = Arithmetic mean moisture content of untreated soil (grams per gram [g/g])
M_t = Arithmetic mean moisture content of treated soil (g/g)

The 95 percent upper confidence limits for mercury in the treated soils are presented in Table 2-9. These results indicate that the Harbauer technology can reduce mercury concentrations in soil to between 21 to 25 mg/kg at the 95 percent upper confidence level.

Table 2-9. 95 Percent Upper Confidence Limit (UCL) Of Mercury Concentrations In Treated Soil

Run No.	95% UCL of Mercury Concentration (milligrams/kilogram)
1	22.6
2	24.2
3	21.7

2.3.2.1.2 Primary Objective P-2

Determine the mercury removal efficiency achieved by the Harbauer treatment system.

To determine the mercury removal efficiency of the system, samples of soil entering and exiting the system were collected during the three test runs. The removal efficiency was calculated based on mercury concentrations in the feed and treated soil (the treated soil is that discharged from the vacuum-distillation unit). For this objective, the difference between the mercury concentrations in the contaminated feed soil and the treated soil is considered the critical parameter. The appropriate number of samples and the sample collection methods were discussed in Section 2.2.

The mercury removal efficiencies were calculated for each run using the following equation:

$$\% R = \frac{C_u - C_{tn}}{C_u} \times 100\%$$

where

$\%R$ = Contaminant reduction efficiency (%)
C_u = Arithmetic mean untreated soil contaminant concentration (mg/kg)
C_{tn} = Arithmetic mean treated soil contaminant concentration normalized to the arithmetic mean moisture content of the contaminated feed soil (mg/kg)

Contaminant removal efficiencies were 97.6 percent, 97.7 percent, and 98.5 percent for the three test runs. The results are presented in Table 2-10.

The results of this demonstration indicate that the Harbauer technology can reduce mercury concentrations in crushed construction debris (concrete, bricks, etc.), sandy loam soil, and loam soil from an average of 875 mg/kg to an average of 17 mg/kg.

2.3.2.1.3 Primary Objective P-3

Document the levels of Toxicity Characteristic Leaching Procedure (TCLP)-leachable mercury in the untreated and the treated soil.

To document the levels of TCLP-leachable mercury, samples of soil entering and exiting the system were collected during the three test runs. For this objective, TCLP-leachable mercury in the

Table 2-10. Mercury Removal Efficiency Achieved By The Harbauer Treatment System

Run No.	Average Mercury Concentration in Feed Soil (mg/kg)[a]	Average Mercury Concentration in Treated Soil (mg/kg)	Average Mercury Removal Efficiency (Percent)
1	780	18.7	97.6
2	766	17.5	97.7
3	1,080	16.2	98.5
Average	875	17.4	98.0

a Milligrams per kilogram

contaminated feed soil and the treated soil are considered critical parameters. The average TCLP mercury concentrations in the contaminated feed soil was 82 μg/L and in the treated soil was 6 μg/L. The U.S. federal maximum contaminant level (MCL) for mercury is 2μg/L. The results are presented in Table 2-11.

2.3.2.1.4 Primary Objective P-4

Document the stack gas and water treatment effluent mercury concentrations.

The mercury concentrations in the water treatment process effluent, which was discharged to the sewer, and mercury concentrations in the stack gas were measured during the three test runs. The average mercury concentration in the stack gas measured on July 13 and 14, 1995, was 2.92 micrograms per normal cubic meter (µg/NCM) (Table 2-12 shows individual test run results). The average mercury concentration in the treated process water was 4.96 µg/L (Table 2-13 shows the individual test run results). The U.S. federal MCL for mercury is 2μg/L. Since the stack gas was not sampled during the November 1994 test runs, additional soil samples were collected during the July 1995 test runs. As shown in Table 2-14, results of soil samples collected during the July 1995 test runs indicate that mercury concentrations in untreated soil samples were much lower (less than half) than during the

Table 2-11. TCLP[a] - Leachable Mercury Concentrations In Untreated And Treated Soil

Run No.	Date	Sampling Location S1 (Untreated Soil)			Sampling Location S3 (Treated Soil)		
		Time	Mercury Concentration in Leachate (µg /L)[b]	Average Mercury Concentration (µg /L)	Time	Mercury Concentration in Leachate (µg /L)	Average Mercury Concentration (µg /L)
1	11/23/94	0800-0900	75.6	**96.17**	0900-1000	0.02	**0.62**
		1000-1100	92.4		1100-1200	0.82	
		1200-1300	122.1		1300-1400	0.59	
		1400-1500	94.5		1500-1600	1.03	
2	11/24/94	0800-0900	57.4	**53.53**	0900-1000	1.15	**2.18**
		1000-1100	34.0		1100-1200	0.12	
		1200-1300	72.6		1300-1400	0.70	
		1400-1500	50.1		1500-1600	6.76	
3	11/24/94	1600-1645	54.1	**97.36**	1645-1730	15.6	**14.6**
		1730-1815	127.6		1815-1900	24.8	
		1900-1945	71.9		1945-2030	7.34	
		2030-2115	135.8		2115-2200	10.5	

[a] Toxicity Characteristic Leaching Procedure
[b] Micrograms per liter

Table 2-12. Mercury Concentrations In Stack Gas (Sampling Location S5)

Run No.	Date	Concentration (μg/NCM)[a]
1A	8/24/95	5.47
2A	8/24/95	1.92
3A	8/24/95	2.28

a Micrograms per normal cubic meter

Table 2-13. Mercury Concentrations In Treated Process Water (Sampling Location S4)

Run No.	Date	Concentration (μg /L)[a]
1	11/23/94	4.39
2	11/24/94	5.80
3	11/24/94	4.68

a Micrograms per liter

November 1994 test runs. This difference may indicate that mercury concentrations measured in the stack gas may not have been indicative of mercury concentrations during the November 1994 test runs. Mercury concentrations in the treated soil were comparable during all test runs.

2.3.2.1.5 Primary Objective P-5

Determine the mercury removal efficiency of the vacuum-distillation process.

To determine the mercury removal efficiency of the vacuum-distillation process, samples of soil entering and exiting the unit were collected during the three test runs. The removal efficiency was calculated

Table 2-14. Mercury Content Of Soil Samples Collected On July 13 And 14, 1995

Run No.	Sampling Location	Mercury Concentration (mg/kg)[a]
1A	Feed Soil	92.8
2A	Feed Soil	144
3A	Feed Soil	78.4
1A	Treated Soil	15.7
2A	Treated Soil	15.8
3A	Treated Soil	29.2
1A	Dewatered Fines	175
2A	Dewatered Fines	131
3A	Dewatered Fines	143
1A	Treated Fines	14.5
2A	Treated Fines	11.2
3A	Treated Fines	8.40

[a] Milligrams per kilogram

based on mercury concentrations in the feed soil entering the vacuum-distillation unit and the treated soil exiting the vacuum-distillation unit. The feed soil is the soil in the vacuum-distillation feed conveyor and the treated soil is that discharged from the vacuum-distillation process. For this objective, mercury concentrations in these two process streams were considered critical parameters. The appropriate number of samples and the sample collection methods are discussed in Section 2.2.

The mercury removal efficiency was calculated using the formula presented in Section 2.3.2.1.2 and the results are presented in Table 2-15.

The mercury removal efficiencies of the vacuum-distillation process were 98.7 percent, 98.4 percent, and 96.6 percent for the three runs.

Table 2-15. Mercury Removal Efficiency Achieved By The Vacuum-Distillation Process

Run No.	Average Mercury Concentration in Dewatered Fines (mg/kg)[a]	Average Mercury Concentration in Treated Soil (mg/kg)[a]	Average Mercury Removal Efficiency (Percent)
1	1,450	18.7	98.7
2	1,070	17.5	98.4
3	483	16.2	96.6

a Milligrams per Kilogram

2.3.2.2 Secondary Objectives

Secondary objectives provide additional information that is useful, but not critical, for the evaluation of the Harbauer technology. Three secondary objectives were selected for the bilateral SITE demonstration of the Harbauer system. The secondary project objectives and the associated noncritical measurement parameters required to achieve those objectives were presented in Table 2-3.

The results of each secondary objective are discussed in the following subsections.

2.3.2.2.1 Secondary Objective S-1

Document the moisture content and the particle size distribution of feed soil and treated soil from the soil washing process and the vacuum-distillation process.

The native moisture content of the contaminated soil from the CFM site ranged from 17.1 percent to 19.5 percent by weight. The moisture content of the dewatered soil from the soil washing process ranged from 19 percent to 37.4 percent by weight. The moisture content of the treated soil exiting the vacuum-distillation unit ranged from 0.00 to 0.03 percent by weight. Because all of the contaminated soil and construction debris was crushed to a fairly uniform size for this demonstration, the process did not significantly change the particle size distribution (break up coarser particles and create more fines) of the soils. The moisture content and particle size distribution results are presented in Tables 2-16 and 2-17, respectively.

2.3.2.2.2 Secondary Objective S-2

Document key, nonproprietary system operating parameters.

The internal temperature of the soil dryer was 130°C during the demonstration. The internal temperature of the vacuum-distillation unit ranged from 430°C to 500°C during the demonstration. The boiling point of elemental mercury is about 350°C at 1 atmosphere pressure. The Harbauer system runs at a temperature more than 100°C higher than the boiling point of mercury and operates under a vacuum, so that the boiling point of mercury is lowered. During the demonstration, the Harbauer system processed soil at rates ranging from 2.4 metric tons per hour (mt/hr) to 3.7 mt/hr. The quantities of soil treated during the three test runs were 29.6 mt (8 hours), 20 mt (8 hours), and 14.4 mt (6 hours). Treated process water was discharged to the sewer at rates ranging from 2.9 cubic meters per hour (m^3/hr) to 10.3 m^3/hr. Quantities of treated process water discharged during the three test runs were 43.2 m^3 (8 hours), 82.4 m^3 (8 hours), and 17.4 m^3 (6 hours). The results are presented in Table 2-18.

Table 2-16. Moisture Content Of Untreated And Treated Soil

Test Run	Feed Soil (Sampling Location S1) (percent)	Dewatered Fines from Soil Washing Process (Sampling Location S2) (percent)	Treated Soil (Sampling Location S3) (percent)
1	17.1	33.8	0.03
2	17.4	37.4	0.00
3	19.5	19.0	0.02

Table 2-17. Particle Size Distribution Of Untreated And Treated Soil

Test Run	Soil Type[a]		
	Feed Soil (Sampling Location S1)	Treated Soil from the Soil Washing Process (Sampling Location S2)	Treated Soil from the Vacuum Distillation Process (Sampling Location S3)
1	Sandy Loam (53.5/36.7/9.7)[b]	Loam (44.9/44.7/10.3)	Sandy Loam (58.1/34.9/7.1)
2	Loam (48.0/40.4/11.6)	Loam (44.9/44.8/10.3)	Sandy Loam (60.7/32.9/6.4)
3	Sandy Loam (54.3/37.1/8.6)	Sandy Loam (72.2/22.1/5.7)	Sandy Loam (55.9/38.5/5.7)

[a] Based on particle size distribution according to an Institut Fresenius standard operating procedure

[b] (% sand/ % silt/ % clay)

Table 2-18. Key System Operating Parameters

Test Run (length of run in hrs)[a]	Soil Processing Rate (mt/hr)[b]	Vacuum-Distillation Unit Processing Rate (mt/hr)	Treated Process Water Flow Rate (m^3/hr)[c]	Stack Gas Flow Rate (NCM/hr)[d]	Dryer Temperature (°C)[e]	Vacuum-Distillation Unit Temperature (°C)
1 (8)	3.7	3.7	5.4	30,300	130	430
2 (8)	2.5	2.4	10.3	30,500	130	450
3 (6)	2.4	2.4	2.9	30,200	130	500

a Hours
b Metric tons per hour
c Cubic meters per hour
d Normal cubic meters per hour
e Degrees Celsius

2.3.2.2.3 Secondary Objective S-3

Document remediation costs per metric ton of soil.

The cost per metric ton is 870 DM ($580, assuming a 1.5 DM to $ exchange rate) for the client Landkreis Wunsiedel. This price covers half of the investment costs of the facility (approximately 45,000,000 DM [$30,000,000]). Harbauer estimated the price without this risk sharing (that is, if the Landkreis Wunsiedel did not have to pay for half of the capital cost of the facility) to be 480 DM ($320) per metric ton.

2.3.3 Data Quality

This section summarizes the data quality for soil, process water, and stack gas samples collected and analyzed during the Harbauer bilateral SITE demonstration. This data quality assessment was conducted to incorporate the analytical data validation results and the field data quality QC results, evaluate the impact of all QC measures on the overall data quality, and remove all unusable values from the investigation data set. The results of this assessment were used to produce the known, defensible information employed to define the investigation findings and draw conclusions.

A validation review of the analytical data for soil, process water, and stack gas samples was conducted to ensure that all laboratory data generated and processed are scientifically valid, defensible, and comparable. Data were validated using both field QC samples and laboratory QC analyses. The field samples included equipment blanks, field blanks, and trip blanks. Laboratory samples included method blanks, surrogate recoveries, initial and continuing calibration, matrix spike/matrix spike duplicate, and samples/sample duplicate. Results from these samples were used to assess data precision, accuracy, representativeness, comparability, and completeness. The results are presented in Tables 2-19, 2-20, and 2-21. Table 2-22 presents the QC results for air, soil, and water samples collected on July 13 and 14, 1995.

Table 2-19.   QC Results For Soil Analyses

	Sample Number		
	HAR-1-1-1-4-S	**HAR-1-4-1-4-S**	**HAR-2-3-1-2-S**
Matrix Spike Recovery (Percent)	95.4	96.2	101.6
Matrix Spike Duplicate Recovery (Percent)	95.7	99.0	104.1
Matrix Spike Relative Percent Difference (RPD)	2.4	2.7	6.2
QA Recovery Objective	75-125	75-125	75-125
QA RPD Objective	<25	<25	<25

Table 2-20. QC Results Of Equipment Blank, Field Blank, And Trip Blank Analyses

Test Run	Sampling Location	Blank Type	Mercury Concentration (µg /Liter)[a]
1	S1	Equipment Blank	1.60
	S3		2.60
	S4		1.67
	S1	Field Blank	1.55
	S3		1.43
	S4		1.82
	S5		1.54
	Compositing Area		1.67
Not Applicable	Not Applicable	Trip Blank	1.82
			0.95
			1.59

[a] Micrograms per Liter

For data used to draw conclusions related to project objectives, all data quality indicators met the QA objectives specified in the QAPP (PRC 1994) for the Harbauer bilateral SITE demonstration, indicating that general data quality was good and that the sample data are useable as reported. However, for soil samples collected during that stack gas sampling on July 13 and 14, 1995, several data quality outliers were noted (See Table 2-22). However, data associated with these QC results were collected only for informational purposes and were not used to draw conclusions related to the performance of the technology. All data quality indicators associated with the November sampling events met all acceptance criteria specified in the QAPP.

Table 2-21. QC Results Of Blank Spike / Blank Spike Duplicate Analyses

Date	December 7, 1994	December 12, 1994	December 19, 1994		January 2-3, 1995
			I	II	
Blank Spike Recovery (percent)	106	90.6	107	99.5	110
Blank Spike Duplicate Recovery (percent)	108	97.8	85.9	81.5	114
Relative Percent Difference (percent)	1.8	7.11	20.9	18.5	3.2
QA Recovery Objective	75-125	75-125	75-125	75-125	75-125
QA RPD Objective	<25	<25	<25	<25	<25

2.3.4 Conclusions

This section presents the conclusions of the Harbauer bilateral SITE demonstration at Marktredwitz, Germany. The overall conclusion of the demonstration is that the Harbauer treatment system consistently reduced mercury concentrations in contaminated soil (sandy loam and loam) and construction debris (concrete and brick) from the CFM site from more than 1,000 mg/kg to less than 27 mg/kg. The soil processing rate ranged from 2.4 to 3.7 metric tons per hour. For the Harbauer technology demonstration, five primary and three secondary objectives were selected. The conclusions for each objective are summarized below:

- The 95 percent upper confidence limit of mercury concentrations in treated soil for the three test runs were 22.6 mg/kg, 24.2 mg/kg, and 21.7 mg/kg.
- The mercury removal efficiencies achieved by the Harbauer treatment system for the three test runs were 97.6 percent, 97.7 percent, and 98.0 percent.

Table 2-22. QC Results For Air, Soil, And Water Samples Collected On July 13 And 14, 1995

Sample Number	Sampling Location	Data Quality Indicator	QC Result	QA Objective
HAR-2-6-1-0-S-1	Stack Gas	Matrix Spike Recovery	80.2 %	75-125
		Blank Spike Recovery	99.0 %	75-125
HAR-1-1-1-0-S	Feed Soil	Matrix Spike Recovery	88.1 %	75-125
		Matrix Spike Recovery	89.2 %	75-125
		Relative Percent Difference	2.2 %	< 25
HAR-3-3-1-0-S	Dewatered Fines	Matrix Spike Recovery	100 %	75-125
		Matrix Spike Recovery	104 %	75-125
		Relative Percent Difference	1.5 %	< 25
HAR-1-2-1-0-S	Treated Soil	Matrix Spike Recovery	82.2 %	75-125
		Matrix Spike Recovery	**69.6 %**[a]	75-125
		Relative Percent Difference	9.3 %	< 25
HAR-1-4-1-0-S	Treated Fines	Matrix Spike Recovery	**73.8 %** [a]	75-125
		Matrix Spike Recovery	**72.0 %** [a]	75-125
		Relative Percent Difference	3.4%	< 25
HAR-2-1-1-0-E	Equipment Blank (Feed Soil)	Concentration	**<0.05 µg/L**[b]	<0.05 µg/L
HAR-2-2-1-0-E	Equipment Blank (Treated Soil)	Concentration	<0.05 µg/L	<0.05 µg/L
HAR-2-3-1-0-E	Equipment Blank (Dewatered Fines)	Concentration	**0.38 µg/L**[a]	<0.05 µg/L
HAR-2-4-1-0-E	Equipment Blank (Treated Fines)	Concentration	<0.05 µg/L	<0.05 µg/L
HAR-2-1-1-0-F	Field Blank (Feed Soil)	Concentration	<0.05 µg/L	<0.05 µg/L
HAR-2-2-1-0-F	Field Blank (Treated Soil)	Concentration	<0.05 µg/L	<0.05 µg/L
HAR-2-3-1-0-F	Field Blank (Dewatered Fines)	Concentration	<0.05 µg/L	<0.05 µg/L
HAR-2-4-1-0-F	Field Blank (Treated Fines)	Concentration	<0.05 µg/L	<0.05 µg/L

[a] Value outside QA objective

b Micrograms per Liter

- TCLP-leachable mercury was reduced from an average of 82 μg/L to 6 μg/L. The TCLP regulatory level for mercury is 200 μg/L and the U.S. federal maximum contaminant level (MCL) for mercury is 2 μg/L.

- The average mercury concentrations in the stack gas discharged to the atmosphere during the demonstration was 2.92 μg/NCM at an average discharge rate of 30,300 NCM/hr.

- Mercury concentrations in the treated process water discharged to the sewer during the three test runs were 4.39 μg/L, 5.80 μg/L, and 4.68 μg/L. These concentrations are close to the U.S. federal MCL for mercury of 2 μg/L.

- The mercury removal efficiencies achieved by the vacuum-distillation process for the three test runs were 98.7 percent, 98.4 percent, and 96.6 percent.

- The native moisture content of the contaminated soil from the CFM site ranged from 17.1 percent to 19.5 percent by weight. The moisture content of the dewatered soil from the soil washing process ranged from 19 percent to 37.4 percent by weight. The moisture content of the treated soil exiting the vacuum-distillation unit ranged from 0.00 to 0.03 percent by weight.

- Because all of the contaminated soil and construction debris was crushed to a fairly uniform size for this demonstration, the process did not significantly change the particle size distribution (break up coarser particles and create more fines) of the soils.

- The internal temperature of the soil dryer was 130°C during the demonstration.

- The internal temperature of the vacuum-distillation unit ranged from 430°C to 500°C during the demonstration.

- During the demonstration, the Harbauer system processed soil at rates ranging from 2.4 metric tons per hour (mt/hr) to 3.7 mt/hr. The quantities of soil treated during the three test runs were 29.6 mt (8 hours), 20 mt (8 hours), and 14.4 mt (6 hours).

- Treated process water was discharged to the sewer at rates ranging from 2.9 m^3/hr to 10.3 m^3/hr. Quantities of treated process water discharged during the three test runs were 43.2 m^3 (8 hours), 82.4 cubic meters (m^3) (8 hours), 17.4 m^3 (6 hours), respectively.

- The cost for treating mercury contaminated soil from the CFM site is 870 DM/mt ($580/mt assuming a 1.5 DM to $ exchange rate) based on an agreement between Harbauer and the client, Landkreis Wunsiedel. This cost covers half the cost of the facility (45,000,000 DM [$30,000,000]). Harbauer estimates that the cost for treating soil from other sites will be 480 DM/mt ($320/mt).

Chapter 3.0

Economic Analysis

This section presents cost estimates for using the Harbauer technology to treat soil contaminated with mercury. Cost estimates presented in this section are based on data provided by Harbauer GmbH & Co. KG.

3.1 Basis Of Economic Analysis

This section describes the factors that may affect the costs associated with the Harbauer treatment system. Although soil treatment costs were not independently estimated, the following cost categories (Evans 1990) should be considered when evaluating the potential cost of treating soil using the Harbauer technology:

- Site preparation
- Permitting and regulatory requirements
- Capital equipment
- Startup
- Labor
- Consumables and supplies
- Utilities
- Effluent treatment and disposal
- Residuals and waste shipping and handling
- Analytical services
- Maintenance and modifications
- Demobilization

3.2 Estimated Costs Of Applying The Technology

For the application of the Harbauer technology at the Marktredwitz site, the cost per metric ton is 870 DM ($580, assuming a 1.5 DM to $ exchange rate) for the client Landkreis Wunsiedel. This cost represents the capital costs factored in to the operation cost extrapolated over the life of the facility. Under a cost sharing agreement between Harbauer and Landkreis Wunsiedel, this price covers half of the investment costs of the facility (approximately 45,000,000 DM [$30,000,000]). Harbauer estimated the price without this cost sharing (that is, if the Landkreis Wunsiedel did not have to pay for half of the capital cost of the facility) to be 480 DM ($320) per metric ton.

Chapter 4.0

Technology Applications Analysis

This section evaluates the general applicability of the Harbauer technology to contaminated waste sites. Information presented in this section is intended to assist decision makers in screening specific technologies for a particular cleanup situation. This section presents the advantages, disadvantages, and limitations of the technology and discusses factors that have a major impact on the performance and cost of the technology. The analysis is based both on the demonstration results and on available information from other applications of the technology.

4.1 Feasibility Study Evaluation Criteria

This section assesses the Harbauer technology against the nine evaluation criteria used for conducting detailed analyses of remedial alternatives in feasibility studies under CERCLA (EPA 1988).

4.1.1 Overall Protection of Human Health and the Environment

The Harbauer technology provides both short-term and long-term protection to human health and the environment by reducing the concentrations of contaminants from soil and the leaching potential of the contaminants from the soil. The Harbauer technology removes mercury from soil by heating the soil to temperatures at which volatile and semivolatile contaminants partition from the solid (soil) phase to the vapor (air) phase. The contaminants are condensed and can be recycled. Treated process water is discharged to the municipal sewer system. Exposure from air emissions is minimized through the removal of contaminants from the system's air process stream using carbon adsorption units before discharge to the atmosphere.

4.1.2 Compliance with ARARs

Although general and specific applicable or relevant and appropriate requirements (ARARs) were not specifically identified for the Harbauer technology, compliance with chemical-, location-, and action-specific ARARs should be determined on a site-specific basis. While location- and action-specific ARARs generally can be met, compliance with chemical-specific ARARs depends on the efficiency of the Harbauer system to remove contaminants from the soil and the site-specific cleanup level. To meet chemical-specific ARARs, contaminated soil may require multiple passes through the treatment system.

4.1.3 Long-Term Effectiveness and Permanence

The Harbauer system permanently removes contaminants from the soil, and treatment residuals (activated carbon) are regenerated on-site. Treatment of dissolved contaminants in the process water and air emissions using carbon adsorption units are permanent solutions for the removal of contaminants. Both of these techniques are well-demonstrated and effectively remove mercury from process water and stack gas.

4.1.4 Reduction of Toxicity, Mobility, or Volume Through Treatment

The reduction of mercury concentrations in the soil is due to the removal of mercury via enhanced volatilization by vacuum distillation. The vapor-phase mercury is condensed to a liquid and recycled.

In addition to reducing contaminant concentrations in the soil, the Harbauer system reduces contaminant leaching potential based on the results of TCLP testing.

4.1.5 Short-Term Effectiveness

Potential short-term risks presented during system operation to workers, the community, and the environment include air emissions.

Because the technology removes mercury, vapor-phase activated carbon abatement controls must be provided for these emissions. Adverse impacts from the air stream are mitigated by passing the emissions through carbon adsorption units before discharge to the ambient air.

Implementation of the Harbauer system involves (1) site preparation, (2) facility construction, and (3) operation, monitoring, and maintenance. Minimal adverse impacts to the community, workers, or the environment are anticipated during site preparation and system installation. Additionally, exposure from fugitive air emissions during operation, monitoring, and maintenance are minimized through the removal of contaminants in the system's air process stream using carbon adsorption units before discharge.

4.1.6 Implementability

Site preparation and access requirements for the technology are significant. The site must be accessible to large trucks. The components of the Harbauer system require approximately 2,500 square meters. However, the equipment and materials that constitute this remedial alternative are commercially available. In addition, installation and operation of the Harbauer system are anticipated to involve few administrative difficulties. The treatment system can be operational within 80 days if all necessary equipment, utilities, and supplies are available. Operation and monitoring can be performed by trained engineers and technicians. Other services and supplies required to implement the system could include a carbon adsorption/regeneration/disposal unit, laboratory analysis to monitor system performance, and electrical utilities.

4.1.7 Cost

For the application of the Harbauer technology at the Marktredwitz site, the cost per metric ton is 870 DM ($580, assuming a 1.5 DM to $ exchange rate) for the client Landkreis Wunsiedel. Based on a cost sharing agreement between Harbauer and Landkreis Wunsiedel, this price covers half of the investment costs of the facility (approximately 45,000,000 DM [$30,000,000]). Harbauer estimated the price without this cost sharing (that is, if the Landkreis Wunsiedel did not have to pay for half of the capital cost of the facility) to be 480 DM ($320) per metric ton.

4.1.8 State Acceptance

State acceptance is anticipated because the Harbauer system uses well-documented and widely accepted processes to remove mercury from soil and to treat the process water and stack gas emissions. If remediation is conducted as part of Resource Conservation and Recovery Act (RCRA) corrective actions, state regulatory agencies will require that permits be obtained before implementing the system, such as a permit to operate the treatment system, an air emissions permit, and a permit to store contaminated soil for greater than 90 days if these items are considered hazardous wastes.

4.1.9 Community Acceptance

The system's size and space requirements as well as the principle of operation (volatilization of mercury) may raise concern in nearby communities. However, proper management and operational controls coupled with minimal short-term risks to the community and the permanent removal of contaminants through in situ processes make this technology likely to be accepted by the public.

4.2 Applicable Wastes

The Harbauer technology demonstrated at Marktredwitz, Germany, was designed to remove mercury from soil. The developer claims that the technology can also remove other contaminants, such as volatile and semivolatile organic compounds as well as certain other metals. However, the technology's applicability to volatile and semivolatile organic compounds and metals other than mercury has yet to be proven.

4.3 Limitations Of The Technology

The developer claims that the only limitation associated with the Harbauer technology is that the contaminants must volatilize under reduced pressure and elevated temperature. The developer claims that there are no concentration limits on the contaminated media that can be treated by the system. However, high concentrations of contaminants may require more than one pass through the system or increased system residence times to achieve remediation goals.

Chapter 5.0

Harbauer Technology Status

The Harbauer technology is patented both in Europe under European (patent no. EP0388387) and in the U.S. According to Harbauer, the system can be used for remediation of contaminated soils, especially those contaminated with volatile and semivolatile organic compounds or heavy metals. The Harbauer technology combines chemical and physical processes for the treatment of adsorbed, dissolved, and free phase contaminants in soil. Since its inception in 1987, the Harbauer technology has been applied at only one site in Europe - the Marktredwitz site. Harbauer is represented in the U.S. by Lockwood Greene Engineers, Inc. There are currently no commercially operating systems in the U.S.

6.0 References

American Society for Testing and Materials (ASTM). 1990. Water Content of Soil/Rock/Soil-Aggregate Mixtures, D2216.

Evans, G. 1990. "Estimating Innovative Technology Costs for the SITE Program." Journal of Air and Waste Management Assessment. Volume 40, Number 7. July.

PRC Environmental Management, Inc. (PRC) 1994. Quality Assurance Project Plan for the Harbauer Soil Washing/Vacuum Distillation System Demonstration at the Chemische Fabrik Marktredwitz Site, Marktredwitz, Germany. November.

PRC. 1995. Amendment to the Quality Assurance Project Plan for the Harbauer Soil Washing/Vacuum-Distillation System Demonstration at the Chemische Fabrik Marktredwitz Site, Marktredwitz, Germany. March.

U.S. Environmental Protection Agency (EPA). 1987. Test Methods for Evaluating Solid Waste, Volumes IA-IC: Laboratory Manual, Physical/Chemical Methods; and Volume II: Field Manual, Physical/Chemical Methods, SW-846, Third Edition, (revision 0), Office of Solid Waste and Emergency Response, Washington, D.C.

EPA. 1988. "Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA." EPA/540/G-89/004. October.

Appendix A

Developer Case Studies

Further Optimization of the Harbauer Soil Washing/Vacuum Distillation System During the Clean-up Action in Marktredwitz/Germany from Fall 1993 until Summer 1996

1.0 INTRODUCTION

The Harbauer soil treatment facility is the first full-scale application of a combined vacuum distillation and soil washing process. The facility in Marktredwitz was designed according to the specific requirements of the remedial action on the former "Chemische Fabrik Marktredwitz (CFM)" site in Marktredwitz, Germany.

For this project the following conditions were specified:

(1) Clean-up criteria (for landfilling of the treated soil):

- 50 milligrams per kilogram (mg/kg) dry weight residual mercury concentration
- 1 milligrams per liter (mg/l) residual arsenic concentration in the elutriate
- 1 mg/l residual antimony concentration in the elutriate

(2) Reduction of emissions during operation of the facility:

- Threshold value for waste water release into the municipal sewer: 10 μg mercury per liter
- Threshold value for air emissions into the atmosphere: 50 μg mercury per m^3 of off gases

(3) Treatment of a broad variety of soil types and building debris

(4) Treatment of a wide range of contaminant concentration levels

(5) Recycling of condensed mercury

(6) Yearly throughput capacity: 35,000 metric tons

Although the initial startup of the soil washing and vacuum distillation system encountered minor difficulties, the problems were appropriately addressed, and all requirements were met during routine operation of the facility. In particular, the emission criteria that were used to subject the system to a strict monitoring program demonstrated that the emissions were successfully below the threshold values. The results of the Superfund Innovative Technology Evaluation (SITE) Technology Demonstration are documented in this report.

To further determine fields of application for the technology and limitations of the process, Harbauer has performed additional testing. The main areas of testing were:

(1) Determination of the maximum clean-up performance of the technology

(2) Testing of application for other contaminants

(3) Testing of the range of treatable soil types

The results of the tests are documented briefly in the following sections.

2.0 MAXIMUM CLEANUP PERFORMANCE

The cleanup criterion of 50 milligrams mercury per dry kilogram of soil was specified by the authorities for the CFM-site, because the soil was designated to be landfilled in a monofill; it was not required to be used as backfill or reuse purposes. In the future, it can be expected that other remedial actions will be required to incoporate reuse of the treated soil in the treatment technology. In this case, a residual contaminant concentration of 50 mg mercury per kg of soil can be assumed to be too high.

Optimization of plant operation during test runs has shown that the technology allows for substantially lower residual contaminant concentrations than expected, as shown in Figures 1 through 4.

Figure 1 shows treatment results achieved during the first phase of plant operation after start-up of the system in Marktredwitz (September 1993 through October 1994). Initially, the average residual mercury concentration in the treated soil exiting the facility was 23 mg/kg dry weight. As plant and operation optimization were realized, the average residual concentration dropped to about 15 mg/kg as shown in Figure 2 (November 1994 through October 1995). The diagram in Figure 3 shows the residual concentrations in the latest phase of operation (November 1995 through February 1996). The average residual concentrations had dropped to 7.9 mg mercury per kg of soil dry weight by this time.

Figure 4 shows the average residual contaminant concentration of the soil stream exiting the vacuum distillation process of the facility, which is 3.6 mg/kg dry weight (October 1995 through April 1996). These results indicate that residual mercury concentrations substantially lower than 50 mg of mercury per kg of soil can be achieved using the soil treatment technology at the Harbauer facility.

Following optimization of the plant operation, the soil exiting the vacuum distillation process is contains significantly less than 10 mg of mercury per kg. Results from monitoring plant operation over an extended time period and from the SITE demonstration also indicate that the successful treatment results were achieved with very high feed soil contaminant concentration levels (Figures 5 and 6).

Trial runs have been performed with mercury contaminated soil from another remedial site, at which a cleanup criterion of 2 mg per kg was specified. The results show that this stringent requirement can be reliably met if the plant configuration is properly adapted.

3.0 APPLICATION TO OTHER CONTAMINANTS

During the system operation, it was tested whether contaminants other than mercury can be effectively removed from contaminated soil with the Harbauer system. In order to be considered for treatability by this system, the contaminants must be volatile within the temperature (between 350° and 450° Celsius) and pressure ranges (50 to 150 hPa) of the system. Results of trial runs performed using a small scale pilot plant, serving as a model for the full-scale system, are documented in Table 1.

Table 1: Results of trial runs using a small scale pilot plant

Compound(s)	**Feed Soil Concentration (mg/kg)[1]**	**Treated Soil Concentration (mg/kg)**
PAHs[2] (sum of 16)	32,000	< 1[5]
Hydrocarbons[3]	102,000	< 10
Cyanides (total)	1,728	< 10
Mercury	8,000	0.5 - 4
Lindane	3,400	0.002
TNT[4]	100,000	0.4

[1] mg/kg: milligrams per kilogram of soil

[2] Polycyclic aromatic hydrocarbons
[3] Analyzed according to DIN standard method 38409-H18 which is similar to MCAWW-Method 418.1 for analysis of Total Recoverable Hydrocarbons (TRPH)
[4] Trinitrotoluene
[5] Lower than 1

4.0 RANGE OF TREATABLE SOIL TYPES

Routine operation of the Harbauer system for more than 2.5 years has shown that the vacuum distillation process is very insensitive to variations in the feed soil type. The process has been operated using a wide range of feed soil grain sizes, with no observed effect.

In addition to sandy soils, the soils that can be treated effectively and efficiently include the following:

- Very fine grained soil with silt- and clay portions of more than 90 % (with a smaller throughput capacity)
- Gravels
- Slags
- Highly contaminated fine residuals from soil washing processes
- Concrete and brick debris
- Other solid materials with mechanical characteristics that are similar to soil

5.0 CONCLUSIONS

Harbauer´s soil washing and vacuum distillation processes operated together or separately can be used to treat contaminated soils effectively and efficiently. Technology selection and plant configuration is based upon the requirements of the particular cleanup project. The following characteristics of the two processes and the primary fields of application are listed:

Soil washing:

- Suitable for soils which are loaded with organic and inorganic contaminations
- High throughput capacity, short treatment time and possibility for immediate re-use of treated materials

- Wet-mechanical process with low impact on the environment
- Low energy consumption (and cost)
- Treated soil can be recultivated
- High cost-efficiency, especially when large amounts of soil are to be treated

Vacuum distillation:

- High clean-up efficiency; efficiency not affected by the feed soil contaminant concentration levels and soil structure
- Small volumes of process-gases
- Oxygen-reduced process; avoidance of chemical reactions by inert gas flushing is optional
- Leakages do not cause any emissions of process-gases due to vacuum conditions
- Vacuum conditions lower the evaporation temperatures; saves energy and lower costs
- Treated soil can be recultivated
- The amount of process-residuals is extremely low (recycling of condensed contaminants)

Harbauer has proven that a combination of these technologies is economically advantageous. If stringent cleanup criteria cannot be met with a single process due to extremely high contaminant concentrations or a wide variety of contaminants present on the site, the treatment sequence can be implemented.

Figure 1

Harbauer treatment results achieved during the first phase of plant operation from September 1993 through October 1994

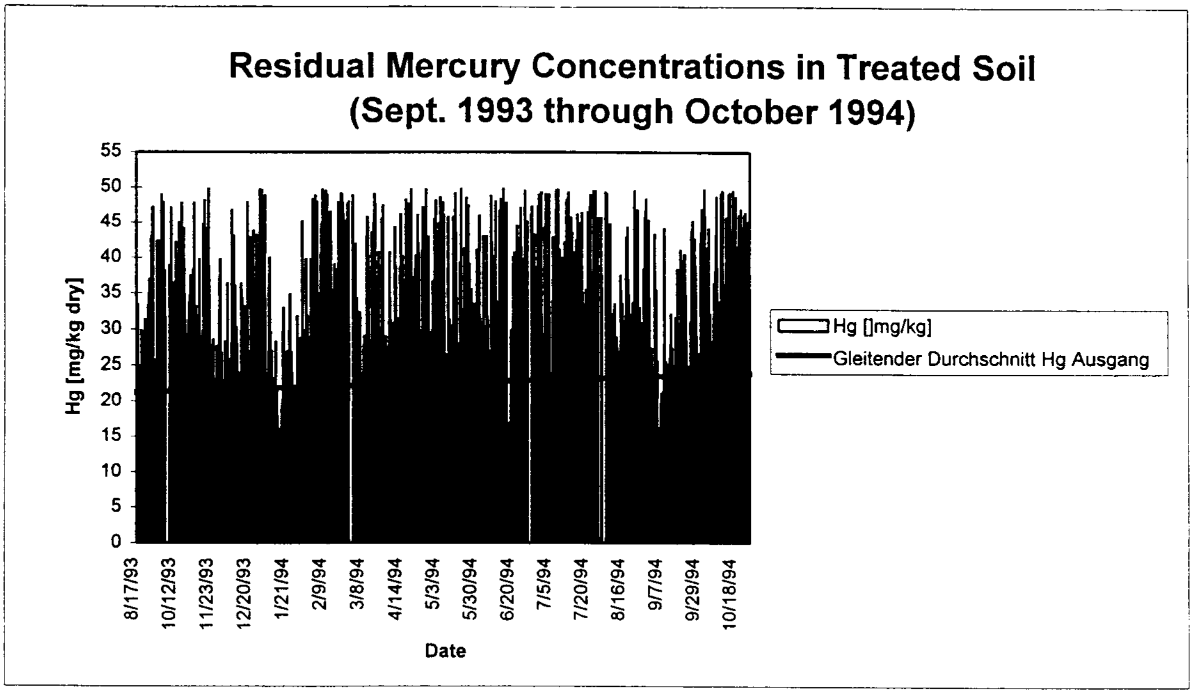


Figure 2

Harbauer data on residual concentrations in the operation phase
November 1994 through October 1995

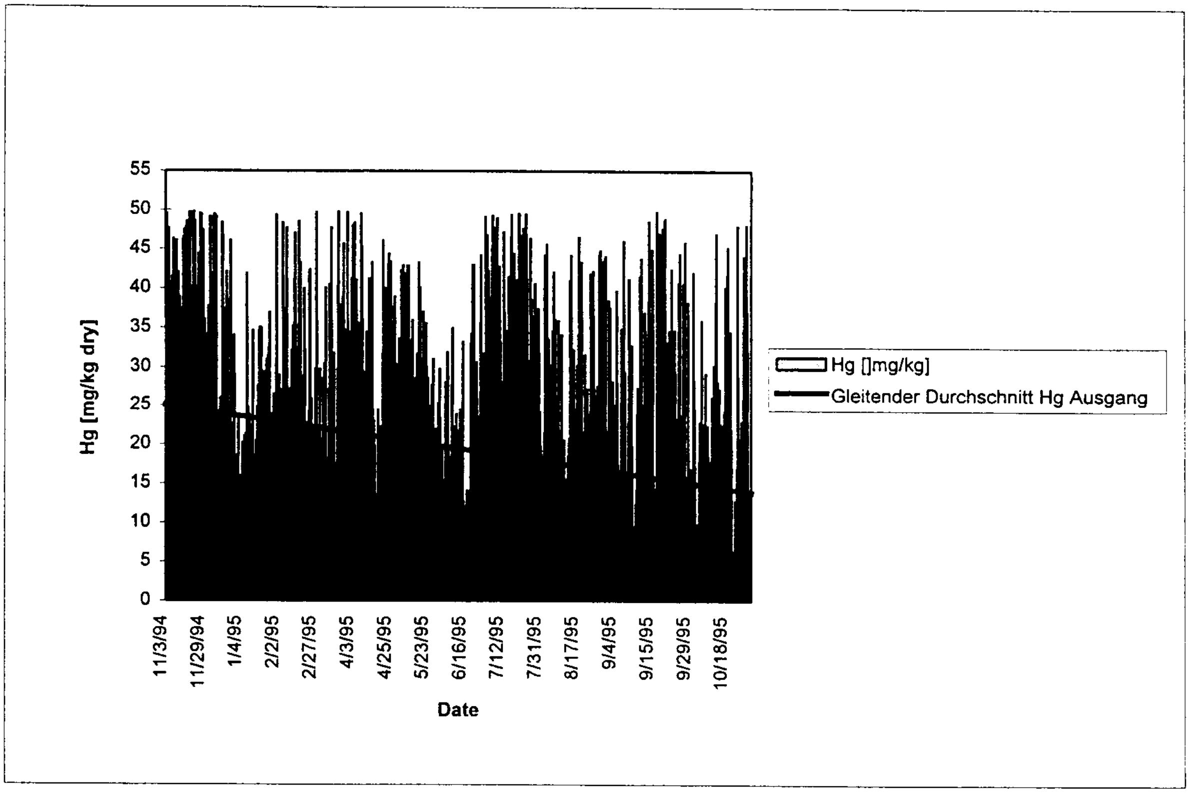


Figure 3

Harbauer data on residual concentrations in the operation phase
November 1995 through February 1996

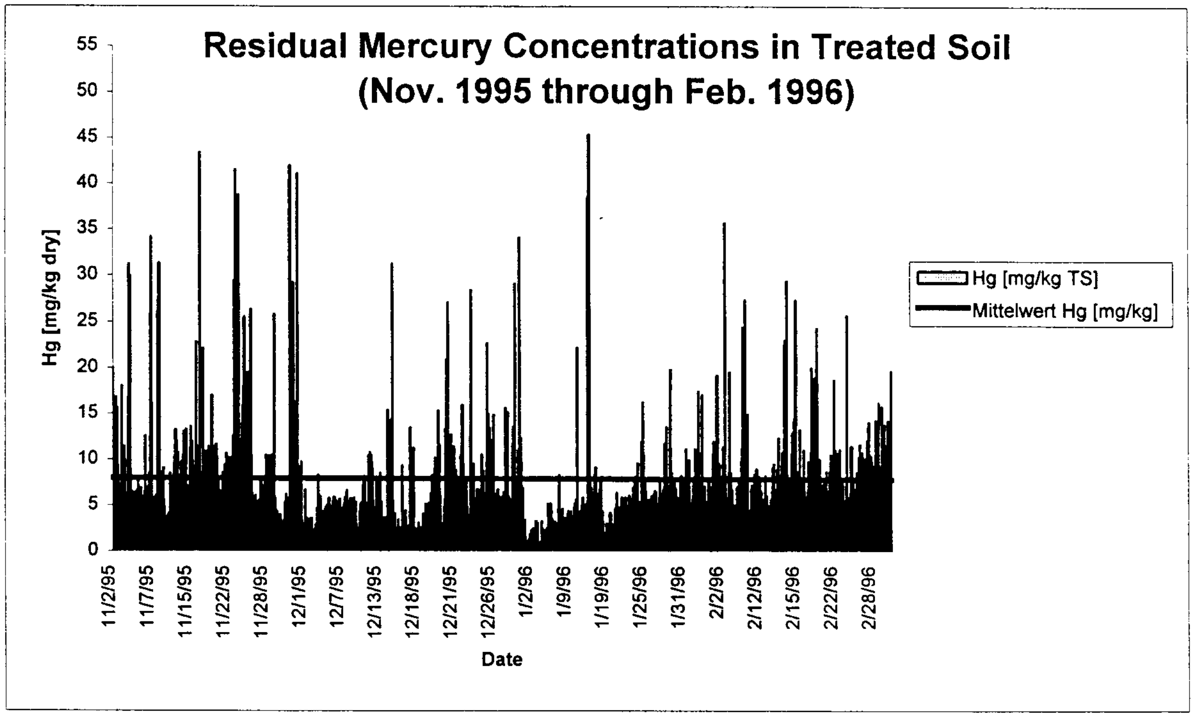


Figure 4

Harbauer data on residual contaminant concentrations of the soil stream exiting the vacuum distillation process
October 1995 through April 1996

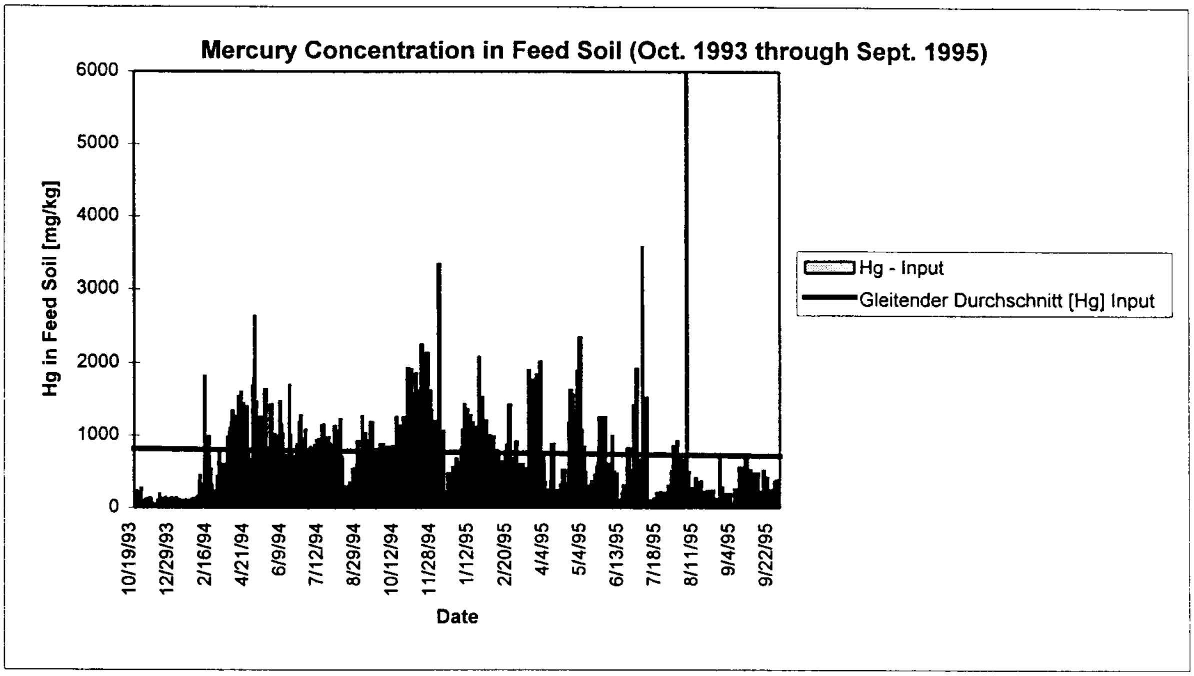


Figure 5

Feed soil mercury concentration levels
October 1993 through September 1995

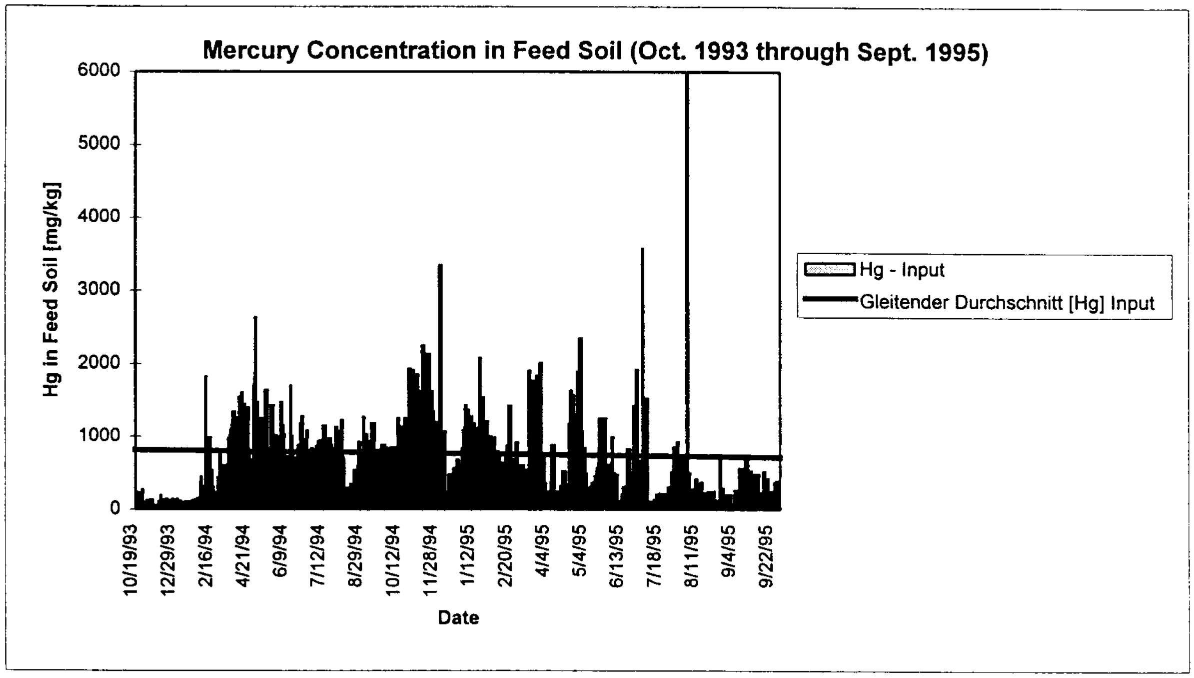
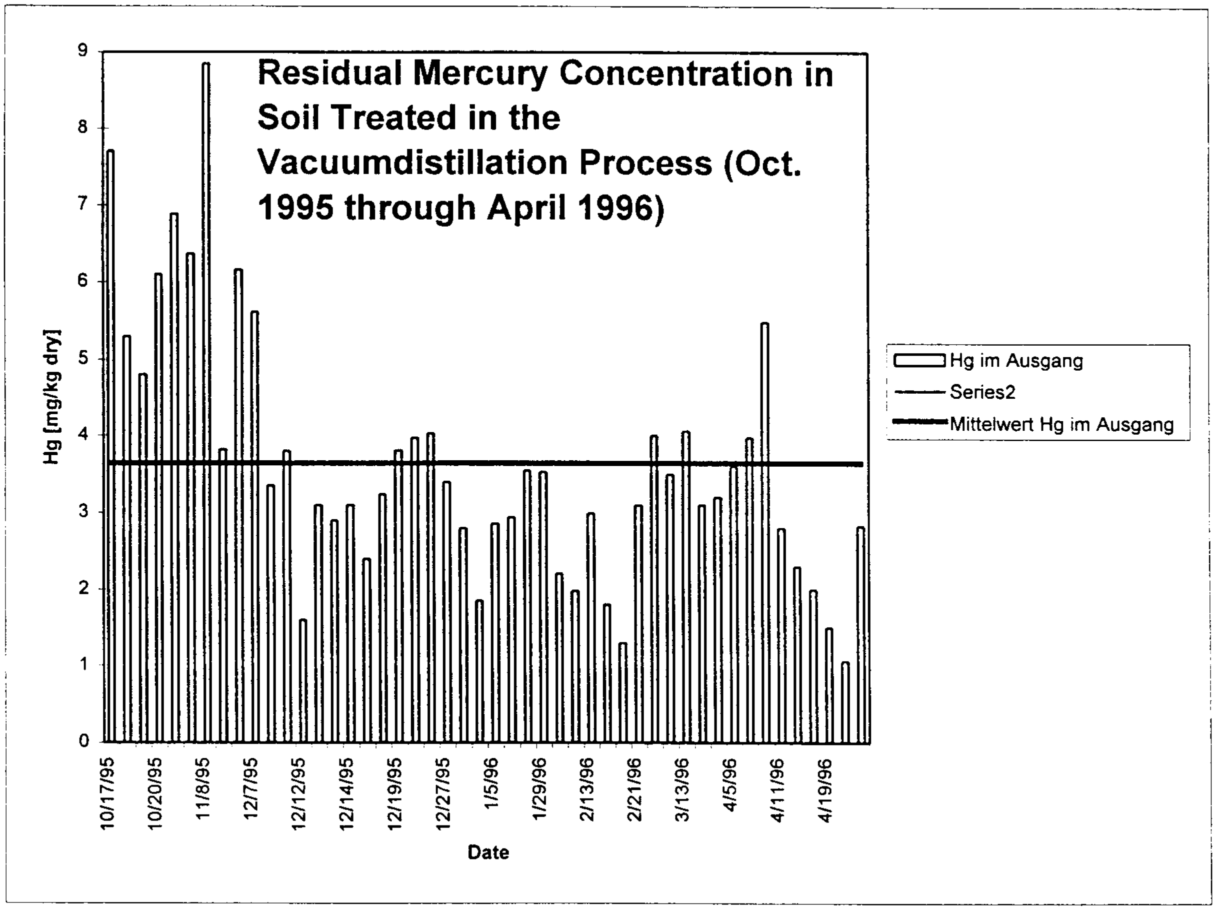


Figure 6

4/30/96

Feed soil mercury concentration levels
October 1995 through September 1996



Appendix B

Observations And Recommendations

Implementation Of US EPA Sampling And Analysis Methods For The Harbauer Soil Washing/Vacuum Distillation Technology Demonstration

OBSERVATIONS AND RECOMMENDATIONS

IMPLEMENTATION OF USEPA SAMPLING AND ANALYSIS METHODS FOR THE HARBAUER SOIL WASHING/VACUUM DISTILLATION TECHNOLOGY DEMONSTRATION

Conducted for the US-German Hazardous Waste
Treatment Technology Bilateral Agreement

Conducted at Harbauer Plant Site , Wölsau, Bavaria
Institut Fresenius, Taunusstein-Neuhof, Hessen
Federal Republic of Germany

QA ID No: 926
EPA TPM: Don Sanning

January 26, 1996

I. INTRODUCTION

During the weeks of 21 November and 28 November 1994, observations were conducted of the sampling and analytical operations associated with the Harbauer Soil Washing/Vacuum Distillation technology demonstration. This technology demonstration is part of the Germany-United States Bilateral Agreement for the demonstration of hazardous waste treatment technologies and is a Category 2 USEPA-NRMRL-Ci project. Harbauer demonstrated this technology at their facility in Wölsau, Bavaria, Federal Republic of Germany. Mr. W. Burton Blackburn, a sub-contractor to S-Cubed, provided quality assurance (QA) support for the USEPA in observing and providing recommendations concerning the subject sampling and analytical operations. This QA support focuses on one of the objectives of the Bilateral Agreement, namely to facilitate information transfer between Germany and the United States regarding QA procedures. The on-site QA support also provided a mechanism for providing QA and technical information to the field and laboratory staff so that they could become more familiar with the requirements of the USEPA sampling and analysis methods and implement them effectively.

The Institut Fresenius, Taunusstein-Neuhof, Hessen, Germany, provided the sampling and analytical support for this project. Dr. R. Kurz was the primary representative for Institut Fresenius. Institut Fresenius performed these activities under a subcontract to Arge focon-Probiotec, the German prime contractor for the Bilateral Agreement. Mr. Kai Steffens represented focon-Probiotec during sampling operations. Sampling and analytical operations are described in the QA Project Plan (QAPP) for this project. To ensure comparability and applicability with US standards, the QAPP specifies USEPA methods for all sampling and analytical operations. For this project, the QAPP specifies mercury concentrations in gas, solids, water and Toxicity Characteristic Leaching Procedure (TCLP) leachates as the critical measurements.

PRC Environmental Management, Inc. (PRC-EMI) provided on-site engineering support for the USEPA. Mr. Roger Argus, PRC-EMI San Diego, and Ms. Sara Woodland, PRC-EMI Germany provided this support during the field demonstration activities.

In March of 1993 and January of 1994, Mr. Blackburn visited Institut Fresenius to observe their implementation of USEPA sampling and analysis methods required for Bilateral Agreement projects. Mercury determination procedures were observed during each of those visits and recommendations were provided to support Institut Fresenius in preparing for analyzing samples using SW-846 Method 7470 and 7471. This report presents observations and recommendations for the critical sampling and analysis operations associated with the Harbauer technology demonstration project. Newly implemented quality assurance procedures in the trace metals laboratory were noted in the January 1994 review and these procedures were used during the subject project.

On arriving at the Harbauer site on 22 November 1994, the reviewer learned of some modifications to the process that Harbauer deemed necessary for continuing operations. Most significantly, the presence of building material rubble and a mixer breakdown required all waste materials to be subjected to vacuum distillation. This change eliminated collection of certain samples. The changes to the sampling locations noted in the QAPP will be documented in a memo from PRC.

II. PROJECT ORGANIZATION AND QUALITY ASSURANCE MANAGEMENT

Both the field and laboratory activities were well-organized and properly staffed such that project objectives and timetables could be met. The field staff were fully cognizant of the requirements for solid and liquid sampling, but communications and readiness problems existed for the gas sampling operations. This situation is discussed in Observation 1 below.

The reviewer also noted some readiness problems with the TCLP procedure at Institut Fresenius. This is discussed in Observation 2 below.

Observations and Recommendations:

(1) The stack gas sampling staff was not prepared or equipped for performing the sampling operations required in the QAPP. The basic equipment and procedures were present for collecting the mercury samples according to Method 101A; however, equipment and preparation was not present for performing USEPA Methods 1 through 4. Methods 1 through 4 are necessary to obtain accurate velocity measurements so that isokinetic sampling can be performed and total sampling volume can be calculated. Also, the sampling equipment did not include the pitot tube and manometer so that isokinetics and gas flow could be measured during sampling according to USEPA Method 5. The Institut Fresenius sampling staff was prepared to use an anemometer to measure gas velocity, but the anemometer had not been recently checked for calibration, nor does this procedure satisfy the requirements of the methods specified in the QAPP. The problem was further complicated by the fact that the Institut Fresenius gas sampling staff did not possess copies of Methods 1 through 4 and were unaware of those requirements.

It should be noted that NRMRL-Ci conducted a readiness review for stack sampling methods in March of 1993 with Dr. Arnold of the Institut Fresenius gas sampling staff. Dr. Arnold indicated at that time that his staff was fully equipped and prepared to perform USEPA Methods 1 through 5. Dr. Arnold also indicated that he was experienced in the USEPA methods since he had used them in an Insitut Fresenius project in India. Unfortunately, Dr. Arnold was not present during the Harbauer field sampling event to direct the sampling operations.

Since the Insitut Fresenius gas sampling staff was not prepared or equipped to perform the required stack gas sampling operations, the decision was made to postpone the USEPA mercury gas sampling methods. This is further discussed in Section III below. Given the cancellation of these operations, recommendations were made to improve QA management and communication procedures between the various parties concerned with the stack gas sampling procedures. It was recommended that Arge. focon-Probiotec coordinate with the Institut Fresenius to develop and document procedures for the early communication of QAPP requirements to all pertinent technical staff. Further, internal QA management procedures could be developed to allow for internal audits and readiness reviews well in advance of the proposed demonstration date. Such a readiness review would help ensure that the technical staff is familiar with the required methods and proper equipment is available to perform the required sampling operations.

Given the time requirements associated with preparation, communication, and QA management, Institut Fresenius may wish to make more time and authority available to the QA specialist responsible for the project. QAPP Section 2.6 states that the laboratory QA manager, Mr. Bonk-Kassner, would be responsible for reviewing field sampling operations and ensuring that "QAPP protocol is adhered to and any deviations documented," as well as conducting internal audits of the laboratory. If more time is allowed to actually implement all these responsibilities detailed in the QAPP, problems similar to those encountered in this project would likely be prevented.

(2) The laboratory at Insitut Fresenius was not fully prepared to perform the TCLP method. The TCLP is required as part of the critical measurements for this project. Section IV below discusses specific technical observations relating to the TCLP at Institut Fresenius. As samples arrived in the laboratory, Institut Fresenius had not practiced the TCLP method and certain necessary equipment and documentation necessary for the TCLP was not present. Further, the laboratory did not have a copy of SW-846 Method 1311–the TCLP reference required by the QAPP. The reviewer provided Institut Fresenius with a copy of Method 1311. The laboratory did possess a copy of the current Code of Federal Regulations (CFR) reference for TCLP. Fortunately, this reference is essentially identical to Method 1311.

Since it is important to be fully equipped and prepared for a method before samples begin arriving at the laboratory, Institut Fresenius may wish to consider fully implementing new USEPA methods and performing proficiency tests before sampling begins. USEPA QA and technical staff can be consulted with questions regarding complicated new procedures such as TCLP. Given more advance preparation , the laboratory and staff will be more efficient and samples will be more likely to be processed within the specified holding times. Institut Fresenius may also wish to consider internal audits or readiness reviews of new USEPA methods that will be required for upcoming Bilateral Agreement projects.

III. FIELD SAMPLING OPERATIONS AND FIELD MEASUREMENTS

The first day of sampling operations was underway on 22 November as the reviewer arrived on-site. A quick review of stack sampling operations revealed that the stack sampling crew was not prepared or equipped to perform the mercury stack sampling procedures according to the requirements given in the QAPP. After consultation with USEPA, focon-Probiotec directed that stack sampling should proceed for one test run according to German protocol. The reference for this method is *Verein Deutscher Ingenieure (VDI) 3868* entitled *Bestimmung der Gasamtemission von Metallen, Halbmetallen, und ihren Verbindungen Messen von Quecksilber Atomabsorptions-spektrometrie mit Kaltdampftechnik (März 1992)*. This method describes both sampling and analysis procedures. A copy is included in Appendix B of this report. If sampling equipment can be procured, stack gas sampling according to the USEPA (Method 101A) method will be performed at a later date. An abbreviated solids sampling program will be included if that sampling event occurs.

Except for the necessary and documented changes to the solids and liquids sampling program discussed in Section I above, the Institute Fresenius staff collected all samples according to the QAPP. After collection, compositing and zinc amalgamation for the appropriate samples were performed according to QAPP requirements. Field and equipment blanks were properly collected at appropriate frequencies.

Documentation was complete and well organized. A field logbook was used to record sampling observations. Pre-printed forms, including field tracking forms, were also used to record sample and sample container information. Chain-of-custody forms were completed for all samples. Sample containers were properly labeled and custody sealed. Sampling staff transported the samples under appropriate conditions to the Institut Fresenius laboratory in Taunusstein.

All process measurements were collected according to the QAPP, with one improvement. On arriving at the site, the sampling staff learned that discharged treated process water had been metered and could be measured directly. The QAPP specified that discharged water would be assumed to be equivalent to the volume of make-up water drawn from the municipal water supply. This change to directly measuring the discharge water will result in more accurate data.

The reviewer was informed that scale calibration records are held by the government and were not available at the site for inspection. A government certification label on the scale indicated that it was certified until 1996 for calibration. The scale had been repaired and recalibrated two months before the demonstration.

Moisture determinations were performed on-site using a Sartorius MA-30 analyzer. Institut Fresenius performed the moisture measurements according to QAPP specified QC requirements, although balance calibration weights were not present to confirm balance accuracy. This is discussed in Observation 1 below.

Two observations and recommendations were made pertaining to the field activities for this project.

Observations and Recommendations:

(1) No standard calibration weights were present at the site to check the accuracy of the balance used for the moisture determinations. Although this is a non-critical measurement, Institut Fresenius agreed that the balance should be checked for accuracy. Dr. Kurz arranged for overnight delivery of calibration weights from the laboratory in Taunusstein. A calibration check using these weights indicated that the balance was acceptably accurate for the range of masses encountered with the moisture determinations.

(2) Some chain-of-custody records contained corrections that were made by writing over the incorrect entry. Proper documentation practice requires that a single line should be drawn through the incorrect entry, then the correct entry should be written beside the previous entry. Corrections should also be initialled by the responsible technician.

IV. SAMPLE LOG-IN AND CUSTODY

Samples for the Harbauer project were received at Institut Fresenius and logged in to the laboratory tracking system. This system allows for custody tracking of samples through the laboratory using a bar code system. Chain-of-custody forms received from the field were signed and completed by the sample custodian, although not immediately on receipt at the laboratory. The samples were delivered by Dr. Kurz to the laboratory and immediately stored in an appropriate, secured storage area. Sample condition and temperature were later documented by the sample custodian. Samples were stored in appropriate refrigerators. Refrigerator temperatures were properly maintained, and temperatures were checked and recorded daily.

All QAPP specifications for sample log-in and custody were implemented at Institut Fresenius. The reviewer made one recommendation.

Observation and Recommendation:

> Although samples were properly transported to the laboratory and promptly stored under appropriate conditions, the chain-of-custody forms were not completed to indicate the time when samples were received at the laboratory. The samples were delivered to the laboratory after normal business hours, so the sample custodian was not available to perform a complete sample check-in. Under such circumstances, it was recommended that the staff delivering samples to the laboratory should record the time that samples are checked into the laboratory. QAPP Section 4.5.3 specifies this requirement.

V. LABORATORY PROCEDURES

At the time of the laboratory visit, the laboratory was in the process of preparing samples for the Harbauer project. No field sample analyses had begun.

As discussed earlier, Institut Fresenius had never performed the TCLP and was not fully equipped to perform the procedure. The reviewer assisted with reviewing the procedure with the laboratory staff and provided recommendations. These are discussed in Observation 1 below.

Dr. A. Brockmann, Inorganic Laboratory Manager at Institut Fresenius, and responsible laboratory staff were consulted regarding SW-846 Method 7470 and 7471. The reviewer observed that Dr. Brockmann and his staff were fully familiar and prepared for these methods. The corresponding German mercury determination methods vary only slightly from the USEPA methods. As previously discussed and approved by the EPA-NRMRL-Ci QA office, Institut Fresenius will use an automated, continuous flow mercury analyzer, rather than the manual, single injection system described in the SW-846 methods. The mercury analyzer at Institut Fresenius operated using the same atomic absorption principles, including the analytical wavelength, as do Methods 7470 and 7471. Given the high concentrations present in the untreated samples at Harbauer, the laboratory intends to vary the starting sample sizes to allow for analytical concentrations to fall within the range of the analyzer.

Documentation for the mercury sample digestion and analysis procedures was examined and found to be complete and well organized. Calibration standards and QC solution preparation documentation was complete and allowed for traceability. Calibration standards are checked against a second source to verify accuracy. From observations made at Institut Fresenius, the Method 7470/7471 mercury determinations are likely to yield data that are of known and adequate quality to meet project requirements.

One recommendation was made for TCLP and one for the mercury determinations. These are discussed below.

<u>Observations and Recommendations</u>:

(1) The laboratory was not completely prepared for the TCLP method. The reviewer observed that the laboratory was not prepared to use a gas regulator that would allow for measurement of pressures so that the required step-wise pressure filtration would be accomplished. Also, the laboratory did not possess the proper filter media as required by the method. The laboratory room used for the TCLP extractions did not have temperature monitoring equipment in place to verify that the extractions occurred at 23 ±2°C as required by Method 1311. Finally, TCLP documentation forms had not yet been prepared.

Institut Fresenius indicated that an appropriate pressure regulator could be located elsewhere at the Institut and would be installed for the TCLP. The proper filtration media were located from a supplier that assured delivery within 2 days. The reviewer noted that Institut Fresenius promptly obtained a recording thermometer to monitor room temperatures during the TCLP extraction periods. The reviewer arranged to have example TCLP documentation forms telefaxed to Institut Fresenius to allow for preparation of appropriate forms. The example forms arrived before the reviewer concluded his visit. Assuming the filter media and pressure regulator arrived within the week, it is anticipated that no significant delay will occur that could risk completion of the TCLP within the 28 day holding time prescribed by Method 1311.

The reviewer also recommended that the responsible analyst complete several test trials of the TCLP to ensure proficiency before starting on the project samples. Dr. Kurz agreed that this would be promptly completed.

(2) Institut Fresenius indicated that the last method detection limit (MDL) study for mercury determination was performed over one year ago. Recognizing the need in this project for known and accurate detection limits, the reviewer recommended that a new MDL study should be performed using Method 7470/7471. Dr. Brockman

agreed and indicated that he would start on an MDL study using SW-846 protocol. This study would be completed before the Harbauer project sample analyses. The reviewer observed laboratory data that indicated very good instrument sensitivity, and it is likely that the MDL will yield target reporting limits much lower than those specified in Table 5 (QA Objectives for Critical Parameters) in the QAPP.

VI. CONCLUSION

As discussed earlier, an apparent lack of communication and preparation resulted in the loss of data for mercury concentrations in gas. An alternate plan was devised, but the original plan specified in the QAPP will not be accomplished. Project participants have discussed reducing the status of these data from primary critical measurement to secondary critical measurements. The reviewer suggested that focon-Probiotec continue to work with Insitut Fresenius to implement a QA management program for the Bilateral Agreement that includes documented communication procedures and internal audits or readiness reviews before beginning field activities. Once fully implemented, this QA program should help avoid problems involved in applying USEPA methods in Germany, such as the problem identified above.

Other field sampling activities seemed likely to produce representative samples that will meet project objectives. Assuming effective preparation is completed for the TCLP, laboratory activities should produce data that are of known and adequate quality to meet project objectives.

At the conclusion of the field and laboratory visits, a closing meeting was held with management, sampling and laboratory staff. Staff from Institut Fresenius, including Dr. Kurz attended the field sampling meeting. Also present were Mr. Steffens, Mr. Argus, and Ms. Woodland. The closing meeting at Institut Fresenius included Dr. Kurz and other pertinent laboratory staff. Preliminary observations and recommendations were discussed at each meeting. The reviewer prepared CAR Statements for both the field and laboratory reviews. These were distributed to meeting participants and telefaxed to the NRMRL-Ci QA staff in Cincinnati. Copies of the CAR Statements are included in Appendix A.

CORRECTIVE ACTION RECOMENDATION (CAR) STATEMENT

QA ID NO.: SC-926 Project Category: II

Project Title: Germany-USA Bilateral Agreement Demonstration: Harbauer Soil Washing/Vacuum Distillation System Demonstration

Organization(s) Reviewed: Institut Fresenius

On-Site Location: Wölsau, Bavaria, Federal Republic of Germany

☐ Sampling Review ☐ Analytical Review ☒ Sampling & Analytical Review

Methods/Compound Group(s) Reviewed: (1) Solid and liquid sampling procedures; (2) Hg in stack gas using EPA Method 101A; (3) Moisture content in solids

Reviewer and Affiliation: W. B. Blackburn, Southwest Research Associates

Review Date: 21-24 November 1994

RREL Technical Manager: D. Sanning

(Note:The interviewed organization(s) should realize that while the reviewers may be able to discuss the more obvious concerns on the spot, others may not become apparent until they have had a chance to review their notes, in detail, after returning to their office. For this reason, the reviewer's debreifing may not be as comprehensive as the written report.)

I. The reviewer should check the appropriate box or boxes:

a. ☐ No concerns were found during the review.

b. ☐ Minor concerns exist, but they are not anticipated to affect data quality.

c. ☐ Concerns exist that would or could have affected data quality; however, immediate on-site response to recommended corrective action prevented the concerns from significantly impacting data quality.

d.* ☐ Concerns exist that would or could affect data quality; however, impact on data quality may be minimized if immediate and proper corrective action is implimented.

e.* ☒ Concerns exist that have affected data quality.

f.* ☐ Other.

* Parts II and III of this form (next page) are to be completed by the reviewer and the EPA technical project manager, respectively, if boxes 'd', 'e', or 'f' are checked.

Project findings have been discussed with the on-site representative(s) of the interviewed organization(s):

Principle On-Site Project Manager (signature)

[illegible] STEFFENS
Print

11/25/94
Date

RREL (CAR)
EPA-407 (Cin 7/92)

CORRECTIVE ACTION RECOMMENDATION (CAR) STATEMENT
(Continued)

QA ID No.: SC-926 Project Category: II

Project Title: Germany-USA Bilateral Agreement Demonstration: Harbauer Soil Washing/Vacuum Distillation System Demonstration

II. To be completed by the reviewer if boxes 'd', 'e', or 'f' are checked under Part I.

Nature of Concerns:

(1) Incomplete communication of procedures regarding Hg stack sampling requirements to sampling staff resulting in postponement of critical stack gas sampling procedure.

(2) No standard weights available to check balance calibration for moisture measurements.

(3) Incorrect documentation procedure for correcting data in chain-of-custody forms.

Nature of Concern(s) continued on next page: ☒ No ☐ Yes

Recommended Action:

(1) Develop and document procedures for communication of QAPP requirements to pertinent technical staff to ensure staff and equipment readiness and implementation of required procedures.

(2) Obtain standard weights, check and document moisture balance calibration.

(3) Use single line through corrected data and initial corrections.

Recommended Action(s) continued on next page: ☒ No ☐ Yes

Project findings have been discussed with the on-site representative(s) of the interviewed organization(s):

Principal On-Site Project Manager (signature)

Print: ULI STEFFENS

Date: 11/25/94

III. To be completed by the EPA technical project manager if boxes 'd', 'e', or 'f' are checked under Part I.

The EPA technical project manager documents the following in an attachment to this form and submits his/her response to the RREL QA Manager within 10 days (Category I and II Projects) or within 20 days (Category III and IV Projects):

- Date of Corrective Action
- Summary of Corrective Action
- QA Activity Implemented to Prevent Future Occurances of the Concern(s)

RREL (CAR)
EPA-407 (Cin 7/92)

CORRECTIVE ACTION RECOMENDATION (CAR) STATEMENT

QA ID NO.: SC-926 Project Category: II

Project Title: Germany-USA Bilateral Agreement Demonstration: Harbauer Soil Washing/Vacuum Distillation System Demonstration

Organization(s) Reviewed: Institut Fresenius

On-Site Location: Taunusstein, Hessen, Federal Republic of Germany

☐ Sampling Review ☒ Analytical Review ☐ Sampling & Analytical Review

Methods/Compound Group(s) Reviewed: (1) TCLP; (2) Hg by Method 7471,7470

Reviewer and Affiliation: W. B. Blackburn, Southwest Research Associates

Review Date: 28-29 November 1994

RREL Technical Manager: D. Sanning

(Note:The interviewed organization(s) should realize that while the reviewers may be able to discuss the more obvious concerns on the spot, others may not become apparent until they have had a chance to review their notes, in detail, after returning to their office. For this reason, the reviewer's debreifing may not be as comprehensive as the written report.)

I. The reviewer should check the appropriate box or boxes:

a. ☐ No concerns were found during the review.

b. ☐ Minor concerns exist, but they are not anticipated to affect data quality.

c. ☒ Concerns exist that would or could have affected data quality; however, immediate on-site response to recommended corrective action prevented the concerns from significantly impacting data quality.

d.* ☐ Concerns exist that would or could affect data quality; however, impact on data quality may be minimized if immediate and proper corrective action is implimented.

e.* ☐ Concerns exist that have affected data quality.

f.* ☐ Other.

* Parts II and III of this form (next page) are to be completed by the reviewer and the EPA technical project manager, respectively, if boxes 'd', 'e', or 'f' are checked.

Project findings have been discussed with the on-site representative(s) of the interviewed organization(s):

	Kurz	29/11/94
Principle On-Site Project Manager (signature)	Print	Date

RREL (CAR)
EPA-407 (Cin 7/92)

CORRECTIVE ACTION RECOMMENDATION (CAR) STATEMENT
(Continued)

QA ID No.: SC-926 Project Category: II

Project Title: Germany-USA Bilateral Agreement Demonstration: Harbauer Soil Washing/Vacuum Distillation System Demonstration

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II. To be completed by the reviewer if boxes 'd', 'e', or 'f' are checked under Part I.

Nature of Concerns:

(1) Requirement for preparation in the laboratory for QAPP required methods.

(2) Sample COC not completed on receipt at laboratory.

(3) Laboratory does not have proper filter media, pressure regulator, or documentation forms for TCLP.

(4) MDL studies lacking for Methods 7470/7471.

Nature of Concern(s) continued on next page: ☒ No ☐ Yes

Recommended Action:

(1) Develop procedures to ensure staff and equipment readiness and implementation of required procedures.

(2) Complete COC form, check samples on receipt at laboratoru (QAPP Section 4.5.3).

(3) Acquire the above and perform TCLP proficiency runs before processing samples.

(4) Perform MDL studies before analyzing samples.

Recommended Action(s) continued on next page: ☒ No ☐ Yes

Project findings have been discussed with the on-site representative(s) of the interviewed organization(s):

Principal On-Site Project Manager (signature)

Kurz
Print

28/11/89
Date

III. To be completed by the EPA technical project manager if boxes 'd', 'e', or 'f' are checked under Part I.

The EPA technical project manager documents the following in an attachment to this form and submits his/her response to the RREL QA Manager within 10 days (Category I and II Projects) or within 20 days (Category III and IV Projects):

- Date of Corrective Action
- Summary of Corrective Action
- QA Activity Implemented to Prevent Future Occurances of the Concern(s)

RREL (CAR)
EPA-407 (Cin 7/92)