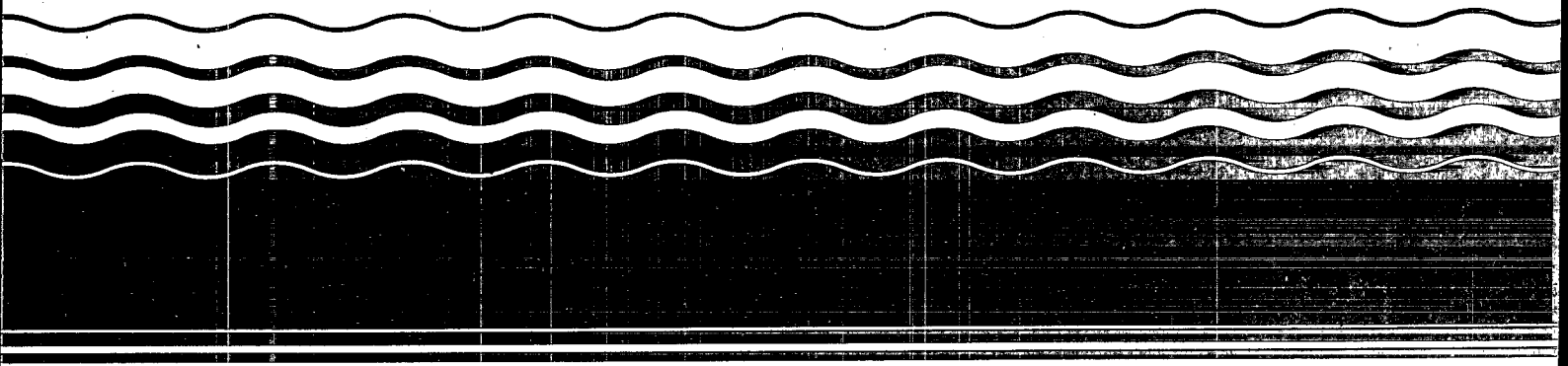


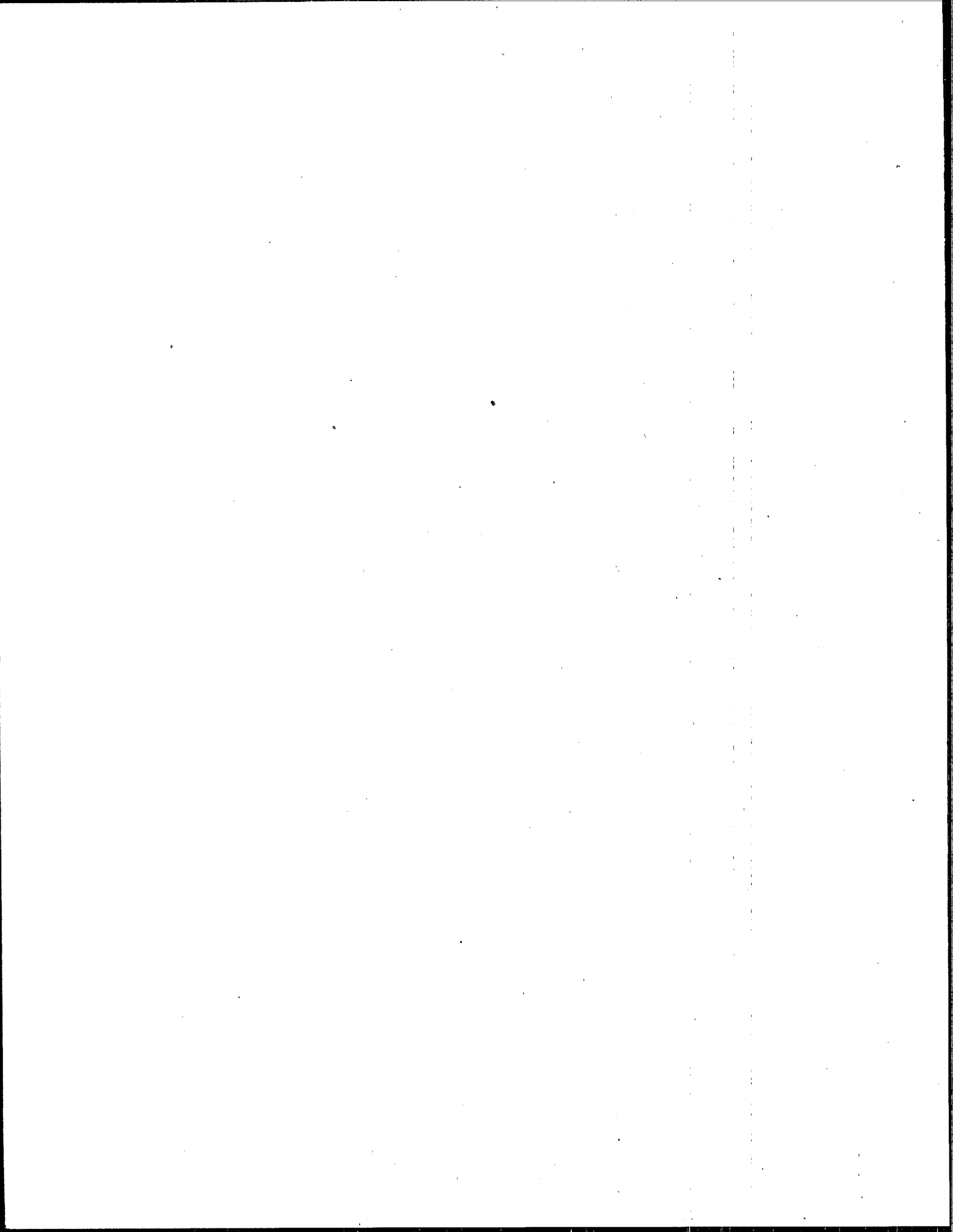
Superfund



Technology Evaluation Report: Design and Development of a Pilot-Scale Debris Decontamination System

Volume I





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August 1991

TECHNOLOGY EVALUATION REPORT:
DESIGN AND DEVELOPMENT OF A PILOT-SCALE
DEBRIS DECONTAMINATION SYSTEM
VOLUME I

RISK REDUCTION ENGINEERING LABORATORY
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NOTICE

The information in this document has been funded by the United States Environmental Protection Agency (EPA) under Contract No. 68-03-3413 and the Superfund Innovative Technology Evaluation (SITE) Program. This document has been subjected to the Agency's administrative and peer review and has been approved for publication as an EPA document. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

FOREWORD

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

This project consists of design, development, and field demonstrations under the SITE Program of an EPA-developed hydromechanical debris washing technology designed for decontamination of debris at Superfund sites. Demonstrations were conducted at a PCB-contaminated debris site in Detroit, Michigan; a PCB-contaminated transformer site in Hopkinsville, Kentucky; and a herbicide-contaminated drum site near Chickamauga, Georgia. The demonstration effort was directed toward obtaining information on performance of the technology for assessing its use at uncontrolled hazardous waste sites. Volume I of this Technology Evaluation Report describes the development, demonstration, and evaluation of the debris decontamination system. Volume II contains copies of the analytical data submitted by the various laboratories involved in the project.

A limited number of copies of this report will be available at no charge from EPA's Center for Environmental Research Information, 26 West Martin Luther King Drive, Cincinnati, Ohio 45268. Requests should include the EPA document number found on the report's front cover. When the limited supply is exhausted, additional copies can be purchased from the National Technical Information Service, Ravensworth Building, Springfield, Virginia 22161, (703) 487-4600. Reference copies will be available at EPA libraries in their Hazardous Waste Collection. You can also call the SITE Clearinghouse hotline at 1-800-424-9346 or 202-382-3000 in Washington, D.C., to inquire about the availability of other reports.

E. Timothy Oppelt, Director
Risk Reduction Engineering Laboratory

ABSTRACT

In support of EPA's SITE Program, this report documents the development, demonstration, and evaluation of an EPA-developed technology, a hydromechanical debris cleaning system, for decontamination of debris at Superfund sites. Although most of the debris at Superfund Sites has no potential for reuse, decontaminated debris could either be returned to the site as "clean fill" or in the case of metallic debris, sold to a metal smelter.

During Phase I of this project, an innovative approach for decontaminating debris--a hydromechanical cleaning system--was developed and evaluated. A bench-scale portable module consisting of an enclosure for washing debris and a closed-loop cleaning solution purification system was tested. Based on bench-scale results, a pilot-scale Experimental Debris Decontamination Module (EDDM) was developed. The EDDM was designed and assembled on a 48-foot semitrailer and field-tested at the Carter Industrial Superfund Site in Detroit, Michigan. Field testing results indicated an average of 70 percent removal of the PCBs.

Phase II was directed toward developing debris washing into a proven technology for removing various contaminants from debris found at hazardous waste sites. During Phase II, tests were performed in a controlled environment to optimize the process. A 20-gallon debris-washing unit with a spray tank and a wash tank was designed and fabricated for bench-scale studies. Results of bench-scale testing showed removal efficiencies of 95 to 99 percent for all contaminants studied (oil and grease, representative PCB's, and pesticides).

Subsequently, also as a part of Phase II, a transportable, pilot-scale, 300-gallon Debris Washing System (DWS) was designed and fabricated. The DWS entails the application of an aqueous solution during a high-pressure spray cycle, followed by a turbulent wash cycle. The aqueous cleaning solution is recovered and reconditioned for reuse concurrently with the actual debris-cleaning process, which minimizes the quantity of process water required to clean the debris. The DWS was field-tested at a PCB-contaminated (transformer casings) site in Hopkinsville, Kentucky, and at an herbicide-contaminated (drums) site near Chickamauga, Georgia.

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ACRONYMS/ABBREVIATIONS

BDAT	--	Best Demonstrated Available Technology
CERCLA	--	Comprehensive Environmental Response Compensation and Liability Act
DWS	--	Debris Washing System
EDDM	--	Experimental Debris Decontamination Module
EPA	--	United States Environmental Protection Agency
GC/MS	--	Gas Chromatograph/Mass Spectrometer
MS	--	Matrix spike
MSD	--	Matrix spike duplicate
NPL	--	National Priorities List
ORD	--	Office of Research and Development
OSWER	--	Office of Solid Waste and Emergency Response
PCB	--	Polychlorinated biphenyl
PRP	--	Potential Responsible Party
QAPJP	--	Quality Assurance Project Plan
RCRA	--	Resource Conservation and Recovery Act
RPD	--	Relative percent difference
RSD	--	Relative Standard Deviation
RREL	--	Risk Reduction Engineering Laboratory
SARA	--	Superfund Amendments and Reauthorization Act
SITE	--	Superfund Innovative Technology Evaluation
TSCA	--	Toxic Substance Control Act

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Analytical Services were provided by Radian Corporation (Austin, Texas), PCS (Dayton, Ohio), and IT Analytical Services (Cincinnati, Ohio).

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SECTION 1

EXECUTIVE SUMMARY

1.1 Introduction

Numerous sites in the United States are contaminated with hazardous waste, and the cleanup of these sites is the top environmental priority of the decade. Currently, more than 1200 sites are included in the National Priorities List (NPL), and many more have been proposed for inclusion on the list.

A typical hazardous waste site contains one or more toxic organic or inorganic chemical residues. These residues are frequently intermingled with remnants of razed structures (e.g., wood, steel, concrete block, bricks) as well as contaminated soil, gravel, concrete, and perhaps metallic debris (e.g., machinery and equipment, transformer casings, drums, and miscellaneous scrap metal). Decontamination of these materials is important as a means of preventing the spread of contamination offsite and reducing exposure levels to future users of the buildings or equipment. To date, no generally applicable decontamination technique has been developed for the removal of contaminants from structures or debris. Currently, large pieces of equipment are typically decontaminated by steam-cleaning, and contaminated buildings and structures are frequently torn down and disposed of in a hazardous waste landfill or incinerator instead of being decontaminated.

Most contaminated debris at hazardous waste sites has no potential for reuse and therefore cleanup of the site typically entails removal and transportation of the debris for off-site disposal in a RCRA Subtitle C hazardous waste landfill or incinerator. These latter options are costly and entail the risk of spreading the contamination well beyond the borders of the site. Methods are needed for the onsite decontamination of debris to reduce the risk of spreading contamination offsite and to permit debris disposal in an economical yet environmentally safe manner.

1.2 Objectives

The project consisted of two phases. The objectives of Phase I were as follows:

- To evaluate a hydromechanical cleaning system, an innovative approach for decontaminating debris.
- To conduct bench-scale testing with a portable module for the decontamination of debris.
- Based on bench-scale results, to develop a pilot-scale Experimental Debris Decontamination Module (EDDM).
- To field-test the EDDM at a hazardous waste site.

The objectives of Phase II were as follows:

- To continue development of the EDDM into a proven technology for removing various contaminants from debris found on hazardous waste sites.
- To conduct bench-scale tests to optimize the process.
- To design and construct a transportable pilot-scale debris washing system (DWS).
- To field-test the pilot-scale DWS at two hazardous waste sites where various types of debris are present.
- To prepare a conceptual design of a full-scale debris washing system.

1.3 Phase I: Development and Testing of Experimental Modules

During Phase I of the project, a hydromechanical cleaning system, an innovative approach to decontaminating debris, was developed and evaluated. A bench-scale, portable module consisting of an enclosure where debris was placed and a closed-loop solvent-delivery system was tested. Based on the bench-scale results, a pilot-scale EDDM was developed and field-tested.

A 300-gal-capacity pilot-scale EDDM was designed, assembled, installed (on a 48-ft semitrailer), and tested at the Carter Industrial Superfund Site in Detroit, MI. This site contained large quantities of different types of PCB-contaminated debris, including scrap metal, 55-gallon metal drums, tools, equipment, and some furniture items.

Two 200-lb batches of metallic debris were cleaned in the system. Before and after treatment, surface-wipe samples were obtained to determine the contaminant removal efficiency of the system. The percentage reduction of PCBs achieved during

cleaning ranged from 33 to 87 percent (average reduction of 58 percent) for Batch 1 and from 66 to 99 percent (average reduction of 81 percent) for Batch 2.

The surfactant solution in the EDDM was sampled twice during the actual cleaning process, and PCB concentrations of 928 and 420 $\mu\text{g/L}$ were found. Upon completion of the debris-washing experiment, the cleaning solution was pumped through a series of particulate filters and finally through activated carbon. The PCB concentration was reduced to 5.4 $\mu\text{g/L}$ during this treatment. Most municipalities allow water containing a PCB concentration of $<1 \mu\text{g/L}$ to be sewerred, and this level was achieved by recycling the process water through carbon a second time.

1.4 Phase II: Design, Construction, and Demonstration of a Transportable Debris-Washing System

Phase II of this project was directed toward further development of debris washing into a proven technology for removing various contaminants from debris found on hazardous waste sites in preparation for a full-scale demonstration at Superfund and other hazardous waste sites. An initial series of bench-scale tests were performed in a controlled environment to optimize the newly-designed washing system. After the bench-scale evaluation, a transportable pilot-scale version of the debris washing system (DWS) was designed, constructed, and demonstrated at actual hazardous waste sites.

Based on experience gained during the Carter site field test, a bench-scale (20 gal of surfactant solution capacity) debris washing unit was designed, constructed, and assembled. This system consisted of a spray tank, wash tank, oil-water separator, and ancillary equipment (i.e., heater, pumps, strainers, metal tray, etc.). This bench-scale DWS was developed to determine the ability of the system to remove contaminants from debris and to facilitate selection of the most efficient surfactant solution.

During these bench-scale experiments, surface-wipe samples of the six pieces of control debris were taken before and after treatment and analyzed for oil and grease. Based on the results, a nonionic surfactant solution was selected as the solution best suited for cleaning oily metal parts and debris.

As part of the continuing investigation into the performance of the DWS, the representative pieces of debris were spiked with a mixture of spiking material (used motor oil, grease, topsoil, and sand) containing representative contaminants (DDT, lindane, PCBs, and lead sulfate) and washed in the DWS with the selected surfactant solution. Three trials were performed. Surface wipe samples of debris from the first two trials were analyzed for PCBs, lindane, and DDT; the surface wipe samples from the third trial were analyzed for total lead.

The average overall reductions of PCBs and pesticides achieved during Trials 1 and 2 were greater than 99 and 98 percent, respectively. The overall reduction of lead was greater than 98 percent.

After completion of the bench-scale debris-washing experiments, the cleaning solution was neutralized to a pH of 8 and then pumped through a series of particulate filters and finally through activated carbon. During this treatment, the PCB, lindane, and DDT concentrations were reduced to <2.0 , 0.03 , and $0.33 \mu\text{g/L}$, respectively. The concentration of lead was reduced to 0.2 mg/L after treatment.

1.5 Design, Fabrication, and Demonstration of Pilot-Scale DWS

Based on the results obtained from bench-scale studies, a 300-gal capacity pilot-scale DWS was designed and constructed. The pilot-scale DWS was assembled in a warehouse in Cincinnati Ohio, and several tests were conducted. After the warehouse testing, the DWS was disassembled, loaded onto a 48-foot semitrailer, and transported to the Gray PCB site in Hopkinsville, Kentucky, which was selected for the field demonstration. The entire DWS was reassembled on a 25-ft X 24-ft concrete pad. A temporary enclosure (approximately 25 ft high) was built on the concrete pad to enclose the DWS and to protect the equipment and the surfactant solution from rain and cold weather. The Gray PCB site contained between 70 and 80 burned-out transformer casings and other large amounts of scrap metal. The demonstration took place in December 1989, and ambient temperatures were at or below freezing during the entire operation.

Before the cleaning process began, the transformer casings (ranging from 5 gal to 100 gal in size) were cut in half with a metal-cutting partner saw. A pretreatment sample was obtained from one-half of each of the transformer casings by a surface-wipe technique. The transformer halves were placed into a basket and lowered into the spray tank, which was equipped with multiple water jets that blast loosely adhered contaminants and dirt from the debris. After the spray cycle, the basket of debris was removed and transferred to the wash tank, where the debris was washed with a high-turbulence wash. Each batch of debris was cleaned for a period of 1 hr in the spray tank and 1 hr in the wash tank. During both the spray and wash cycles, a portion of the cleaning solution was cycled through a closed-loop system in which the oil/PCB-contaminated cleaning solution was passed through an oil/water separator, and the cleaned solution was then recycled into the DWS. After the wash cycle, the basket containing the debris was returned to the spray tank, where it was rinsed with fresh water.

Upon completion of the cleaning process, posttreatment surface wipe samples were obtained from each of the transformer pieces to assess the post-decontamination PCB levels. The before-treatment concentrations ranged from 0.1 to $98 \mu\text{g}/100$

cm². The after-treatment analyses showed that all the cleaned transformers had a PCB concentration lower than the acceptable level of 10 µg/100 cm².

After treatment of all the transformers at the site, the surfactant solution and the rinse water were placed in the water treatment system, where they were passed through a series of particulate filters, then through an activated-carbon drum, and finally through an ion-exchange column. The before- and after-treatment water samples were collected and analyzed for PCBs and selected metals (cadmium, copper, chromium, lead, nickel, and arsenic).

The water treatment system reduced the PCB concentration in the water to below the detection limit. The concentrations of each of the metals (except arsenic) were reduced to the allowable discharge levels set by the city of Hopkinsville. Upon receipt of the analytical results, the treated water was pumped into a plastic-covered 10,000-yd³ pile of contaminated soil at the site.

During this site cleanup, 75 transformers (approximately 5000 lb) were cleaned in the DWS. All of them are now considered clean and acceptable for sale to scrap metal dealers or to a smelter for reuse.

1.6 Demonstration at the Shaver's Farm Drum Disposal Site

In August 1990, a second demonstration of the DWS was conducted at the Shaver's Farm drum disposal site near Chickamauga, Georgia, where 55-gal drums containing varying amounts of a herbicide, Dicamba (2-methoxy-3,6-dichlorobenzoic acid), and benzonitrile (a precursor in the manufacture of Dicamba) were buried. EPA Region IV had excavated more than 4000 drums from one location on this 5-acre site when this demonstration occurred.

The pilot-scale system was transported to this site on a 48-ft semitrailer and assembled on a 25 x 24 ft concrete pad. The temporary enclosure used at the Gray site was reassembled to protect the equipment from rain. Ambient temperature at the site during the demonstration ranged from 75° to 105°F.

The 55-gal herbicide-contaminated drums were cut into four sections, and pretreatment surface-wipe samples were obtained from each section. The drum pieces were first placed in the spray tank of the DWS for 1 hr of surfactant spraying, then in the wash tank for an additional hour of surfactant washing, and finally in the spray tank for 30 min of water rinsing. The drum pieces were then allowed to air-dry before the posttreatment surface-wipe samples were taken. Ten batches of 1 to 2 drums per batch were treated during this demonstration.

Pretreatment concentrations of benzonitrile in surface wipe samples ranged from 8 to 47,000 µg/100 cm² and averaged 4556 µg/100 cm²; posttreatment samples

ranged from below detection limit to $117 \mu\text{g}/100 \text{ cm}^2$ and averaged $10 \mu\text{g}/100 \text{ cm}^2$. Pretreatment Dicamba values ranged from below detection limit to $180 \mu\text{g}/100 \text{ cm}^2$ and averaged $23 \mu\text{g}/100 \text{ cm}^2$; posttreatment concentrations ranged from below detection limit to $5.2 \mu\text{g}/100 \text{ cm}^2$ and averaged $1 \mu\text{g}/100 \text{ cm}^2$.

All Superfund site activities described in this document were governed by EPA-approved Health and Safety and Quality Assurance Plans.

1.7 Conclusions

Field-test results obtained with the pilot-scale DWS during demonstrations at two Region IV hazardous waste sites showed the unit to be both transportable and rugged. Extreme high and low temperatures had little effect on the operation of the equipment. The system successfully removed PCBs from transformer casing surfaces and herbicides, pesticides, dioxins, and furan residues from drum surfaces.

The cleaning solution was recovered, reconditioned, and reused during the actual debris-cleaning process; this minimized the quantity of process water required for the decontamination procedure. The water treatment system was effective in reducing contaminant concentrations, with the exception of arsenic and possibly Dicamba, to below the detection limit.

Planned progression of this EPA-developed technology includes design, development, and demonstration of a full-scale, transportable version of the DWS unit.

SECTION 2

INTRODUCTION

2.1 Background

Congress enacted the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) of 1980 to address past hazardous waste disposal practices and the environmental and human health effects of those practices. In the reauthorization of CERCLA, called the Superfund Amendments and Reauthorization Act of 1986 (SARA), Congress expressed concern over the use of land-based disposal and containment technology to mitigate releases of hazardous substances at hazardous waste sites.

In response to SARA, the EPA's Office of Solid Waste and Emergency Response (OSWER) and Office of Research and Development (ORD) established a formal program to promote the development and use of innovative technologies to clean up Superfund sites across the country. This program is called the Superfund Innovative Technology Evaluation (SITE) Program.

2.2 SITE Program

The overall goal of the SITE Program is to "carry out a program of research, evaluation, testing, development and demonstration of alternative or innovative treatment technologies... which may be utilized in response actions to achieve more permanent protection of human health and welfare and the environment." Specifically, the program's goal is to maximize the use of alternatives to land disposal in cleaning up Superfund sites by encouraging the development and demonstration of new, innovative treatment and monitoring technologies.

The SITE Program comprises four major elements:

- Demonstration Program
- Emerging Technologies Program
- Measurement and Monitoring Technologies Program
- Technology Information Services

The Demonstration Program is one of the most important aspects of the SITE Program which evaluates field- or pilot-scale technologies that can be scaled up for commercial use. The Demonstration Program is the primary focus of the SITE Program because the technologies evaluated are close to being available for remediation of Superfund sites. The main objective of the Demonstration Program is to develop extensive performance engineering and cost information for new technologies. With this information, potential users can make informed decisions on whether to use these technologies to remediate hazardous waste sites.

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

Technologies are selected for the SITE Demonstration Program through annual requests for proposal. Proposals are reviewed by EPA to determine the technologies with the most promise for use at Superfund sites. To qualify for the program, a new technology must have been developed to pilot or full scale and must offer some advantage over existing technologies. Mobile technologies are of particular interest.

Once EPA has accepted a proposal, the Agency and the developer work with the EPA Regional Offices and State agencies to identify a site containing wastes suitable for testing the capabilities of the technology. The developer is responsible for demonstrating the technology at the selected site, and is expected to pay the costs to transport, operate, and remove the equipment. The EPA is responsible for project planning, sampling and analysis, quality assurance and quality control, preparing reports, and disseminating information.

The Emerging Technologies Program focuses on conceptually proven, but untried technologies. These technologies are in an early stage of development involving laboratory or pilot testing. Successful technologies are encouraged to advance to the Demonstration Program.

The Monitoring and Measurement Technologies Program identifies existing technologies that can improve field monitoring and site characterizations. It supports the development and demonstration of new technologies that provide faster, more cost-effective real-time data on contamination and cleanup levels. Finally, it formulates the protocols and Standard Operating Procedures for demonstrated methods and equipment.

An Applications Analysis Report and Technology Evaluation Report are published at the conclusion of each demonstration. Research reports on emerging technology projects are also produced. Results and status updates are distributed to the user community - EPA Regions, state agencies, remediation contractors, and responsible parties - through many media and activities.

2.3 Program Objectives

The following were the objectives of this two-phased SITE Project for decontamination of debris, using the debris washing system. The objectives of Phase I were as follows:

- To evaluate a hydromechanical cleaning system, an innovative approach for decontaminating debris.
- To conduct bench-scale testing with a portable module for the decontamination of debris.
- Based on bench-scale results, to develop a pilot-scale Experimental Debris Decontamination Module (EDDM).
- To field-test the EDDM at a hazardous waste site.

The objectives of Phase II were as follows:

- To continue development of the EDDM into a proven technology for removing various contaminants from debris found on hazardous waste sites.
- To conduct bench-scale tests to optimize the process.
- To design and construct a transportable pilot-scale debris washing system (DWS).
- To field-test the pilot-scale DWS at two hazardous waste sites where various types of debris are present.
- To prepare a conceptual design of a full-scale debris washing system.

2.4 Purpose of This Report

The Technology Evaluation Report provides a comprehensive description of the demonstration and its results. This report is intended for engineers performing a detailed evaluation of the technology for a specific site and waste situation. The

purpose of these technical evaluations is to obtain a detailed understanding of the performance of the technology during the demonstration and to ascertain the advantages, risks, and costs of the technology for the given application. This information is used to produce conceptual designs in sufficient detail to enable the preparation of preliminary cost estimates for the demonstrated technology.

2.5 Report Organization

This Technology Evaluation Report is presented in two volumes. Volume I describes the design and development of the pilot-scale debris decontamination system and presents the results of the demonstrations conducted at three hazardous waste sites. Section 3 discusses Phase I of the project, which included the development and testing of the EDDM. Section 4 presents information on Phase II, which entailed the design, construction and field demonstrations of a transportable debris washing system. Section 5 contains a summary of the Quality Assurance aspects of the analyses performed during the field testing of the pilot-scale DWS. Section 6 contains a summary of cost of demonstrations. Section 7 presents conclusions and recommendations. Section 8 addresses the conceptual design of a full-scale debris washing system.

Volume II contains copies of the analytical data submitted by the various laboratories involved in the project. Both volumes are available from NTIS.

SECTION 3

PHASE I: DEVELOPMENT AND TESTING OF AN EXPERIMENTAL DEBRIS-DECONTAMINATION MODULE

During Phase I of the project, a hydromechanical cleaning system, an innovative approach to decontaminating debris, was developed and evaluated. A bench-scale portable module consisting of an enclosure for placement of debris and a closed-loop solvent-delivery system was tested for decontamination of debris at hazardous waste sites. Based on the bench-scale results, a pilot-scale Experimental Debris Decontamination Module (EDDM) was then developed and field-tested.

The purpose of Phase I was to develop and prove the concept of this innovative approach to cleaning contaminated debris. This section presents the development, procedure, and results of the bench- and field-testing of the EDDM.

3.1 Bench-Scale Experiments

Before the details of the pilot-scale design of the EDDM were finalized, a bench-scale experiment was performed to determine the best commercially available cleaning solution possible for cleaning PCB-contaminated debris and metal parts in the field. Four cleaning solutions were selected for the experiment: tap water, 10 percent sulfuric acid, and the two detergents BB-100 and Power Clean (BB-100 and Power Clean are nonionic, biodegradable industrial degreasers).

The experimental procedure involved the application of measured quantities of used motor oil, grease, and soil to rusted iron parts to simulate the kind of grime likely to be encountered on oily, PCB-contaminated metal parts and debris in the field. Experiments were performed in a 10-gallon hydromechanical cleaning unit, that contains an axial flow pump, a propeller shaft, a propeller, a pressure chamber, and a calm fluid section. Three tests were performed with each cleaning solution, and a fresh set of oil/grease-contaminated metal parts were used in each test. For consistency, each set of contaminated parts was matched closely with regard to the size, shape, and type of metal. The parts were also arranged in the same order in the parts-washer basket during washing.

Upon completion of each test, two aliquots of cleaning solution were collected; one aliquot was submitted for oil and grease analysis and the other, for total suspended solids analysis. Two surface-wipe samples from selected metal parts were also collected for oil and grease analysis to determine the level of oil/grease remaining on the metal surfaces after treatment in the parts washer. The skimmer oil from each of the three runs was mixed together for oil and grease analysis. All the samples were analyzed at PCS, Inc. (now called the Hayden Environmental Group, Inc.) in Dayton, Ohio.

3.1.1 Results of Bench-Scale Studies

Table 1 summarizes the results of the oil/grease and total suspended solids analyses. The analytical results obtained for the wipe samples indicate that the amount of oil and grease remaining on the metal surfaces was significantly higher after cleaning with tap water and 10 percent sulfuric acid and comparatively lower after cleaning with BB-100 and Power Clean. This demonstrates the poor cleaning performance of water and sulfuric acid. Moreover, the handling of 10 percent sulfuric acid was difficult, and the acid had a corroding effect on the hydromechanical cleaning equipment. Hence, it was concluded that water and sulfuric acid should not be considered as potential cleaning solutions for oily PCB-contaminated debris.

Based on the results of the surface-wipe testing listed in Table 1, a BB-100 solution appears to clean better than a Power Clean solution. This is also shown graphically in Figure 1, which plots the results of wipe samples (in milligrams of oil and grease per square centimeter) for each run. The results tend to indicate that BB-100 removed solids from metal surfaces more effectively than did Power Clean. The data also show that upon completion of the third run, the BB-100 solution still had more cleaning capacity to remove dirt from metal parts than did Power Clean. Hence, of the four cleaning solutions tried, BB-100 was selected as the cleaning solution best suited for cleaning oily PCB-contaminated metal parts and debris in the field.

3.2 Field Demonstration of EDDM at the Carter Industrial Site

3.2.1 Site Description

Carter Industrial, a Superfund site, located at 4690 Humboldt Avenue, near Interstate Highway 98 in Detroit, Michigan, was made available by Region V for demonstration of the EDDM. The site consists of a U-shaped area into which Humboldt Avenue enters at the center. The site contains two buildings plus an incinerator and a smelter. A 20-ft x 20-ft concrete pad is located in the area where Humboldt Avenue dead ends at the site, and a small flat dirt area is located adjacent to the concrete pad. A vacant lot is located 500 ft south of the site at the corner of Humboldt and Forest Avenues. Figure 2 presents a site diagram of the Carter Site. Previous operations at Carter may have involved draining PCB-contaminated oil from transformers and burning materials in onsite incinerators. The site contains large quantities of

**TABLE 1. SUMMARY OF RESULTS FOR OIL/GREASE AND TOTAL
SUSPENDED SOLIDS ANALYSIS**

Experimental Run No.	Sample Type ^a	Analysis	Cleaning Solution			
			Water	Sulfuric Acid Conc. 10%, wt./vol.	BB-100 Conc. 15%, v/v	Power Clean Conc. 1:6 Ratio
1	Cleaning solution	Oil and grease, mg/liter	42	161	7	1670
2	Cleaning solution	Oil and grease, mg/liter	151	143	182	1470
3	Cleaning solution	Oil and grease, mg/liter	241	138	319	2440
1	Cleaning solution	Total suspended solids, mg/liter	5	128	600	206
2	Cleaning solution	Total suspended solids, mg/liter	7	255	904	576
3	Cleaning solution	Total suspended solids, mg/liter	15	148	1000	484
1	Wipe No. 1	Oil and grease, mg/cm ²	1.77	1.48	0.32	0.50
1	Wipe No. 2	Oil and grease, mg/cm ²	1.82	1.75	0.25	0.49
2	Wipe No. 1	Oil and grease, mg/cm ²	10.54	0.7	0.15	0.43
2	Wipe No. 2	Oil and grease, mg/cm ²	4.40	4.8	0.42	0.48
3	Wipe No. 1	Oil and grease, mg/cm ²	2.43	3.81	0.26	0.34
3	Wipe No. 2	Oil and grease, mg/cm ²	NA ^b	3.27	0.33	0.61
Total of 1, 2, & 3	Oil from skimmer	Oil and grease, mg/liter	NA	1540	3380	3900

^a All samples are posttreatment samples.

^b Not Analyzed.

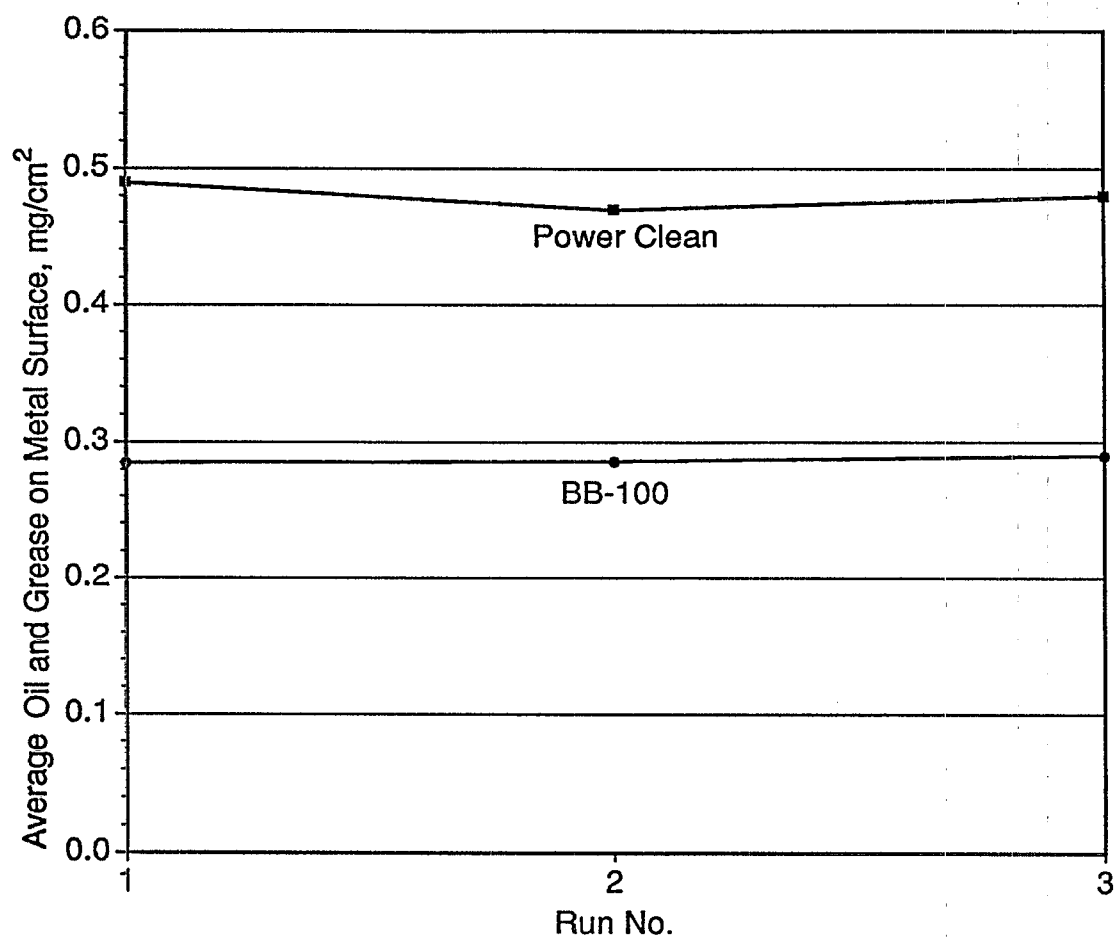


Figure 1. Amount of oil and grease on metal surface after completion of cleaning cycle.

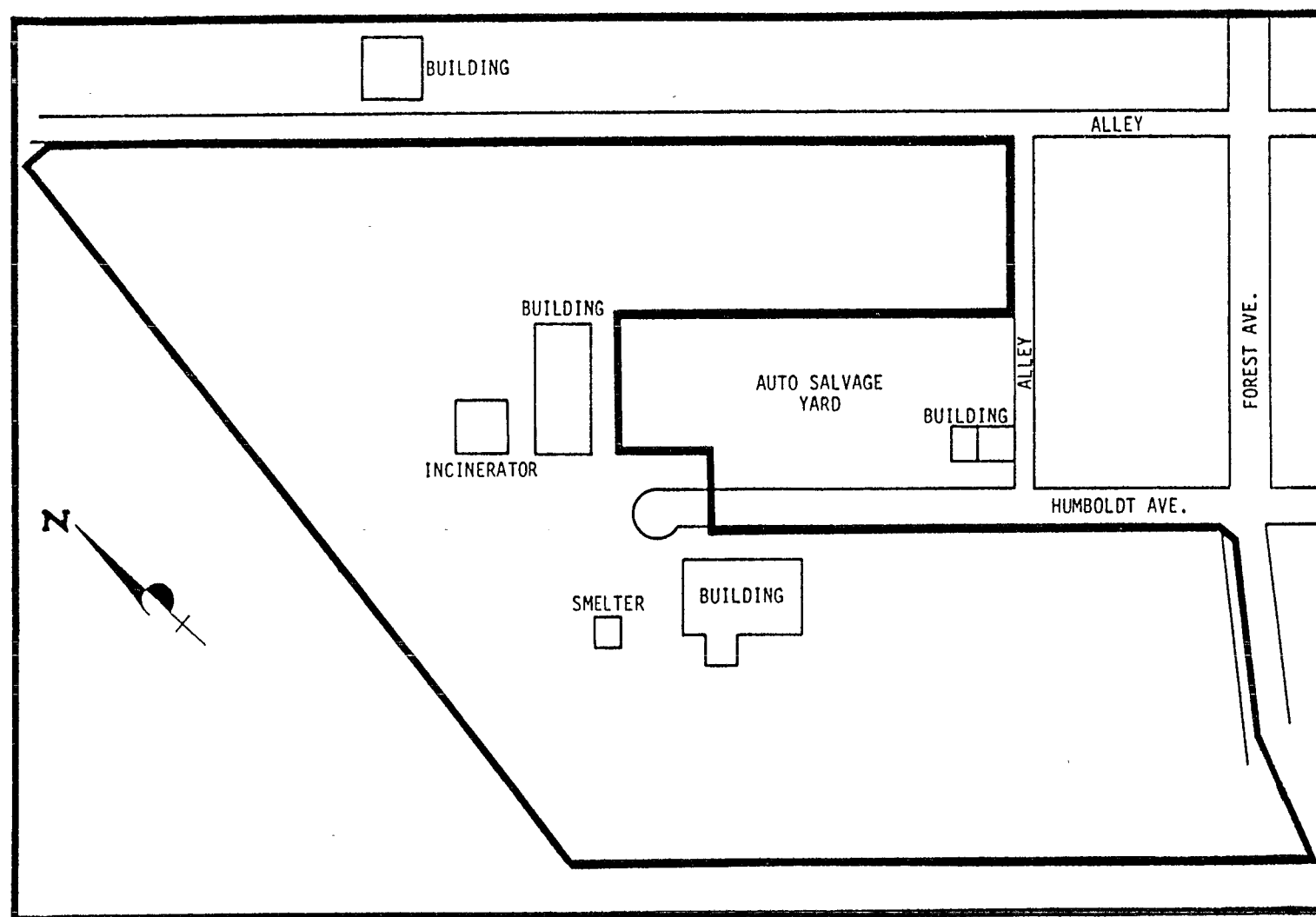


Figure 2. Carter Industrial site map, Detroit, Michigan.

different types of PCB-contaminated debris, including large quantities of scrap metal, 55-gallon metal drums, tools, equipment, and some furniture items.

3.2.2 Process Operation of EDDM During Field Testing

A 300-gallon-capacity EDDM was assembled on a 48-foot trailer in a warehouse in Cincinnati, Ohio, and was transported to the Carter Site, where it was field-tested. The EDDM consisted of a hydromechanical cleaning tank, a chip-removal system, and an oil/water separator.

The hydromechanical cleaning tank (300-gallon capacity) is an off-the-shelf, patented device consisting of an axial flow pump, a propeller shaft, propeller, pressure chamber, and calm fluid section. During washing, the unit removes oil from the parts. The oil is suspended in the cleaning fluid and carried into the calm fluid section of the tank, where the oil is allowed to accumulate on the surface. A skimmer is provided to collect oil in the calm fluid section of the tank. The oil is wiped off the skimmer wheel by the wiper blades and drained to a container for disposal.

The chip removal system is a mobile, self-contained, portable unit consisting of a centrifugal pump and a perforated, stainless steel strainer basket. During washing, the chip removal system automatically removes chips that accumulate from parts washing. Figure 3 represents a flow diagram of the pilot-scale module.

Two 200-lb batches of metallic debris were cleaned with a solution of BB-100 surfactant in the hydromechanical cleaning unit. Before the cleaning process was initiated, five individual pieces of metal from each batch were sampled for PCBs by a surface-wipe technique (1). The debris items were placed in a basket, the basket was transferred into the EDDM, and the cleaning process was initiated. Each batch of debris was cleaned for a period of 2 hours. The cleaning solution was cycled through a continuous closed-loop system, where the oil/PCB-contaminated wash solution was passed through an oil/water separator, and the clean solution was then recycled into the module. At the completion of the cleaning process, five additional wipe samples were obtained from the same pieces of metallic debris to assess the post-decontamination level of PCBs. The surface-wiping procedure was carried out as described in the Field Manual for Grid Sampling of PCB Spill Sites to Verify Cleanup (1), which entails the use of a hexane-soaked cotton gauze pad to wipe a 100-cm² area on the surface of the object being sampled. A detailed description of surface-wipe sampling is presented in Section 5.

3.2.3 Results of Field Demonstration

Table 2 shows the quantity of PCBs on the surface of each piece of metal before and after cleaning. The percentage reduction of PCBs achieved during cleaning ranges from 33 to 87 percent (average reduction of 58 percent) for Batch 1 and from

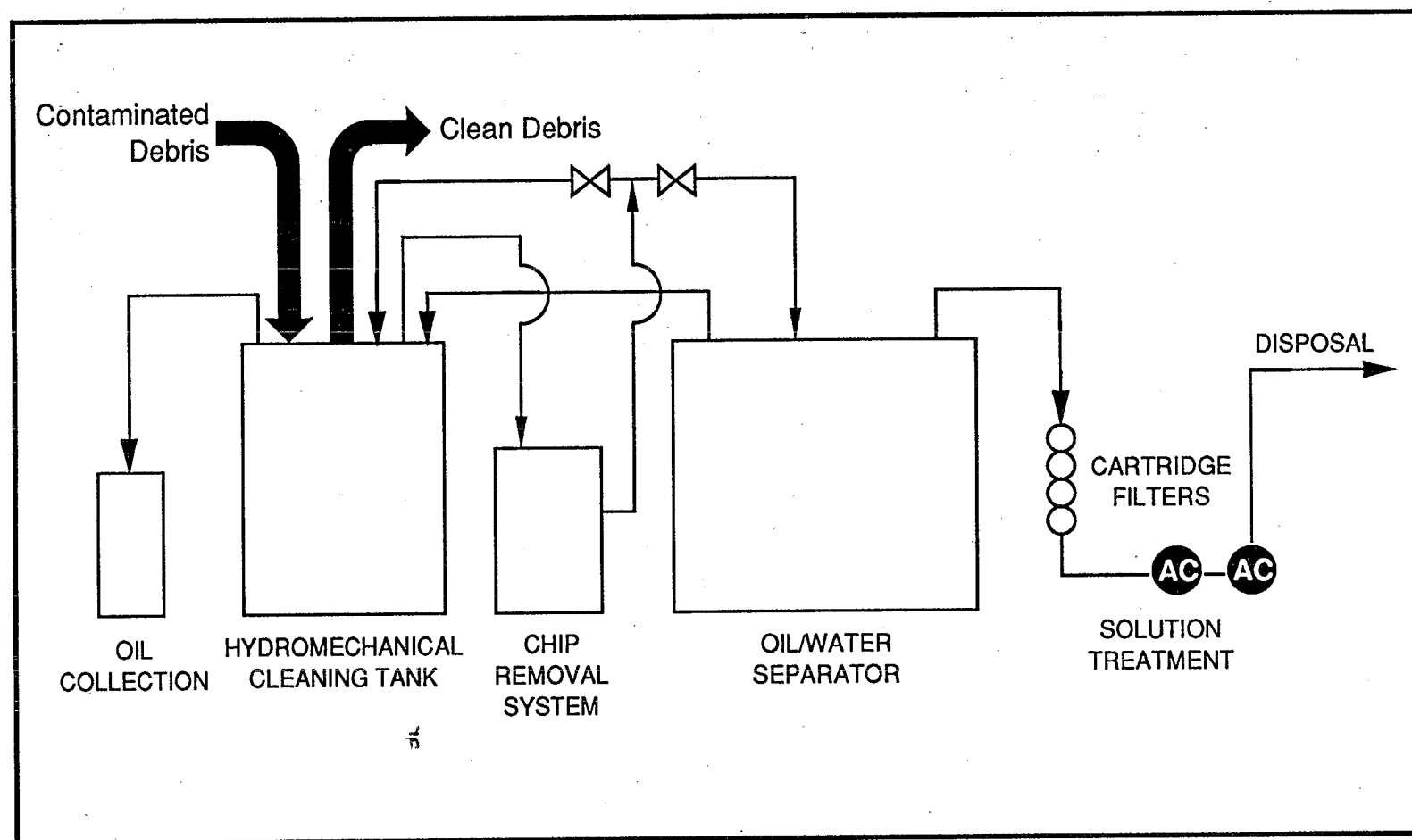


Figure 3. Schematic of pilot-scale experimental debris decontamination module.

**TABLE 2. ANALYTICAL RESULTS OBTAINED DURING FIELD DEMONSTRATION OF EDDM
AT CARTER INDUSTRIAL SUPERFUND SITE**

Batch Number	Sample ID Number		PCB Concentration on Surfaces ($\mu\text{g}/100\text{cm}^2$)		% Reduction
	Pretreatment	Posttreatment	Pretreatment	Posttreatment	
1	9/7-DET-B1-S1	9/7-DET-B1-S7	134	50	63
	9/7-DET-B1-S2	9/7-DET-B1-S8	490	178	64
	9/7-DET-B1-S3	9/7-DET-B1-S9	1280	856	33
	9/7-DET-B1-S4	9/7-DET-B1-S10	73	43	41
	9/7-DET-B1-S5	9/7-DET-B1-S11	203	23	87
	FB-1-9/8		Field Blank: <1.0		Avg: 58
2	9/8-DET-B2-S1	9/8-DET-B2-S6	8	13	- 63
	9/8-DET-B2-S2	9/8-DET-B2-S7	6090	1800	70
	9/8-DET-B2-S3	9/8-DET-B2-S8	374	128	66
	9/8-DET-B2-S4	9/8-DET-B2-S9	96	10	90
	9/8-DET-B2-S5	9/8-DET-B2-S10	1690	18	99
	FB-2-9/8		Field Blank: 1.0		Avg: 81

66 to 99 percent (average reduction of 81 percent) for Batch 2. In the case of Sample 1 in Batch 2, however, the PCB concentration apparently increased after the cleaning process, probably because the posttreatment wipe sample in this case was obtained from a location on the debris surface that was initially more heavily contaminated with PCBs.

The results seem to indicate that more effective removal of PCBs was achieved during the cleaning of Batch 2 than during the cleaning of Batch 1. Better cleaning results for Batch 2 may be explained by the following. In the case of Batch 2, the basket containing the debris was removed from the washer after 1 hour of cleaning, and the parts were manually rearranged so that all the sides of debris were exposed to the cleaning solution with the same force of the turbo washer. The basket was then lowered back into the washer and cleaning was continued for 1 more hour. In the case of Batch 1, however, the cleaning process was continued for 2 hours without the debris in the basket being rearranged.

The surfactant solution in the Turbowasher was sampled twice during the actual cleaning process, and PCB concentrations of 928 and 420 $\mu\text{g/L}$ were found. After completion of the debris-washing experiment, the cleaning solution was pumped through a series of particulate filters and finally through activated carbon. The PCB concentration was reduced to 5.4 $\mu\text{g/L}$ during this treatment. Most municipalities allow water containing a PCB concentration of $<1 \mu\text{g/L}$ to be sewerred, and this level was achieved by recycling the process water through carbon a second time.

The copies of bench-scale and pilot-scale analytical data provided by the PCS, Inc., Laboratory are included in Volume II of this report.

SECTION 4

PHASE II: DESIGN, CONSTRUCTION, AND DEMONSTRATION OF A TRANSPORTABLE DEBRIS-WASHING SYSTEM

Phase II of this project was directed toward further development of debris washing into a proven technology for removing various contaminants from debris found on hazardous waste sites in preparation for full-scale demonstrations at Superfund and other hazardous waste sites. An initial series of bench-scale tests were performed in a controlled environment for further optimization of the newly designed equipment and washing process. After bench-scale evaluation, a transportable pilot-scale version of the debris washing system (DWS) was designed, constructed, and demonstrated at actual hazardous waste sites.

The knowledge and experience acquired during the debris decontamination demonstration at the Carter site were used in the design and construction of a bench-scale debris washing unit. This unit was a smaller version of the pilot-scale unit that was designed and constructed later in the project. The bench-scale DWS was developed to assess the ability of the system to remove contaminants from debris and to facilitate selection of the most efficient surfactant solution.

The bench-scale system (20-gallon size) consisted of a spray tank, a wash tank, an oil/water separator, and ancillary equipment (such as heaters, pumps, strainers, metal tray, etc.). Although the bench-scale DWS and the pilot-scale DWS developed in Phase II were substantially different from the EDDM used in Phase I, the concept of the system remained the same. Some of the major changes that were introduced in the development of DWS compared with EDDM are as follows:

- 1) A high-pressure spray tank was added to the system for the removal of excessive or firmly attached gross contamination from the debris prior to its cleaning in the wash tank. Experience gained at the Carter site indicated that removing gross contamination before placing debris in the washer would increase the cleaning efficiency of the system.
- 2) The wash tank was modified to give high turbulence to increase surface-cleaning performance.

- 3) The EDDM was a single-step process (only a wash step), whereas the DWS was a three-step process (a high-pressure spray step followed by a high-turbulence wash step, and finally a water-rinse step).
- 4) The DWS was assembled on a concrete pad on the ground, whereas the EDDM was mounted on a trailer.

Details of the bench-scale and pilot-scale DWS are presented in this section.

4.1 Bench-Scale Experiments and Results

The best cleaning solution tested in Phase I, BB-100, was used at the Carter Industrial Site during the pilot-scale demonstration. Although BB-100 was found to be an effective cleaning solution, a decision was made to test other nonionic surfactants that might be more effective and/or more economical than BB-100.

An extensive survey was made of several different types of commercially available cleaning solutions. Four additional nonionic, nontoxic, low foaming, metal-cleaning surfactant solutions (BG-5, MC-2000, LF-330, and L-422) were selected for an experimental evaluation with BB-100 to determine their capacity to solubilize and remove contaminants from the surface of the debris. The extracting power of these undissociated surfactants is related to the presence of a proper balance of hydrophilic and lipophilic groups within the molecule, which enable surfactants to interact with both polar and nonpolar substances. Unlike the anionic and cationic types, the effectiveness of nonionic surfactants are not susceptible to moderate pH changes and the presence of electrolytes. Surfactants with a proper balance in their hydrophilic and lipophilic affinities are effective emulsifying agents because they tend to concentrate at the oil/water interface.

During the bench-scale experiments, an attempt was made to select debris that was representative of the types of material that might be found on a hazardous waste site. For purposes of consistency with regard to the shape, size, type of metal, and sampling procedure, a set of representative or typical debris was selected, which included three rusted metal plates, a brick, a concrete block, and a piece of plastic. These six pieces of representative debris were used in each of the bench-scale tests. Automotive parts obtained from an auto salvage yard were also included with each batch of representative debris. Cleanliness of these salvage yard items was visually checked at the conclusion of each trial run to determine the surfactant's performance on aged greasy and oily debris.

Prior to each bench-scale experiment, the six pieces of control debris were "contaminated" by dipping them into a spiking material consisting of known amount of used motor oil, grease, topsoil, and sand. The pieces of "contaminated" debris were then arranged on a metal tray with some assorted parts from an auto salvage yard.

The tray containing the debris was inserted in the spray tank and subjected to a high-pressure spray of surfactant solution for 15 minutes. At the end of spray cycle, the tray was transferred to the high-turbulence wash tank, where the debris was washed for 30 minutes with a solution of the same surfactant as that in the spray tank. After the wash cycle was completed, the tray was removed from the wash tank and the debris was allowed to air-dry. The bench-scale DWS is shown in Figure 4.

Surface-wipe samples were obtained from the six pieces of control debris before and after treatment. These wipe samples were analyzed for oil and grease, and the results are summarized in Table 3. Based on the results of the wipe testing, L-422 was selected as the solution best suited for cleaning oily metal parts and debris.

As part of the continuing investigation into the performance of the DWS, representative pieces of debris were spiked with a mixture of spiking material (used motor oil, grease, topsoil, and sand) containing representative contaminants (DDT, lindane, PCB, and lead sulfate) and washed in the DWS with L-422 as the cleaning solution. Three trials were performed. Surface-wipe samples from debris from the first two trials were analyzed for PCB, lindane, and DDT, and the surface-wipe samples from the third trial were analyzed for total lead.

Tables 4 and 5 summarize the quantities of PCBs and pesticides on the surface of each piece of debris before and after cleaning. Table 6 summarizes the quantities of lead found before and after treatment. The average overall percentage reductions of PCBs and pesticides achieved during Trials 1 and 2 were greater than 99 and 98 percent respectively. The overall percentage reduction of lead was greater than 98 percent.

After the completion of the bench-scale debris-washing experiments, the cleaning solution was neutralized to a pH of 8 and then pumped through a series of particulate filters and finally through activated carbon. During this treatment, the PCB, lindane, and DDT concentrations were reduced to <2.0 , 0.03 , and $0.33 \mu\text{g/L}$, respectively. The concentration of lead was reduced to 0.2 mg/L after treatment. During the water treatment, it was noticed that a gel-like precipitate was formed when the L-422 cleaning solution was neutralized to pH 8. This precipitate quickly plugged the particulate filters and had the potential for clogging the activated carbon drums. As a result, BG-5, which performed almost as well as L-422 and did not form any precipitate when neutralized, was selected as the cleaning solution for the pilot-scale study.

After BG-5 was selected as the optimal cleaning solution, experiments were conducted with 3 percent and 5 percent concentration solutions of BG-5 to determine the optimum concentration. Again, oily debris from a salvage yard was cleaned; based on the visual inspection, the 5 percent BG-5 solution obviously performed better than the 3 percent solution. The optimum temperature recommended by the

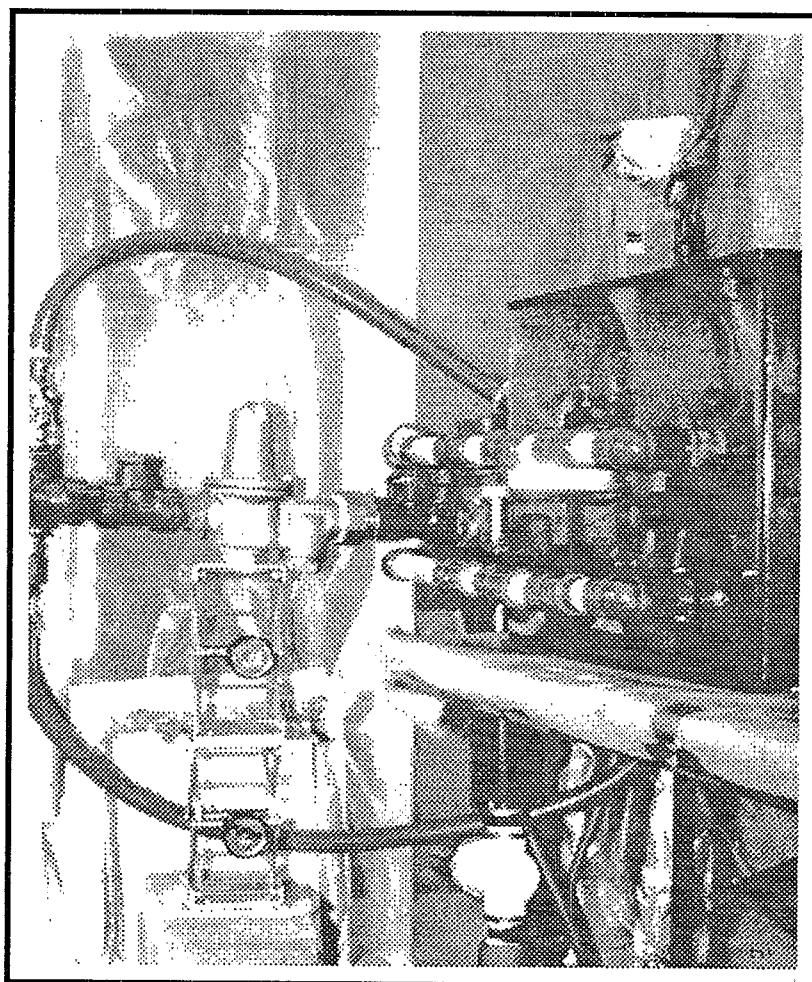


Figure 4. Bench-scale Debris Washing System.

TABLE 3. A COMPARISON OF THE CLEANING CAPABILITIES OF SURFACTANT SOLUTIONS BASED ON REMOVAL OF OIL AND GREASE

	Sample ID Number	Pretreatment (g/100 cm ²)	Posttreatment (g/100 cm ²)	Percent Reduction	Avg. Percent Reduction
BG-5	C1-1-6/20	6.7131	0.0071	99.89	99.82
	C2-1-6/20	5.2936	0.0171	99.68	
	C3-1-6/20	5.5088	0.0111	99.80	
	C4-1-6/20	5.4138	0.0060	99.89	
	C5-1-6/20	4.7310	0.0074	99.84	
	C6-1-6/20	5.4889	0.0107	99.80	
MC-2000	C1-2-6/22	4.1462	0.0313	99.25	99.57
	C2-2-6/22	4.0274	0.0155	99.61	
	C3-2-6/22	4.0025	0.0322	99.19	
	C4-2-6/22	6.7795	0.0057	99.91	
	C5-2-6/22	7.3356	0.0162	99.78	
	C6-2-6/22	4.2899	0.0144	99.66	
LF-330	C1-5-6/30	5.2878	0.0688	98.70	98.74
	C2-5-6/30	5.0433	0.0878	98.26	
	C3-5-6/30	5.8143	0.0811	98.61	
	C4-5-6/30	6.0277	0.0533	99.11	
	C5-5-6/30	4.1388	0.0262	99.37	
	C6-5-6/30	4.1278	0.0667	98.38	
BB-100	C1-6-6/30	4.5929	0.0221	99.52	98.71
	C2-6-6/30	4.9409	0.0349	99.29	
	C3-6-6/30	5.3973	0.1315	97.56	
	C4-6-6/30	4.9976	0.1498	97.00	
	C5-6-6/30	3.9820	0.0067	99.83	
	C6-6-6/30	4.9440	0.0477	99.03	
L-422	C1-7-7/6	5.1850	0.0017	99.97	99.96
	C2-7-7/6	4.8263	0.0028	99.94	
	C3-7-7/6	5.1807	0.0011	99.98	
	C4-7-7/6	5.8047	0.0023	99.96	
	C5-7-7/6	5.0469	0.0015	99.97	
	C6-7-7/6	4.7127	0.0019	99.96	

**TABLE 4. SUMMARY OF BENCH-SCALE RESULTS OF CONTROLLED
DEBRIS ANALYZED FOR PCBs AND PESTICIDES (TRIAL 1)**

	Sample ID Number	Contaminant	Pretreatment (µg/100 cm ²)	Posttreatment ^a (µg/100 cm ²)	Percent Reduction	Average Performance
Metal	CM1-SP1-7/21	Lindane	13,800	0.75	99.99	≥99.87
		4, 4' DDD	1010	3.8 U	≥99.62	
		4, 4' DDT	6710	5.0 U	≥99.93	
		PCB-1260	3550	2.0 U	≥99.94	
	CM2-SP1-7/21	Lindane	12,500	0.7	99.99	
		4, 4' DDD	1020	3.8 U	≥99.63	
		4, 4' DDT	7610	5.67	99.93	
		PCB-1260	3230	2.0 U	≥99.94	
	CM3-SP1-7/21	Lindane	12,300	0.7	99.99	
4, 4' DDD		1020	3.8 U	≥99.63		
4, 4' DDT		7800	5.0 U	≥99.93		
PCB-1260		2990	2.0 U	≥99.93		
Brick	CB1-SP1-7/21	Lindane	14,600	5.8	99.96	≥98.88
		4, 4' DDD	1220	3.8 U	≥99.69	
		4, 4' DDT	7640	11.6	99.85	
		PCB-1260	2570	20.3	99.21	
	CB2-SP1-7/21	Lindane	12,900	130	98.99	
		4, 4' DDD	1170	4.9	99.58	
		4, 4' DDT	10,100	360	96.43	
		PCB-1260	3360	90.4	97.31	
Concrete Block	CC1-SP1-7/21	Lindane	14,000	11.1	99.92	≥99.72
		4, 4' DDD	1240	3.8 U	≥99.69	
		4, 4' DDT	10,200	28.3	99.72	
		PCB-1260	3410	15.3	99.55	
Plastic	CP1-SP1-7/21	Lindane	9370	1.1	99.99	≥99.62
		4, 4' DDD	952	3.8 U	≥99.60	
		4, 4' DDT	7120	12.6	99.82	
		PCB-1260	2500	23.4	99.06	
Average Overall Performance						≥99.39

^aU indicates that the target compound was not detected at this level.

TABLE 5. SUMMARY OF BENCH-SCALE RESULTS OF CONTROLLED DEBRIS ANALYZED FOR PCBs AND PESTICIDES (TRIAL 2)

	Sample ID Number	Contaminant	Pretreatment (µg/100 cm ²)	Posttreatment ^a (µg/100 cm ²)	Percent Reduction	Average Performance
Metal	CM1-7/24	Lindane	11,800	0.13 U	100	≥99.91
		4, 4' DDT	9320	2.32	99.97	
		PCB-1260	1770	2.0 U	≥99.89	
	CM2-7/24	Lindane	8180	0.31 U	100	
		4, 4' DDT	7540	4.8	99.94	
		PCB-1260	1780	2.79	99.84	
	CM3-7/24	Lindane	6150	0.41	99.99	
		4, 4' DDT	5840	2.61	99.95	
		PCB-1260	1450	2.0 U	≥99.86	
Brick	CB1-7/24	Lindane	5810	3.49	99.94	99.80
		4, 4' DDT	5660	10.5	99.81	
		PCB-1260	1220	4.1	99.66	
Concrete Block	CC1-7/24	Lindane	6440	397	93.83	94.39
		4, 4' DDT	6610	389	94.11	
		PCB-1260	1390	66.1	95.24	
Plastic	CP1-7/24	Lindane	10,300	52	99.49	98.22
		4, 4' DDT	8400	223	97.34	
		PCB-1260	1620	35	97.84	
Average Overall Performance						≥98.08

^aU indicates that the target compound was not detected at this level.

TABLE 6. SUMMARY OF BENCH-SCALE RESULTS OF CONTROLLED DEBRIS ANALYZED FOR LEAD (TRIAL 3)

	Sample ID Number	Contaminant	Pretreatment ($\mu\text{g}/100\text{ cm}^2$)	Posttreatment ($\mu\text{g}/100\text{ cm}^2$)	Percent Reduction	Average Performance
Metal	CM1-SP3-7/28	Lead	876	6.0	99.31	>99.06
	CM2-SP3-7/28	Lead	414	6.0	98.55	
	CM3-SP3-7/28	Lead	450	<3.0	>99.33	
Brick	CB1-SP3-7/28	Lead	508	<3.0	>99.41	
Concrete Block	CC1-SP3-7/28	Lead	414	<3.0	>99.27	
Plastic	CP1-SP3-7/28	Lead	446	<3.0	>99.33	
Average Performance on All Materials Tested						>98.08

manufacturer of BG-5 was 140°F; therefore, all the bench-scale and subsequent pilot-scale testings were conducted at 140°F.

4.2 Design, Fabrication, and Initial Testing of Pilot-Scale DWS

Based on the results obtained from bench-scale studies, a pilot-scale DWS was designed and constructed. The pilot-scale DWS consists of a 300-gallon spray tank; a 300-gallon wash tank; a surfactant holding tank; a rinse-water holding tank; an oil/water separator; and a solution treatment system comprised of a diatomaceous earth filter, a series of activated-carbon columns, and an ion exchange column. The system also includes other ancillary equipment, such as a heater for the 300-gallon spray tank (to heat the cleaning solution), a stirrer motor, a metal basket, particulate filters, and pumps (centrifugal and air-diaphragm). Figure 5 presents a schematic of the pilot-scale DWS.

The pilot-scale system was scaled up to be 15 times larger than the bench-scale system. Some of the engineering considerations involved in the design of the pilot-scale DWS were a scale-up factor, spray characteristics and distribution, spray angle and mean droplet diameter, flow and pressure balance, solution residence time, pump performance curves, materials and codes, and compatibility of construction materials with cleaning solution.

The pilot-scale system was assembled in a warehouse located in Cincinnati, Ohio. Several tests were conducted with pieces of oil/grease-coated metallic objects found in the warehouse. Surface-wipe samples were obtained before and after the debris was washed in the pilot-scale system and were analyzed for oil and grease. Table 7 summarizes the results of this testing. The warehouse testing also involved the optimization of test parameters such as duration of spray cycle, duration of wash cycle, and temperature of the cleaning solution. Based on the results and a visual inspection of the washed debris, the system was determined to be effective in removing oil and grease from the surface of these objects.

4.3 Field Demonstration of DWS at the Gray PCB Site

4.3.1 Site Description

The Gray PCB site is approximately 25 acres in size and located in Hopkinsville, Kentucky. From 1968 to 1987, a metal reclaiming facility was operated at this site. Operations included the open burning of electrical transformers to recover copper for resale. The soil where the transformers were burned is contaminated with lead and PCBs. On March 19, 1987, representatives from the Kentucky Department of Environmental Protection conducted an inspection at the site and observed the following conditions: 1) the facility was no longer in operation; 2) approximately 70 to 80 burned-out transformers were still on site, along with other large amounts of materials such as

Figure 5. Schematic of pilot-scale Debris Washing System.

**TABLE 7. RESULTS OF SURFACE WIPE SAMPLES ANALYZED FOR OIL
AND GREASE DURING WAREHOUSE TESTING**

	Sample ID Number	Pretreatment (mg/100 cm ²)	Posttreatment (mg/100 cm ²)	Percent Reduction	Avg. Percent Reduction
TRIAL 1	D1-1-10/5	142	47.1	66.7	70.2
	D2-1-10/5	431	41.2	90.4	
	D3-1-10/5	690	76.1	89.0	
	D4-1-10/5	94	76.2	19.0	
	D5-1-10/5	486	67.3	86.1	
TRIAL 2	D1-2-10/9	312	32.2	90.0	89.0
	D2-2-10/9	422	53.2	87.4	
	D3-2-10/9	283	39.1	86.2	
	D4-2-10/9	583	55.2	90.5	
	D5-2-10/9	2602	233.0	91.0	
TRIAL 3	D1-3-10/10	174	48.0	72.4	67.9
	D2-3-10/10	602	62.7	90.0	
	D3-3-10/10	286	55.4	80.6	
	D4-3-10/10	227	61.2	73.0	
	D5-3-10/10	73	55.9	23.3	

asbestos-covered pipes, automobiles, and miscellaneous scrap metal; and 3) multiple burn areas and two sink holes were noted.

4.3.2 Operating Parameters

During the demonstration, the operating parameters (such as surfactant concentration and temperature, flowrate and pressure during wash and spray cycle, and washing time) were established by ITEP based on the results of bench-scale testing and on the engineering design of the pilot-scale DWS.

The optimum cleaning solution concentration of 5 percent was recommended by the manufacturer of the surfactant solution. Prior to initiation of the process operation, a 5 percent surfactant solution was prepared in the detergent holding tank by adding 15 gallons of surfactant to 285 gallons of water. The mixture was agitated and heated to 140°F. An additional 300-gallon of 5 percent surfactant solution was also prepared in the oil/water separator. Since the same cleaning solution was reused during the entire demonstration, the solution mixture was recharged after every five batches of debris washing by adding approximately 5 to 10 gallons of fresh surfactant solution in both tanks (surfactant holding tank and oil/water separator). The amount of fresh surfactant added to these tanks was based upon the visual observation of the dirt in the solution mixture. Although this was a crude way to maintain the concentration of the cleaning solution, this method was found to be most practical during the demonstration.

The optimum solution temperature of 140°F was recommended by the manufacturer of surfacant solution. During the first batch of debris washing, it was noted that the temperature of the cleaning solution dropped 15° to 20°F due to heat loss in pipes, tank, and contact with the debris. To avoid this loss in temperature, for all the subsequent batches of debris washing, the cleaning solution was heated to about 160°F at the beginning of each new batch. Hence, by doing so, the temperature of the cleaning solution was maintained around 140°F during the washing process.

The flow rates and pressure of the cleaning solution during spray, wash and rinse cycles in the respective tanks remain within ± 10 percent of the desired values. The operating parameters matrix for the DWS demonstration are summarized in Table 8.

4.3.3 Process Operation of DWS During Field Testing

After the warehouse testing, the DWS was disassembled and loaded onto a 48-foot semitrailer. The system and ancillary equipment were transported to the Gray PCB site on December 5, 1989. The entire DWS was reassembled on a 25-ft x 24-ft concrete pad that had been poured on the site prior to the arrival of the equipment. A

temporary enclosure (approximately 25 ft high) was built on the concrete pad to enclose the DWS and to protect the equipment and the surfactant solution from rain and cold weather. During the course of this demonstration, the ambient temperature at the site ranged from -30°F to 50°F. Figures 6 and 7 show the assembled DWS at a hazardous waste site and the DWS enclosure at the Gray PCB site.

TABLE 8. OPERATING PARAMETERS MATRIX FOR DWS DEMONSTRATIONS

Operating parameters	Optimum value
1. Concentration of surfactant solution	5% (v/v)
2. Temperature of surfactant solution	140°F
3. Flow rate during spray/rinse cycles in spray tank	100 gpm
4. Pressure during spray/rinse cycles in spray tank	60 psi
5. Flow rate during wash cycle in wash tank	350 gpm
6. Pressure during wash cycle in wash tank	40 psi
7. Flow rate during solution recycle in oil/water separator	5 gpm
8. Time of spray cycle	60 min
9. Time of wash cycle	60 min
10. Time of rinse cycle	30 min

Before the cleaning process was begun, the transformer casings (ranging from 5 to 100 gallons in size) were cut in half with a circular metal cutting saw. A pretreatment sample was obtained from each half of the transformer casings by a surface-wipe technique (1).

The transformer halves were placed in the metal basket, and lowered into the spray tank. The tank was equipped with multiple water jets to blast loosely adhered contaminants and dirt from the debris. The cleaning solution (BG-5), which had a concentration of 5 percent and a temperature of 140°F, was pumped from the surfactant holding tank into the spray tank and then recycled back into the holding tank. After the spray cycle, the entire quantity of the cleaning solution in the surfactant holding tank was pumped into the wash tank. The basket was removed from the spray tank and transferred to the wash tank, where the debris was subjected to a high-turbulence wash. Each batch of debris was cleaned for 1 hour in the spray tank and 1 hour in the wash tank. During both spray and wash cycles, a portion of the cleaning solution was cycled through a closed-loop system in which the oil/PCB-contaminated cleaning

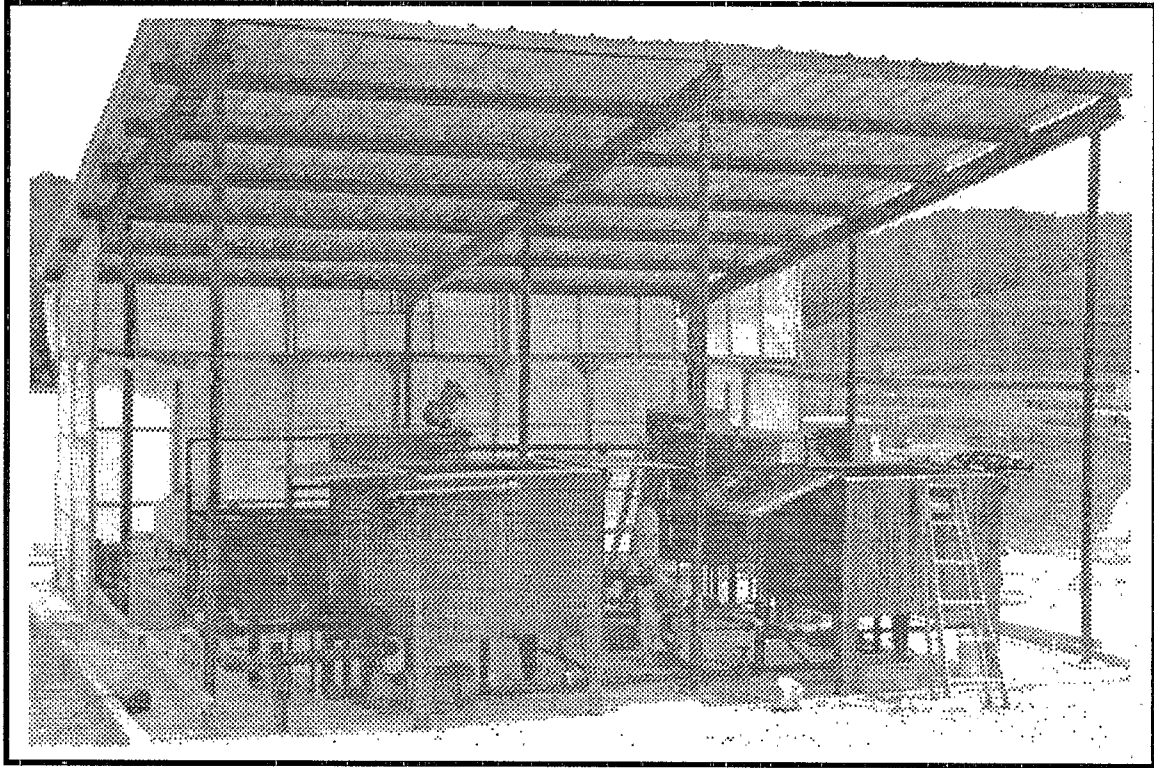


Figure 6. The assembled pilot-scale DWS at a hazardous waste site.



Figure 7. Enclosure for the pilot-scale DWS at the Gray site.

solution was passed through an oil/water separator and then through a diatomaceous earth filter. The clean solution was then recycled into the DWS. After the wash cycle, the basket containing the debris was returned to the spray tank, where it was rinsed with fresh water.

Upon completion of the cleaning process, posttreatment wipe samples were obtained from each of the transformer pieces to assess the post-decontamination levels of PCBs. The Quality Assurance Project Plan was strictly followed during sampling. The surface-wiping procedure was carried out as described in the Field Manual for Grid Sampling of PCB Spill Sites to Verify Cleanup (1). (A detailed description of surface-wipe sampling is presented in Section 5.)

4.3.4 Results of Field Demonstration

Table 9 summarizes the average concentrations of PCBs on the internal surfaces of the transformer casings before and after cleaning. The before-treatment concentration ranged from 0.1 to 98 $\mu\text{g}/100\text{ cm}^2$. The posttreatment analysis showed that all but seven of the cleaned transformer pieces had a PCB concentration of less than the acceptable level of 10 $\mu\text{g}/100\text{ cm}^2$. The seven transformer pieces with a concentration greater than the acceptable level were again washed in the DWS, and posttreatment samples were again obtained and analyzed. The PCB concentration in these seven samples after the second wash was below the detection limit of 0.1 $\mu\text{g}/100\text{ cm}^2$. The Quality Assurance/Quality Control of analyses performed on wipes is discussed in Section 5.

After all transformers at the site were treated, the surfactant solution and the rinse water were neutralized to a pH of around 8 with concentrated sulfuric acid. The neutralized surfactant solution and rinse water were treated in the water-treatment system by passing the solutions through a series of particulate filters, then through activated-carbon drums, and finally through an ion-exchange column. The treated water was stored in a 1000-gallon polyethylene tank pending analysis. The before- and after-treatment water samples were collected and analyzed for PCBs and selected metals (cadmium, copper, chromium, lead, nickel, and arsenic).

The PCB concentration in the water was reduced by the treatment system to below the detection limit. The concentrations of each of the six metals (except arsenic) were reduced to the allowable discharge levels set by the city of Hopkinsville. Table 10 summarizes the results of pre- and posttreatment water samples. Upon receipt of the analytical results of the water, the stored treated water was pumped into a plastic-covered, 10,000-yd³ pile of contaminated soil at the site through a 3/4-in. incision in the plastic covering at the top of the soil pile with a rubber hose. After all of the water was pumped into the contaminated soil, the hose was removed and the incision was covered with a duct tape.

TABLE 9. ANALYTICAL RESULTS OBTAINED DURING FIELD DEMONSTRATION OF DWS AT GRAY PCB SITE

Batch Number	Average PCB Concentration on Surfaces (µg/100 cm ²)				Average Percentage Removed
	Before Cleaning		After Cleaning		
	Average	Range	Average	Range	
1	19.7 (N ^a = 10)	<0.1 – 94.0	1.5 (N = 10)	<0.1 – 9.7	92
2	9.9 (N = 6)	4.8 – 17.0	1.5 (N = 6)	<0.1 – 4.7	85
3	6.6 (N = 4)	5.0 – 9.9	1.4 (N = 4)	<0.1 – 3.3	79
4	4.1 (N = 6)	<0.1 – 12.0	0.8 (N = 6)	<0.1 – 4.1	80
5	4.0 (N = 8)	<0.1 – 28.0	<0.1 (N = 8)	<0.1 – <0.1	>98
6	2.0 (N = 4)	<0.1 – 7.8	2.9 (N = 4)	<0.1 – 10.0	b
7	2.8 (N = 2)	1.4 – 4.3	3.9 (N = 2)	<0.1 – 7.7	b
8	23.5 (N = 5)	<0.1 – 70.0	1.3 (N = 5)	<0.1 – 3.8	94
9	8.3 (N = 4)	2.9 – 23.0	3.1 (N = 4)	1.5 – 4.9	63
10	5.2 (N = 4)	<0.1 – 9.7	1.9 (N = 4)	<0.1 – 2.8	63
11	9.4 (N = 4)	<0.1 – 17.0	3.0 (N = 4)	<0.1 – 9.5	67
12	48.8 (N = 4)	2.3 – 98.0	1.1 (N = 4)	<0.1 – 3.2	98
13	12.3 (N = 2)	9.6 – 15.0	5.1 (N = 2)	<0.1 – 10.0	59
14	16.7 (N = 2)	8.7 – 25.0	<0.1 (N = 2)	<0.1 – <0.1	>99
15	18.5 (N = 4)	8.1 – 27.0	<0.1 (N = 4)	<0.1 – <0.1	>99
16	11.3 (N = 2)	8.6 – 14.0	2.0 (N = 2)	1.5 – 2.5	82
17	24.8 (N = 4)	1.1 – 80.0	2.2 (N = 4)	<0.1 – 8.4	91
18	8.4 (N = 5)	<0.1 – 19.0	3.4 (N = 5)	<0.1 – 7.4	60
19	8.3 (N = 4)	<0.1 – 18.0	3.2 (N = 4)	<0.1 – 5.3	61
20	24.0 (N = 3)	13.0 – 45.0	3.3 (N = 3)	<0.1 – 9.8	86
21	18.6 (N = 8)	<0.1 – 44.0	0.4 (N = 8)	<0.1 – 2.1	98
22	25.0 (N = 4)	12.0 – 35.0	<0.1 (N = 4)	<0.1 – <0.1	>99
23	8.6 (N = 4)	1.5 – 18.0	<0.1 (N = 4)	<0.1 – <0.1	>99
24	6.8 (N = 8)	<0.1 – 31.0	0.3 (N = 8)	<0.1 – 1.4	96

^a N indicates the number of samples.

^b The distribution of PCB contamination on the surfaces of these transformers is obviously not uniform and therefore in some cases a meaningful comparison of post-treatment PCB levels with pre-treatment levels cannot be achieved.

**TABLE 10. ANALYTICAL RESULTS OF PROCESS WATER GENERATED
DURING GRAY PCB SITE DEMONSTRATION**

Sample ID Number		Analyses	Pretreatment	Posttreatment
Pretreatment	Posttreatment			
		<u>PCB (µg/L)</u>		
Pre-Ows-1/18 ^a	Post1-Det-1/18	Cleaning Solution	12/7.8 ^{a,b}	4.2 ^b
Pre-Det-1/18 ^a	Post3-Det-1/19	Cleaning Solution	5.4/5.4 ^{a,b}	1.3 ^b
Pre-Rins-1/18 ^a	Post2-Rins-1/19	Rinse Water	18/9.1 ^{a,b}	<1.0
		<u>METALS (µg/L)</u>		
		Cleaning Solution		
Pre-Owws-1/18 Pre-Det-1/18 ^a	Post2-Det-1/18	Copper	915/1027 ^a	723
		Lead	31160/15607 ^a	17400
		Cadmium	80/138 ^a	63
		Chromium	6150/4459 ^a	4860
		Arsenic	3870/3770 ^a	3720
		Rinse Water		
Pre-Rinse-1/18	Post1-Rins-1/19	Copper	614	103
		Lead	13210	3834
		Cadmium	45	<10
		Chromium	2056	882
		Arsenic	1940	3650

^a Duplicate analyses.

^b Estimated concentration due to matrix interferences.

Finally, the equipment was decontaminated with a high-pressure wash. The wash water generated during decontamination was collected and treated in the water treatment system. The system and the enclosure were then disassembled and loaded into the semitrailer for transport back to Cincinnati, Ohio.

During this site cleanup, 75 transformers (approximately 5000 lb) were washed in the DWS. A total of 1000 gallons of process water was used in the demonstration of the DWS. All of these transformers are now considered to be clean and can be sold to scrap metal dealers or to a smelter for reuse.

The copies of bench- and pilot-scale analytical data provided by the Hayden Environmental Group, Inc., are included in Volume II of this report.

4.4 Demonstration of DWS at Shaver's Farm Site

4.4.1 Site Description

The Shaver's Farm Drum Disposal Site consists of a 5-acre tract of land in Walker County, Georgia. Drums containing potentially hazardous wastes (Dicamba, benzonitrile, 2,4-D, and 2,4,5-T) and nonregulated latex-related wastes (butadiene and styrene) were allegedly buried on site by a construction and waste company in the early 1970's. At least three Potentially Responsible Parties (PRPs) contracted with this firm to transport and dispose of their manufacturing residues. The drum disposal area slopes nearly 25 degrees at the base of a small ridge and drains into a series of small sinkholes situated approximately 400 to 450 feet downgradient.

To date, EPA Region IV has excavated more than 4000 drums from one location on this site. The drums contained residues from the production of benzonitrile, a pesticide, and waste from the production of Dicamba, a herbicide. Prior to the initiation of excavation activities, no waste materials were evident at land surface. Available information suggests that there is at least one more location on site where drums are buried. It is estimated by EPA Region IV that the number of drums at this second location might range between 6,000 and 12,000. Figure 8 presents an aerial photograph of the site.

4.4.2 Bench-Scale Experiments and Results

Before final arrangements were made for the field demonstration of DWS at the Shaver's Farm Site in Georgia, a series of bench-scale experiments were conducted to determine the capability of the DWS to remove the primary contaminants (Dicamba and benzonitrile) found at the site.

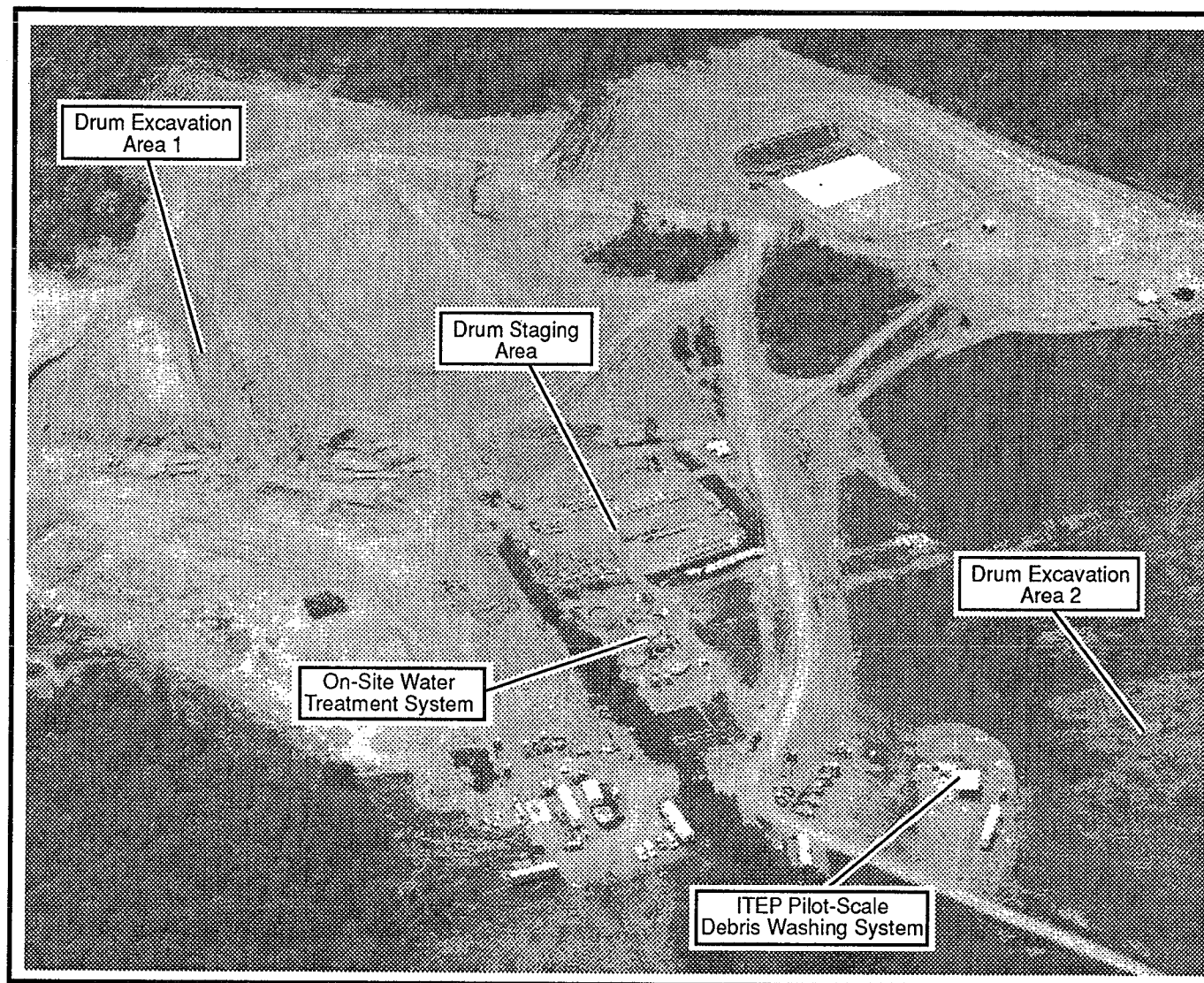


Figure 8. Aerial photograph of Shaver's Farm site.

A spike solution was prepared by adding a mixture of benzonitrile (12 g) and Dicamba (3 g) in acetone (81.5 g). To simulate the actual site scenario, a used, rusted 55-gallon drum was cut into small pieces and using a brush, each piece was evenly coated with the mixture of Dicamba and benzonitrile. The pieces were allowed to dry for about 12 hours. The spiked debris was cleaned in the bench-scale DWS. The surface-wipe samples taken before and after cleaning were obtained and analyzed for Dicamba and benzonitrile. The results of bench-scale experiments for benzonitrile and Dicamba are summarized in Tables 11 and 12, respectively.

The benzonitrile concentration was reduced from an initial average of 1660 $\mu\text{g}/100\text{ cm}^2$ (range of 54 to 4600 $\mu\text{g}/100\text{ cm}^2$) to below the detection limit (5 $\mu\text{g}/100\text{ cm}^2$). In the Dicamba analysis, an unknown interference caused all the analytical results (before and after treatment) to show Dicamba concentration of below the detection limit (0.3 $\mu\text{g}/100\text{ cm}^2$). Although Dicamba analytical results were not useful, the results for benzonitrile were certainly conclusive. As described below, the validity of the analytical method for Dicamba was verified by analyses of matrix spike/matrix spike duplicates.

The validity of the method used to analyze Dicamba and benzonitrile was verified by spiking four samples (two wipes and two metal strips) with a known quantity of Dicamba and two wipe samples with a known quantity of benzonitrile and submitting them to the laboratory for Dicamba and benzonitrile analyses. The results showed that, in the case of Dicamba, the recovery was 122 percent in the wipe samples and 104 percent in the metal strip samples; the benzonitrile recovery was 94 percent in the wipe sample. Tables 13 and 14 present the percent recoveries of Dicamba and benzonitrile.

The contaminated cleaning solution generated during bench-scale experiments was treated with activated carbon to reduce the contaminant concentrations to acceptable levels. Samples of wash solution before and after carbon treatment were analyzed for benzonitrile and Dicamba. The concentration of benzonitrile in the washing solution was reduced to below the detection limit (5 $\mu\text{g}/\text{L}$) from an initial concentration of 92 $\mu\text{g}/\text{L}$. The Dicamba concentration in the solution was reduced from 12,100 to 347 $\mu\text{g}/\text{L}$. The results of water analysis for benzonitrile and Dicamba are also shown in Tables 11 and 12, respectively.

4.4.3 Process Operation of DWS During Field Testing

After the bench-scale testing, the DWS and the ancillary equipment were transported to the Shaver's Farm site on a 48-foot semitrailer. The entire DWS was assembled on a 25-ft x 25-ft concrete pad that was poured on the site before the equipment arrived. The temporary enclosure used during the demonstration in Hopkinsville was used again at the Shaver's Farm site. The enclosure was built on the concrete pad to enclose the DWS and to protect the equipment and the surfactant solution from rain

TABLE 11. BENCH-SCALE RESULTS: ANALYSES FOR BENZONITRILE

	Sample ID Number		Benzonitrile	
	Pretreatment	Posttreatment	Pretreatment	Posttreatment
Wipes ($\mu\text{g}/100\text{cm}^2$)	P3-1-4/10	PT3-1-4/10	54	ND ^a (<5.0) ^b
	P3-2-4/10	PT3-2-4/10	92	ND (<5.0)
	P3-3-4/11	PT3-3-4/11	4600	ND (<5.0)
	P3-4-4/11	PT3-4-4/11	1900	ND (<5.0)
Process Water ($\mu\text{g}/\text{L}$)	PRE-W-4/13	POST-W-4/13	92	ND (<5.0)

^a Not detected at specified detection limit.

^b Numbers in parenthesis indicate the minimum detectable concentration of the analyte.

TABLE 12. BENCH-SCALE RESULTS: ANALYSES FOR DICAMBA

	Sample ID Number		Dicamba	
	Pretreatment	Posttreatment	Pretreatment	Posttreatment
Wipes ($\mu\text{g}/100\text{cm}^2$)	P1-1-4/10	PT1-1-4/10	<0.3	<0.3
	P2-1-4/10	PT2-1-4/10	<0.3	<0.3
	P1-2-4/10	PT1-2-4/11	<0.3	<0.3
	P2-2-4/10	PT2-2-4/11	<0.3	<0.3
	P1-3-4/11	PT1-3-4/11	<0.3	<0.3
	P2-3-4/11	PT2-3-4/11	<0.3	<0.3
	P1-4-4/11	PT1-4-4/11	<0.3	<0.3
	P2-4-4/11	PT2-4-4/11	<0.3	<0.3
Process Water ($\mu\text{g}/\text{L}$)	PRE-W-4/12	Post-W-4/12	12100	347

TABLE 13. METHOD VALIDATION: RESULTS OF DICAMBA ANALYSES OF GAUZE AND METAL STRIP SPIKED WITH DICAMBA

Sample ID	Sample Type	Quantity Spiked, μg	Analytical Results, μg		Average, μg	Recovery, %
			Sample 1	Sample 2 (duplicate)		
WL-1-6/14	Wipe	25,000	32,000	29,000	30,500	122
M-H-1-6/14	Metal Strips	60,000	63,000	62,000	62,500	104

TABLE 14. METHOD VALIDATION: RESULT OF BENZONITRILE ANALYSIS OF GAUZE SPIKED WITH BENZONITRILE

Sample ID	Sample Type	Quantity Spiked, μg	Analytical Results, μg		Average, μg	Recovery, %
			Sample 1	Sample 2 (duplicate)		
Wipe MS/MSD	Wipe	1,000	843	1,029	936	94

and hot weather. During the course of this demonstration, the ambient temperature ranged from 75 to 105 °F.

Before starting the cleaning process, the contaminated 55-gal drums were cut into four pieces with a circular metal cutting saw. A pretreatment sample was obtained from each of these four pieces by the surface-wipe technique. For corroboration of the results of the surface-wipe tests and to test whether contaminants were imbedded in the surfaces of the drums, a nibbler was used to take four or five metal strips (approximate size 6 cm x 3 cm) from one of the four drum pieces before the cleaning process was initiated.

The drum pieces were placed directly into the spray tank and the cleaning process was begun. The basket was not used because it was easier to place the drum pieces in the spray tank manually than to use the forklift and the basket. After completion of the cleaning process, posttreatment wipe samples were obtained from each of the drum pieces to assess the post-decontamination levels of herbicides and pesticides. Posttreatment metal strip samples were also taken from the same piece of drum where the pretreatment sample was obtained. The Quality Assurance Project Plan (QAPJP) was strictly followed during sampling.

Ten batches of drums were treated during this demonstration. After completion of the treatment, the cleaning solution and rinse water resulting from the decontamination of the debris in the DWS were treated in the water-treatment system. The treated water was stored in the 1000-gallon polyethylene tank pending analysis. In this demonstration, however, an ion exchange column was not used in the water-treatment system because the drums did not have any heavy metal contamination. The entire debris-cleaning procedure and the water-treatment process used at the Shaver's Farm site were similar to those used during the Gray PCB site demonstration, which were described in detail in Subsection 4.3.

The surface wipes, the metal strips, and the water samples obtained during the demonstration were sent to Radian Corporation (Austin, TX) for analyses. All these samples were analyzed for four semivolatiles (benzonitrile, 2,4-dichlorophenol, 2,6-dichlorophenol, and 1,2,4,-trichlorobenzene), 3-herbicides (Dicamba, 2,4-D, and 2,4,5-T), dioxins, and furans. Although the primary contaminants suspected on the Shaver's Farm site were benzonitrile and Dicamba, analyses of the remaining three semivolatiles, and two herbicides were performed at the request of EPA Headquarters in Washington, D.C.

For PCBs, the EPA has established a cleanup standard of 10 $\mu\text{g}/100\text{ cm}^2$ for surfaces. Because neither Dicamba nor benzonitrile is a frequently occurring contaminant on a hazardous waste site, the cleanup criteria for surfaces has not yet been established for these two contaminants. For the Shaver's Farm site, EPA has set a cleanup target concentration of 25 ppm in soil for both Dicamba and benzonitrile.

Based on the surface cleanup standards for PCBs and the soil standards for Dicamba and benzonitrile, it is assumed that when EPA sets up the cleanup criterion for surface concentration of Dicamba and benzonitrile at a later date, it will not exceed $10 \mu\text{g}/100 \text{ cm}^2$. Hence, during the field demonstration of DWS at Shaver's Farm, it was decided that all wipe samples to be analyzed for Dicamba and benzonitrile will have a detection limit of $5 \mu\text{g}/100 \text{ cm}^2$ or less.

4.4.4 Results of Field Demonstration

Table 15 summarizes the surface-wipe concentrations of semivolatiles on the internal surfaces of the drums before and after cleaning. The before-treatment concentration of benzonitrile ranged from 8 to $47,000 \mu\text{g}/100 \text{ cm}^2$ (average, $4556 \mu\text{g}/100 \text{ cm}^2$), and the posttreatment concentration ranged from below the detection limit to $117 \mu\text{g}/100 \text{ cm}^2$ (average $10 \mu\text{g}/100 \text{ cm}^2$). The pretreatment concentrations of 2,4-dichlorophenol in all the samples except two samples from Batch 2 were below the detection limit. The posttreatment concentrations of 2,4-dichlorophenol in all samples were below the detection limit. The remaining two semivolatiles (2,6-dichlorophenol and 1,2,4-trichlorobenzene) were not detected in any of the pre- or the posttreatment samples.

Table 16 summarizes the concentrations of semivolatiles in the metal strips. The pretreatment concentration of benzonitrile ranged from below the detection limit to $190 \mu\text{g}/\text{g}$ (average $42 \mu\text{g}/\text{g}$), and the posttreatment concentration ranged from below the detection limit to $0.89 \mu\text{g}/\text{g}$ (average $0.35 \mu\text{g}/\text{g}$). The concentrations of pre- and posttreatment samples analyzed for 2,4-dichlorophenol, 2,6-dichlorophenol, and 1,2,4-trichlorobenzene were below the detection limit with the exception of the sample from Batch 3 (analyzed for 2,4-dichlorophenol), which had a pretreatment concentration of $26 \mu\text{g}/\text{g}$ and a posttreatment concentration of $1.9 \mu\text{g}/\text{g}$.

Table 17 summarizes the results of surface-wipe samples analyzed for herbicides (Dicamba, 2,4-D, and 2,4,5-T). The pretreatment concentrations of Dicamba ranged from below the detection limit to $180 \mu\text{g}/100 \text{ cm}^2$ (average $23 \mu\text{g}/100 \text{ cm}^2$) and posttreatment concentration ranged from below the detection limit to $5.7 \mu\text{g}/100 \text{ cm}^2$ (average $1 \mu\text{g}/100 \text{ cm}^2$). The remaining two herbicides (2,4-D and 2,4,5-T) were not detected in any of the pre- or the posttreatment samples.

Table 18 presents the results of metal strip samples analyzed for Dicamba, 2,4-D, and 2,4,5-T. The pretreatment concentrations of Dicamba ranged from 0.057 to $82 \mu\text{g}/\text{g}$ (average $16 \mu\text{g}/\text{g}$), and the posttreatment concentrations ranged from below the detection limit to $13 \mu\text{g}/\text{g}$ (average $2 \mu\text{g}/\text{g}$). Concentrations of 2,4-D, and 2,4,5-T were not detected in any of the pre- or the posttreatment samples.

In addition to the semivolatiles and herbicides analyses, two metal strip samples were analyzed for dioxins and furans. Table 19 summarizes the results of these two

**TABLE 15. RESULTS OF SURFACE WIPE SAMPLES ANALYZED FOR BENZONITRILE,
2,4-DICHLOROPHENOL, 2,6-DICHLOROPHENOL, AND 1,2,4-TRICHLOROBENZENE
DURING FIELD DEMONSTRATION OF DWS AT SHAVER'S FARM SITE
($\mu\text{g}/100\text{cm}^2$)**

Batch Number	Sample ID Number		Benzonitrile		2,4-Dichlorophenol		2,6-Dichlorophenol		1,2,4-Trichlorobenzene	
	Pretreatment	Posttreatment	Pretreatment	Posttreatment	Pretreatment	Posttreatment	Pretreatment	Posttreatment	Pretreatment	Posttreatment
1	P1-1-7/17	PT1-1-7/17	180 ^a (50) ^b	ND ^c	ND (50)	ND	ND (50)	ND	ND (50)	ND
	P2-1-7/17	PT2-1-7/17	130 ^a (50)	ND	ND (50)	ND	ND (50)	ND	ND (50)	ND
2	P1-2-7/18	PT1-2-7/20	125	117	34	ND	ND	ND	ND	ND
	P2-2-7/18	PT2-2-7/20	90	7.8 ^a (5)	43	ND	ND	ND	ND	ND
3	P1-3-7/18	PT1-3-7/20	43	ND	ND	16 ^a (5)	ND	ND	ND	ND
	P2-3-7/18	PT2-3-7/20	28	ND	ND	14 ^a (5)	ND	ND	ND	ND
4	P1-4-7/19	PT1-4-7/20	4400	ND	NA ^d	NA	NA	NA	NA	NA
	P4-4-7/19	PT4-4-7/20	2700	ND	NA	NA	NA	NA	NA	NA
5	P1-5-7/19	PT1-5-7/20	47000	10 ^a (5)	ND	ND	ND	ND	ND	ND
	P2-5-7/19	PT2-5-7/20	22000	7.9 ^a (5)	ND	ND	ND	ND	ND	ND
6	P3-6-7/20	PT3-6-7/20	10 ^a (5)	ND	ND	ND	ND	ND	ND	ND
	P4-6-7/20	PT4-6-7/20	8 ^a (5)	ND	ND	ND	ND	ND	ND	ND
7	P1-7-8/2	PT1-7-8/2	200	ND	ND	ND	ND	ND	ND	ND
	P2-7-8/2	PT2-7-8/2	320	10 ^a (5)	ND	ND	ND	ND	ND	ND
8	P3-8-8/2	PT3-8-8/2	1400	28	ND	ND	ND	ND	ND	ND
9	P3-9-8/2	PT3-9-8/3	3000	ND	ND	ND	ND	ND	ND	ND
	P4-9-8/2	PT4-9-8/3	3500	7 ^a (5)	ND	ND	ND	ND	ND	ND
10	P1-10-8/2	PT1-10-8/3	22 ^a (5)	ND	ND	ND	ND	ND	ND	ND
	P2-10-8/2	PT2-10-8/3	1400	ND	ND	ND	ND	ND	ND	ND

^a Estimated result less than 5 times detection limit.

^b Numbers in parenthesis indicate the minimum detectable concentration of the analyte.

^c None detected in excess of the minimum detectable concentration of 5 $\mu\text{g}/100\text{cm}^2$ unless otherwise specified.

^d Not analyzed.

**TABLE 16. RESULTS OF METAL STRIP SAMPLES ANALYZED FOR BENZONITRILE,
2,4-DICHLOROPHENOL, 2,6-DICHLOROPHENOL, AND 1,2,4-TRICHLOROBENZENE
DURING FIELD DEMONSTRATION OF DWS AT SHAVER'S FARM SITE
(µg/g)**

Batch Number	Sample ID Number		Benzonitrile		2,4-Dichlorophenol		2,6-Dichlorophenol		1,2,4-Trichlorobenzene	
	Pretreatment	Posttreatment	Pretreatment	Posttreatment	Pretreatment	Posttreatment	Pretreatment	Posttreatment	Pretreatment	Posttreatment
1	P3-1-7/17	PT3-1-7/17	4.9	0.89	0.33 ^a (0.33) ^b	ND ^c (0.15)	ND (0.15)	ND (0.15)	ND (0.15)	ND (0.15)
2	P3-2-7/18	PT3-2-7/20	5.4 ^a (1.2)	0.31 ^a (0.12)	5.0 ^a (1.2)	ND (0.12)	ND (1.2)	ND (0.12)	ND (1.2)	ND (0.15)
3	P3-3-7/18	PT3-3-7/20	ND (0.15)	ND (0.10)	26	1.9	ND (0.15)	ND (0.10)	ND (0.15)	ND (0.10)
4	P5-4-7/19	PT5-4-7/20	ND	ND	NA ^d	NA	NA	NA	NA	NA
5	P5-5-7/19	PT5-5-7/20	190	0.8 ^a (0.22)	ND (0.28)	ND (0.22)	ND (0.28)	ND (0.22)	ND (0.28)	ND (0.22)
6	P5-6-7/20	PT5-6-7/20	1.0	ND (0.08)	ND (0.085)	ND (0.08)	ND (0.085)	ND (0.08)	0.13 ^a (0.085)	ND (0.080)
7	P5-7-8/2	PT5-7-8/2	140	0.28 ^a (0.16)	ND (0.19)	ND (0.16)	ND (0.19)	ND (0.16)	ND (0.19)	ND (0.16)
8	P5-8-8/2	PT5-8-8/2	16	0.41 ^a (0.15)	ND (0.14)	ND (0.15)	ND (0.14)	ND (0.15)	ND (0.14)	ND (0.15)
9	P5-9-8/2	PT5-9-8/3	61	0.18 ^a (0.17)	ND (0.10)	ND (0.17)	ND (0.10)	ND (0.17)	ND (0.10)	ND (0.17)
10	P5-10-8/2	PT5-10-8/3	ND (1.2)	ND (0.11)	ND (0.12)	ND (0.11)	ND (0.12)	ND (0.11)	ND (0.12)	ND (0.11)

^a Estimated result less than 5 times detection limit.

^b Numbers in parenthesis indicate the minimum detectable concentration of the analyte.

^c None detected at specified detection limit.

^d Not analyzed.

**TABLE 17. RESULTS OF SURFACE WIPE SAMPLES ANALYZED FOR DICAMBA,
2,4-D, AND 2,4,5-T DURING FIELD DEMONSTRATION OF DWS
AT SHAVER'S FARM SITE
($\mu\text{g}/100\text{cm}^2$)**

Batch Number	Sample ID Number		Dicamba		2,4-D		2,4,5-T	
	Pretreatment	Posttreatment	Pretreatment	Posttreatment	Pretreatment	Posttreatment	Pretreatment	Posttreatment
4	P2-4-7/19	PT2-4-7/20	1.9	0.63 ^a (0.27) ^b	ND ^c	ND	ND	ND
	P3-4-7/19	PT3-4-7/20	3.4	ND	NA ^d	NA	NA	NA
5	P3-5-7/19	PT3-5-7/20	ND	ND	ND	ND	ND	ND
	P4-5-7/19	PT4-5-7/20	ND	2.6	ND	ND	ND	ND
6	P1-6-7/20	PT1-6-7/20	ND (2.7)	ND	ND (12)	ND	ND (2.0)	ND
	P2-6-7/20	PT2-6-7/20	ND (2.7)	ND (2.7)	ND (12)	ND (12)	ND (2.0)	ND (2.0)
7	P3-7-8/2	PT3-7-8/2	7.3 ^a (2.7)	1.8	ND	ND	ND	ND
	P4-7-8/2	PT4-7-8/2	15	2.3	ND (12)	ND	ND (2.0)	ND
8	P1-8-8/2	PT1-8-8/2	55	5.7 ^a (2.7)	ND (12)	ND (12)	ND (2.0)	ND (2.0)
	P2-8-8/2	PT2-8-8/2	13	0.62 ^a (0.27)	ND	ND	ND	ND
9	P1-9-8/2	PT1-9-8/3	1.7	0.63 ^a (0.27)	ND	ND	ND	ND
	P2-9-8/2	PT2-9-8/3	ND (2.7)	ND	ND (12)	ND	ND (2.0)	ND
10	P3-10-8/2	PT3-10-8/3	41	0.30 ^a (0.27)	ND (12)	ND	ND (2.0)	ND
	P4-10-8/2	PT4-10-8/3	180	0.34 ^a (0.27)	ND (12)	ND	ND (2.0)	ND

^a Estimated result less than 5 times detection limit.

^b Numbers in parenthesis indicate the minimum detectable concentration of the analyte.

^c None detected in excess of minimum detectable concentration of Dicamba at 0.27; 2,4-D at 1.2; and 2,4,5-T at 0.20 unless otherwise specified.

^d Not analyzed.

**TABLE 18. RESULTS OF METAL STRIP SAMPLES ANALYZED FOR DICAMBA, 2,4-D, AND 2,4,5-T
DURING FIELD DEMONSTRATION OF DWS AT SHAVER'S FARM SITE
($\mu\text{g/g}$)**

Batch Number	Sample ID Number		Dicamba		2,4-D		2,4,5-T	
	Pretreatment	Posttreatment	Pretreatment	Posttreatment	Pretreatment	Posttreatment	Pretreatment	Posttreatment
2	1P3-2-7/18	1PT3-2-7/20	0.37	0.030 ^a (0.0097) ^b	ND ^c (0.34)	ND (0.043)	ND (0.0056)	ND (0.0072)
3	1P3-3-7/18	1PT3-3-7/20	82	13	ND (0.36)	ND (0.38)	ND (0.060)	ND (0.064)
4	1P5-4-7/19	1PT5-4-7/20	0.057	0.020 ^a (0.0065)	ND (0.029)	ND (0.029)	ND (0.0048)	ND (0.0048)
6	1P5-6-7/20	1PT5-6-7/20	25	ND (0.059)	ND (0.26)	ND (0.26)	ND (0.044)	ND (0.044)
7	1P5-7-8/2	1PT5-7-8/2	2.3	0.023 ^a (0.062)	ND (0.50)	ND (0.28)	ND (0.084)	ND (0.046)
9	1P5-9-8/2	1PT5-9-8/3	2.3	0.11	ND (0.32)	ND (0.040)	ND (0.054)	ND (0.0066)
10	1P5-10-8/2	1PT5-10-8/3	0.40	0.017 ^a (0.0062)	ND (0.24)	ND (0.028)	ND (0.040)	ND (0.0046)

^a Estimated result less than 5 times detection limit.

^b Numbers in parenthesis indicate the minimum detectable concentration of the analyte.

^c None detected at specified detection limit.

**TABLE 19. RESULTS OF METAL STRIP SAMPLES ANALYZED FOR
DIOXINS AND FURANS DURING FIELD DEMONSTRATION
OF DWS AT SHAVER'S FARM SITE
(ng/g)**

Analyses	Sample ID: 1P3-1-7/17		Sample ID: 1P5-8-8/2	
	Pretreatment	Posttreatment	Pretreatment	Posttreatment
HpCDD	1.2	ND ^a (0.30) ^b	ND (0.22)	ND (0.31)
HpCDF	ND (0.16)	ND (0.23)	ND (0.17)	ND (0.23)
HxCDD	2.8	ND (0.24)	ND (0.18)	0.71 ^c (0.27)
HxCDF	ND (0.12)	ND (0.11)	ND (0.11)	ND (0.17)
OCDD	4.1	ND (0.66)	1.6 ^c (0.45)	5.5
OCDF	ND (0.19)	ND (0.36)	ND (0.22)	ND (0.34)
PeCDD	ND (0.11)	ND (0.14)	ND (0.10)	ND (0.16)
PeCDF	ND (0.066)	ND (0.085)	ND (0.065)	ND (0.092)
TCDD	ND (0.10)	ND (0.13)	ND (0.10)	ND (0.15)
2,3,7,8-TCDD	ND (0.10)	ND (0.13)	ND (0.10)	ND (0.15)
TCDF	ND (0.062)	ND (0.079)	ND (0.065)	ND (0.089)

^a Not detected at specified detection limit.

^b Numbers in parenthesis indicate the minimum detectable concentration of the analyte.

^c Estimated result less than 5 times detection limit.

samples. In the first sample, only three congeners were present in the pretreatment sample. The pretreatment concentrations of these three congeners (HpCDD, HxCDD, and OCDD) were 1.2, 2.8, and 4.1 ng/g, respectively. The posttreatment concentrations of all congeners were below the detection limit. In the second sample, only OCDD was identified. The pretreatment concentration was 1.6 ng/g (estimated value), and the posttreatment concentration was 5.5 ng/g. In the case of Sample 2, the OCDD concentration apparently increased after the cleaning process, probably because of interferences during analysis.

The pre- and posttreatment samples of process water were analyzed for benzonitrile, 2,4-dichlorophenol, 2,6-dichlorophenol, 1,2,4-trichlorobenzene, Dicamba, 2,4-D, 2,4,5-T, dioxins, and furans. The concentrations of all these compounds except benzonitrile and dicamba were below detection limit in pre- and posttreatment samples. The concentration of benzonitrile in the pretreatment water samples was 250 and 400 $\mu\text{g/L}$ (analyzed in duplicate), and the posttreatment concentration was below the detection limit. The concentration of Dicamba in the pretreatment samples was 6800 and 6500 $\mu\text{g/L}$ (analyzed in duplicate), and the posttreatment concentration was ≤ 630 $\mu\text{g/L}$ (estimated value). Table 20 summarizes the results of process water samples.

The quality assurance/quality control of analyses performed on wipes, metal strips, and water samples is discussed in Section 5. Copies of the bench-scale and pilot-scale analytical data provided by the analytical laboratories are included in Volume II of this report.

Because the concentration of Dicamba in the posttreatment water could not be accurately measured, the treated water stored in the polyethylene holding tank was pumped into an onsite water-treatment system for further treatment before its discharge into a nearby creek.

Finally, the equipment was decontaminated with a high-pressure wash. The wash water generated during this decontamination was also collected and pumped into the onsite water-treatment system. The system and the enclosure were disassembled and loaded into the semitrailer for transport back to Cincinnati, Ohio.

**TABLE 20. ANALYTICAL RESULTS FOR PROCESS WATER GENERATED
DURING SHAVER'S FARM SITE DEMONSTRATION**

Sample ID Number		Analyses	Pretreatment	Posttreatment
Pretreatment	Posttreatment			
Det-Pre-8/3 Ows-Pre-8/3 ^a	Post-8/3	<u>Semi-Volatiles (µg/L)</u>		
		Benzonitrile	250/400 ^a	ND ^b (4.8) ^c
		2,4-Dichlorophenol	ND (4.8)	ND (4.8)
		2,6-Dichlorophenol	ND (4.8)	ND (4.8)
		1,2,4-Trichlorobenzene	ND (4.8)	ND (4.8)
Det-Pre-8/3 Ows-Pre-8/3 ^a	Post-8/3	<u>Herbicides (µg/L)</u>		
		Dicamba	6800/6500 ^a	630 ^d (260)
		2,4-D	ND ^b (1100)	ND (1100)
		2,4,5-T	ND (190)	ND (190)
Det-Pre-8/3	Post-8/3	<u>Dioxins/Furans (ng/L)</u>		
		HpCDD	ND ^b (10)	ND (4.8)
		HpCDF	ND (7.4)	ND (3.0)
		HxCDD	ND (8.4)	14 ^d (3.6)
		HxCDF	ND (5.0)	ND (2.0)
		OCDD	ND (23)	ND (11)
		OCDF	ND (10)	ND (5.7)
		PeCDD	ND (4.8)	ND (2.1)
		PeCDF	ND (3.0)	ND (1.3)
		TCDD	ND (4.8)	ND (2.1)
		2,3,7,8-TCDD	ND (4.8)	ND (2.1)
		TCDF	ND (2.9)	ND (1.2)

^a Duplicate analyses.

^b Not detected at specified detection limit.

^c Numbers in parenthesis indicate the minimum detectable concentration of the analyte.

^d Estimated result less than 5 times detection limit.

SECTION 5

QUALITY ASSURANCE/QUALITY CONTROL ANALYSES

The objective of this study was to design, develop, and evaluate a pilot-scale debris decontamination system. The data generated consisted of the results of chemical analyses performed during the project. All data and observations were recorded in permanent laboratory notebooks. This section presents the quality assurance/quality control (QA/QC) analyses performed on surface-wipe, metal, and water samples taken during the field demonstrations of the DWS.

5.1 Demonstration of DWS at Gray PCB Site

During the field demonstration of the DWS at the Gray PCB site, surface-wipe samples of pieces of metallic debris (transformer casings) were taken to ascertain the extent of PCB contamination on the surfaces of the debris. The contamination of a piece of debris may vary at different points on its surface, and pieces of debris used in the demonstration probably will exhibit varying degrees of contamination. Many factors can contribute to these varying degrees of contamination on a particular piece of debris, such as the concentration of contaminants in the soil, the contact time (if the piece was buried), the extent of surface oxidation, the porosity of the material, and exposure to the elements. As a way of minimizing these differences, pretreatment and posttreatment samples were taken directly adjacent to each other, but not at the same location. Because the initial wiping of the surface of a sample removes the contamination, if one were to wipe the same surface after cleaning, the result obtained would be biased low. Results were reported as the relative percent difference (RPD) between the pretreatment and posttreatment levels of total PCBs. The PCB concentration in the cleaning solution and rinse water after filtration/sorbent treatment indicated the ultimate method at disposal.

5.2 Sampling and Analysis

5.2.1 Wipe Sampling Theory

Under the Toxic Substance Control Act (TSCA), the EPA has established the PCB Spill Cleanup Policy for releases of materials containing PCBs at concentrations of 50 parts per million (ppm) or greater (2). The Policy states that high-contact solid

surfaces should be cleaned to a level of $10 \mu\text{g}/100 \text{ cm}^2$. The method of determining contamination on hard surfaces such as metal, wood, concrete, plastic, and glass is to analyze surface-wipe samples (3).

Procedures for standard wipe tests vary, but generally include wiping a specific area with an absorbent tissue or cotton gauze swatch that has been wetted with a solvent (hexane). The EPA policy recommends that a standard-size template (10 cm by 10 cm) be used to delineate the area of cleaning. The gauze pad or glass wool of known size should be saturated with hexane. The hexane-prepared wipe is stored in a sealed glass vial both before and after sample collection. The before and after surface-wipe samples will indicate the efficiency of the cleaning process on the basis of the percentage of PCBs removed from the debris surfaces. The EPA also requires the collection and testing of field blanks.

The PCB concentrations in surface-wipe samples can be determined by first extracting the wipe samples in accordance with Method 3550 and then quantitating them in accordance with Method 8080, which involves injecting the sample extract into a gas chromatograph (GC) set at the operating conditions specified in the Method and equipped with an electron-capture detector (4). The result is reported in micrograms PCB per wipe or micrograms per 100 cm^2 .

A QA plan for wipe samples is outlined in an EPA report (3). This plan includes chain of custody, field blanks, check samples, replicate samples, spiked samples, etc.

Analyzing surface-wipe samples provides a systematic method of determining the extent of PCB contamination or the effectiveness of the cleanup operation. The method produces varying results, depending on the sample collector, the porosity of the surfaces, and the details of the sampling procedure. This method, however, is relatively quick and inexpensive, and it is easily adapted to statistical sampling. It currently affords the most reasonable way to sample surfaces for PCB contamination.

5.2.2 Surface-Wipe Sampling Procedures

The surface-wipe samples were obtained in accordance with procedures outlined in the Field Manual for Grid Sampling of PCB Sites to Verify Cleanup (1). The following procedure was used to take surface-wipe samples from pieces of metallic debris.

A wide-mouth jar was first filled with 3-in. by 3-in. cotton gauze pads and then filled with hexane, which effectively saturated the pads. With gloved hands, the sampler obtained the sample by thoroughly wiping a 100-cm^2 area (delineated by a template) with the moistened gauze pad, moving it from left to right and then from top to bottom of the metallic debris surface. The sampler then folded the gauze pad with the sampled side inward and placed it in the sample bottle, which was then capped, labeled, affixed with a yellow TSCA PCB mark, and placed in an ice chest (to keep the

sample at 4°C). The sample collection data were entered into the field log book and on the Chain-of-Custody Form. The template was then thoroughly rinsed with hexane and wiped with a disposable wiping cloth. The rubber gloves worn by the sampler when taking the wipe samples and the wiping cloth were discarded in a plastic bag for disposal as PCB-contaminated materials. One field blank and two additional wipes [one for the matrix spike (MS) and one for the matrix spike duplicate (MSD)] were also placed in sample containers at this time.

5.2.3 Process Water Sampling

Upon completion of the treatment of all transformers at the site, the contaminated process water was treated in the treatment system. Prior to the treatment, two baseline samples were collected from each tank, composited, and analyzed for PCBs and selected metals. Subsequently, the contaminated water was treated, and two samples (total of 1 gallon each) of treated water was collected during the process. One of these two posttreatment samples was collected earlier during the treatment process at 15-minute intervals and composited until about the first half of the total process water was treated. The second posttreatment water sample was then collected of the remaining process water in the similar fashion. Both of these samples were analyzed for PCBs and selected metals.

5.2.4 Sample Containers

The surface-wipe samples were collected in a 4-oz clean glass jar with a Teflon-lined lid. The process water samples were collected in a 1-gallon amber glass bottle equipped with a Teflon-lined cap. Plastic containers or lids were not used in the storage of samples because the samples could become contaminated by the phthalate esters and other hydrocarbons within the plastic.

5.3 Summary of QA/QC Procedures Used by Hayden Environmental Group

The Quality Assurance Objectives listed in the QAPjP for the project are shown in Table 21. The QA data corresponding to each of the objectives shown in Table 21 are discussed in the following subsections.

5.3.1 Polychlorinated Biphenyls (PCBs)

The analytical method used to quantitate PCBs in surface-wipe samples was taken from the third edition of EPA's Test Methods for Evaluating Solid Waste (4). The procedures outlined in SW-846 Method 3550 were used to extract PCBs from surface wipes, and EPA Method 8080 was used to quantitate the PCBs in these extracts.

**TABLE 21. QA OBJECTIVES FOR PRECISION, ACCURACY, COMPLETENESS, AND
METHOD DETECTION LIMIT (GRAY PCB SITE DEMONSTRATION)**

Critical Measurement	Matrix Type	Method Reference	Measured Units	MDL	Precision ^a	Accuracy ^b	Completeness, %	Reference
Sonication extraction	Surface wipes	SW-846 Method 3550	—	—	—	—	90	4
PCB concentration	Surface wipes, water	SW-846 Method 8080	Wipes: $\mu\text{g}/100\text{cm}^2$	5 $\mu\text{g}/100\text{cm}^2$	± 30	30 - 130	80	4
			Water: $\mu\text{g}/\text{ml}$	5 $\mu\text{g}/\text{ml}$	± 30	30 - 130	80	4
Liquid-liquid extraction	Process water	SW-846 Method 3510	—	—	—	—	90	4
Oil and grease	Surface wipes	SW-846 Method 9070	Milligrams	0.2 mg	± 20	75 - 125	80	4

^a As Relative Percent Difference (RPD) of matrix spike duplicates

^b As Percent Recovery Range of laboratory matrix spikes unless otherwise indicated

5.3.1.1 Analyte Calibration

A five-point calibration was performed by using standard concentrations from 0.25 to 0.75 ng/ μ L to generate response factors. These calibration-check standards were reanalyzed after every 20 samples to verify that the response factor remained within ± 15 percent of that generated from an average of the five-point standards.

5.3.1.2 Accuracy

The target accuracy range was 30 to 130 percent, as determined by the percentage of recovery of laboratory matrix spikes. The QAPjP states that matrix-spiked samples are to be analyzed at a frequency of one for every 20 samples of each matrix type analyzed. The surface-wipe samples were spiked with Aroclor 1242 in duplicate. Summaries of spike recoveries for surface-wipe samples are shown in Table A-1 in Appendix A. The mean percent recovery falls within the targeted limits of 30 to 130 percent.

5.3.1.3 Precision

The precision of the analytical method was evaluated by calculating the RPD for the percentage recoveries of MS and MSD samples. The calculated RPD values ranged from 3 to 29 percent. All values were within the 30 percent RPD required. The precision results are summarized in Table A-1 in Appendix A.

5.3.1.4 Blanks

The field blanks from each batch were extracted and analyzed by Method 8080 in the same manner as the other surface-wipe samples to check for background contamination. The results of all the field blanks were below the detection limit of $<0.1 \mu\text{g}/100 \text{ cm}^2$. Method blanks were also analyzed to check for any contamination. During the analysis of the method blank, a purified solid matrix is carried through the entire analytical scheme (extraction, concentration, and analysis). No contamination was observed in any of the method blanks. A summary of field and method blanks results is presented in Table A-2 in Appendix A.

5.3.2 Metals

The analytical methods used to quantitate metals (copper, lead, cadmium, chromium, and arsenic) in process water samples were taken from the third edition of EPA's Test Methods for Evaluating Solid Waste (4) and are summarized in Table 22.

**TABLE 22. METHODS USED TO QUANTITATE SELECTED METHODS
(GRAY PCB SITE DEMONSTRATION)**

Measurement	Matrix type	Method reference	Measured units
Copper	Water	SW-846-7210 Method 220.1	$\mu\text{g/L}$
Lead	Water	SW-846-7420 Method 239.1	$\mu\text{g/L}$
Cadmium	Water	SW-846-7130 Method 213.1	$\mu\text{g/L}$
Chromium	Water	SW-846-7190 Method 218.1	$\mu\text{g/L}$
Arsenic	Water	SW-846-7060 Method 206.2	$\mu\text{g/L}$

5.4 Demonstration of DWS at Shaver's Farm Site

During the field demonstration of the DWS at the Shaver's Farm site, surface-wipe samples were taken from pieces of metallic debris (55-gal metallic drums) to ascertain the extent of benzonitrile and Dicamba contamination on the surfaces of the debris. Subsequent to cleaning the debris in the DWS, surface-wipe samples were again taken and the efficacy of the cleaning process was judged on the basis of whether the benzonitrile and Dicamba on the debris surfaces had been reduced to the "acceptable level" of $5 \mu\text{g}/100 \text{ cm}^2$ in surface wipes and $5 \mu\text{g/g}$ in metal strips.

In addition to surface-wipe sampling, pieces of the metallic strips were also obtained before and after treatment and extracted to determine the amount of Dicamba, benzonitrile, and (in two cases) PCDD and PCDF present in metallic strips.

The concentrations of Dicamba and benzonitrile in the cleaning solution and rinse water after filtration/sorbent treatment will indicate the ultimate method of disposal.

5.5 Sampling and Analysis

5.5.1 Wipe Sampling Theory

The basic theory of surface-wipe sampling was explained earlier in Subsection 5.1.1.1. Dicamba and benzonitrile on the surface of metallic debris were sampled by the surface-wipe technique. The concentrations of these contaminants were then

quantitated by first extracting the wipe samples by Method 3540 (4). During the Soxhlet extraction procedure, the entire gauze wipe was put into the extraction solvent. The Dicamba concentration was then quantitated by Method 8150, which involves injecting an aliquot of the gauze extract into a GC set at the operating conditions specified in the method and equipped with an electron-capture detector (4). The benzonitrile concentration was quantitated in accordance with Method 8270, which involves injecting an aliquot of the gauze extract into a gas chromatograph/mass spectrometer (GC/MS) set at the specified operating conditions (4). The analytical results are reported in micrograms of contaminant per wipe or micrograms of contaminant per 100 cm².

5.5.2 Wipe Sampling Procedure

The following procedure was used to collect surface-wipe samples from pieces of debris. A wide-mouth jar was filled with a 1:1 mixture of pesticide-grade hexane and acetone for effective saturation of the pads. This jar was kept tightly sealed until the samples were taken. During sampling, individual gauze pads were removed with stainless steel forceps and handed to the sampler, who compressed each pad with his gloved hand to remove excess solvent. The solvent-soaked gauze pad was then used to thoroughly wipe a 100-cm² area of the metallic debris surface (delineated by a template), from left to right and then from the top to bottom to obtain the sample. The sampler then folded the gauze pad with the sampled side inward and placed it in a 4-oz glass jar, which was then capped, labeled, and placed in an ice chest (to keep the sample at 4°C). The sample collection data were entered into the field log book and on the chain-of-custody form. The template was thoroughly rinsed with solvent and wiped with a disposable wiping cloth. The sampler's rubber gloves and the wiping cloth were then discarded in a plastic bag used for disposal of hazardous-waste-contaminated materials.

When debris was covered with scale or caked-on deposits, the pretreatment sampling was performed on an area that was free of surface deposits. Multiple surface wipes were obtained to ensure that all material in the wipe area was removed from the surface and transferred to the sample container.

5.5.3 Sampling of Metal Matrix

To corroborate the results of the surface-wipe tests and to demonstrate whether contaminants were imbedded in the surfaces of metallic debris, the sampler obtained one pretreatment and one posttreatment sample from each batch in the following manner. A partner saw was used to remove a 10-in. by 10-in. piece of metal from the debris item (e.g., a 55-gallon drum that had been removed from a burial trench) and from the approximate center of the 10-in. by 10-in. piece (to avoid areas where heating resulting from the sawing process may have dislodged the contaminants); approximately 100 g of metal strips were removed by power shears (no heat generation).

These metal strips were immediately placed in a tightly capped jar, which was placed in a 4°C sample cooler for shipment.

5.5.4 Process Water Sampling

Upon completion of the treatment of all batches of drums at the site, the contaminated process water was treated in the treatment system. Prior to the treatment, two baseline samples were collected from each tank, composited, and analyzed for Dicamba, benzonitrile, dioxins, and furans. During the subsequent treatment of the contaminated water, two 1-gal samples of treated water were collected. One of these two posttreatment samples was collected at 15-min intervals early in the treatment process and composited. This composite sample represented roughly the first half of the process water being treated. The second posttreatment water sample was collected in a similar manner and represented the remaining half of the process water. Both of these samples were analyzed for Dicamba, benzonitrile, dioxins, and furans.

5.5.5 Sample Containers

The surface-wipe samples and metal strip samples were collected in a 4-oz clean glass jar with a Teflon-lined lid. The process water samples were collected in a 1-gallon amber glass bottle with a Teflon-lined cap. Plastic containers or lids were not used to store samples because the samples could become contaminated by the phthalate esters and other hydrocarbons within the plastic.

5.6 Summary of QA/QC Procedures Used by Radian Corporation

The analytical method used to quantitate organics in surface-wipe, metal strip, and process water samples was taken from the third edition of EPA's Test Methods for Evaluating Solid Waste (4). Radian Corporation used the procedures outlined in SW-846 Methods 3540 and 3510 to extract organics from surface wipes/metal strips and process water, respectively. Method 3540 is a Soxhlet extraction process, whereas Method 3510 is a liquid-to-liquid extraction process. Methods 8150, 8270, and 8280 from this same document were used to quantitate herbicides, semivolatiles, and PCDD/PCDF, respectively, from extracts of surface wipes/metal strips and process water.

The QA objectives listed in the QAPjP are presented in Table 23. When the QAPjP for this project was written, the critical measurements were Dicamba and benzonitrile. During the field demonstration of DWS at the Shaver's Farm site, EPA Headquarters in Washington, D.C., requested that analyses also be made for 2,4-dichlorophenol, 2,6-dichlorophenol, 1,2,4-trichlorobenzene, 2,4-D, and 2,4,5-T. Because it was too late in the project to include these compounds in the QAPjP, Table 23 shows only Dicamba and benzonitrile as the critical measurements. The QA data corresponding

**TABLE 23. QA OBJECTIVES FOR PRECISION, ACCURACY, COMPLETENESS, AND
METHOD DETECTION LIMIT (SHAVER'S FARM SITE DEMONSTRATION)**

Critical Measurement	Matrix Type	Method Reference	Measured Units	MDL	Precision ^a	Accuracy ^b	Completeness, %	Reference
Dicamba	Surface wipes, metal strips	Extraction: SW-846 Method 3540	—	—	—	—		4
		Analysis: SW-846 Method 8150	Wipes: $\mu\text{g}/100\text{cm}^2$ Strips: $\mu\text{g}/\text{g}$	$5 \mu\text{g}/100\text{cm}^2$ $5 \mu\text{g}/\text{g}$	± 50 ± 50	30 - 130 30 - 130	80 80	4
Benzonitrile	Surface wipes, metal strips	Extraction: SW-846 Method 3540						
		Analysis: SW-846 Method 8270	Wipes: $\mu\text{g}/100\text{cm}^2$ Strips: $\mu\text{g}/\text{g}$	$5 \mu\text{g}/100\text{cm}^2$ $5 \mu\text{g}/\text{g}$	± 50 ± 50	30 - 130 30 - 130	80 80	4 4
	Process water	Extraction: SW-846 Method 3510 Analysis: SW-846 Method 8150	Water: $\mu\text{g}/\text{L}$	$1 \mu\text{g}/\text{L}$	± 30	30 - 130	90	4

^a As Relative Percent Difference (RPD) of matrix spike duplicates

^b As Percent Recovery Range of laboratory matrix spikes unless otherwise indicated

to each of the objectives shown in Table 23 are discussed in the following subsections.

5.6.1 Semivolatiles

All semivolatile (benzonitrile, 2,4-dichlorophenol, 2,6-dichlorophenol, and 1,2,4-trichlorobenzene) extracts were analyzed using the procedures outlined in Method 8270 of SW-846 (4).

5.6.1.1 Analyte Calibration

A five-point calibration was performed with Best Demonstrated Available Technology (BDAT) standards at concentrations of 5, 10, 50, 100, and 200 $\mu\text{g/L}$ for benzonitrile analysis and 20, 50, 80, 120, and 160 $\mu\text{g/L}$ for 2,4-dichlorophenol, 2,6-dichlorophenol, and 1,2,4-trichlorobenzene. Specific ion-response factors for the calibration check compounds were verified to have less than 30 percent Relative Standard Deviation (RSD) over the range calibrated. These calibration check compounds were reanalyzed every 12 hours to verify that the response factor remained within ± 30 percent of that generated from an average of the five-point standard.

5.6.1.2 Accuracy

Accuracy was to be within the range of 30 to 130 percent, as determined by the percentage recovery of the surrogates spiked into each sample. Surrogate recoveries for pre- and posttreatment wipe samples are summarized in Tables B-1 and B-2 respectively in Appendix B. The mean percentage recovery of all surrogates falls within the limits of 30 to 130 percent, as specified in the QAPjP.

Surrogate recoveries for pre- and posttreatment metal strip samples are summarized in Tables B-3 and B-4 respectively in Appendix B. The mean percentage recovery of all surrogates falls within the limits of 30 to 130 percent specified in the QAPjP.

5.6.1.3 Precision

Precision of the analytical method was evaluated by calculating the RPD for the percentage recoveries of MS and MSD samples. Two sets of samples (MS and MSD) were submitted to the laboratory for precision testing. Sample 1 was spiked with benzonitrile, and Sample 2 was spiked with surrogate compounds. The average of the RPD obtained for surface-wipes was well below the limit stipulated in the QAPjP (i.e., 50 percent). The results for the precision for surface-wipes are summarized in Table B-5 in Appendix B. The recovery of MS and MSD samples for metals could not be calculated because the concentration in the native sample was very high.

The matrix-spiked duplicates prepared for the purpose of assessing method precision were analyzed at a frequency of 1 for every 20 samples analyzed for each sample type.

5.6.1.4 Blanks

Three field blanks (wipes) were extracted and analyzed by Method 8270 in the same manner as the other surface-wipe samples to check for background contamination. No target analytes were detected in any of the field blanks. In addition, reagent blanks were analyzed to assess the purity of the reagents used for the analyses. No contamination was found in any of the reagent blanks. Test results of the blanks for surface-wipes and metal strips are shown in Tables B-6 and B-7 respectively in Appendix B.

5.6.2 Herbicides

All herbicide extracts (Dicamba, 2,4-D, and 2,4,5-T) were analyzed in accordance with the procedures outlined in Method 8150 of SW-846 (4).

5.6.2.1 Analyte Calibration

A six-point calibration was performed with standards at concentrations of 0.25, 0.5, 1, 2.5, 5, and 10 ppb for generating response factors. This also indicated an RSD of less than 15 percent for all calibration check compounds. These calibration check compounds were reanalyzed after every 10 samples to verify that the response factor remained within ± 15 percent of that generated from an average of the six-point standard.

5.6.2.2 Accuracy

Each sample was spiked with a surrogate (2,4-dichlorophenylacetic acid) before analysis to monitor extraction efficiency. Surrogate recoveries for pre- and posttreatment surface-wipe samples are summarized in Tables B-8 and B-9 respectively and those for metal samples are shown in Tables B-10 and B-11, respectively, all in Appendix B. The mean percentage recoveries of all surrogates were within acceptable QA/QC limits.

5.6.2.3 Precision

The RPD was calculated for precision of the analytical method. The average of the RPDs obtained for surface-wipes and metal strips was well below the 50 percent limit specified in the QAPjP. The precision results for surface-wipes and metal strips are summarized in Tables B-12 and B-13 respectively in Appendix B.

5.6.2.4 *Blanks*

The field and reagent blanks for surface-wipes and metal strips were analyzed to check for background contamination. No target analytes were detected in any of the blanks. Analytical results of the blanks for surface-wipes and metal strips are presented in Tables B-14 and B-15 respectively in Appendix B.

5.6.3 *Dioxins/Furans*

All dioxin and furan extracts were analyzed in accordance with the procedures outlined in Method 8280 of SW-846 (4).

5.6.3.1 *Analyte Calibration*

A five-point calibration of 0.2-, 0.5-, 1-, 2-, and 5-ppb standards was used to generate response factors. The calibration check compounds were reanalyzed every 12 hours to verify that the response factor remained within ± 30 percent of that generated from an average of the five-point standard.

5.6.3.2 *Accuracy*

All samples were spiked with Carbon-13-labeled surrogate standards. Percentage recoveries for each of the Carbon-13-labeled surrogates are shown in Table B-16 in Appendix B. The mean percentage recovery of all surrogates ranges from 82 to 118.

5.6.3.3 *Blanks*

The reagent blanks for metal strips were analyzed to check for background contamination. No target analytes were detected in any of the blanks. Results of reagent blanks are shown in Table B-17 of Appendix B.

SECTION 6

COST OF DEMONSTRATIONS

This section summarizes the capital equipment cost of pilot-scale DWS and the cost of conducting the EPA SITE demonstrations of DWS at the Gray PCB site in Hopkinsville, KY, and at the Shaver's Farm site near Chickamauga, GA.

6.1 Capital Equipment Cost

The cost for the design, engineering, equipment procurement, fabrication, and installation of the pilot-scale DWS was approximately \$75,000. The cost includes all the subsystems and components installed and also the initial shakedown of the system.

6.2 Cost of Demonstration

The pilot-scale DWS was demonstrated at the Gray PCB site and at the Shaver's Farm site. The costs (rounded to the nearest \$100) for each field demonstration are summarized in Table 24. The costs of the demonstration (as shown in Table 24) may not be representative of any actual site operation because the pilot-scale DWS represents an experimental system which is highly labor-intensive with a relatively low processing rate. However, the operation cost could be greatly reduced when the economy of a semiautomatic, large-scale system is considered. The field activities conducted during both demonstrations included:

- Site preparation
- Mobilization
- Equipment setup
- Operations/test runs
- Sample collections
- Chemical analyses
- Demobilization

TABLE 24. SUMMARY OF PILOT-SCALE DWS DEMONSTRATION COSTS

Category	Gray PCB Site Demonstration		Shaver's Farm Site Demonstration	
	Cost, \$	Cost, %	Cost, \$	Cost, %
Labor (including per diem)	47,000	39	35,000	25
Equipment and supplies	16,000	13	12,000	9
Equipment transportation	1,500	1	2,000	1
Equipment rental	15,000	12	8,000	6
Travel expenses for crew	4,000	3	10,000	7
Subcontractor	19,500	16	12,000	9
Chemical analysis	19,000	16	61,000	43
Total	122,000	100	140,000	100

6.2.1 *Cost of Demonstration at Gray PCS Site*

The cost incurred during the demonstration of the pilot-scale DWS at the Gray PCB site was approximately \$122,000. All the field activities lasted for 33 days. The crew included one project engineer and two technicians working an average of 10 hours per day during the course of the entire demonstration.

The rental equipment included a forklift, generator, air compressor, and 48-ft semitrailer. The subcontractor costs included a concrete pad, temporary enclosure, and site security.

6.2.2 *Cost of Demonstration at Shaver's Farm Site*

The cost incurred during the second demonstration of the pilot-scale DWS at the Shaver's Farm site was approximately \$140,000. All the field activities at this site lasted for 17 days. The crew included one project engineer and three technicians working an average of 10 hours per day during the course of the entire demonstration.

The rental equipment included a forklift, generator, air compressor, and 48-ft semitrailer. The subcontractor costs included a concrete pad and temporary enclosure.

SECTION 7

CONCLUSIONS AND RECOMMENDATIONS

7.1 Conclusions

On the basis of the bench-scale and pilot-scale demonstrations performed in Phases 1 and 2, the following conclusions have been drawn:

- 1) The DWS and ancillary equipment proved to be portable and rugged.
- 2) Overall performance of the DWS was exceptional. Only minor, readily resolvable difficulties and problems were encountered, as is the case with any system startup.
- 3) The DWS was successfully employed to remove PCBs from the surfaces of transformer casings found at a hazardous waste site.
- 4) The DWS was also effective in removing herbicides, pesticides, dioxins, and furan residues from the surfaces of contaminated drums found at a hazardous waste site.
- 5) Significant quantities of metals were removed during the pilot-scale testing, which suggests that the system may also be applicable for treating debris contaminated with metals.
- 6) The cleaning solution was successfully recovered and reconditioned for reuse concurrently with the actual debris-cleaning process, which minimizes the quantity of process water required to clean the debris.
- 7) The two demonstrations of DWS were carried out during the months of December and June, when ambient temperatures ranged from -30° to 50°F and from 75° to 105°F, respectively. The effectiveness of the system's operation during these extreme temperatures was indicative of its durability and versatility.

- 8) The water treatment system was effective in reducing the concentrations of all the organic and metal contaminants (except arsenic and Dicamba) to below the detection limit.

7.2 Recommendations

The following recommendations have been made concerning the field-scale DWS:

- 1) Additional bench- or pilot-scale studies should be performed to improve the efficiency of water treatment systems.
- 2) The labor intensity of the system should be reduced by modifying it with materials-handling equipment and automation.
- 3) Alternative equipment (preferably fully automated) should be identified to cut the large and bulky pieces of debris before cleaning in the DWS.
- 4) Design of a full-scale, fully portable, self-contained DWS should be initiated for the removal of various contaminants from debris found on hazardous waste sites.
- 5) The utility of the DWS in "treatment trains" in which two or more processes are used for sequential treatment of contaminated wastes should be investigated.

SECTION 8

FULL-SCALE DEBRIS WASHING SYSTEM: CONCEPTUAL DESIGN

This section describes the conceptual design of a full-scale DWS, which evolved from the bench- and pilot-scale work. The lessons learned from these two stages are incorporated into the design, and the elements that worked well have been retained. It may be possible to modify an existing commercial system; however, the design described here is conceptual design.

Figure 9 is a schematic block diagram of the full-scale DWS. The debris will be loaded in a basket, lifted by a monorail crane, and lowered into the wash tank. The wash tank will be sealed and filled with hot detergent solution from the detergent tank. The debris will be washed by the solution recycled by the wash pump. A small bleed stream will be sent to the water treatment system

At the end of the wash cycle, the basket will be transferred to the spray/rinse tank, which may have the ability to rotate the basket or the sprays. The batch will be sprayed with the hot detergent solution and rinsed with cold water. Again, a bleed stream will be sent to the water treatment system.

The unit will be equipped with a control panel and a hot oil heating system. The heating system can be an electric or oil-fired unit.

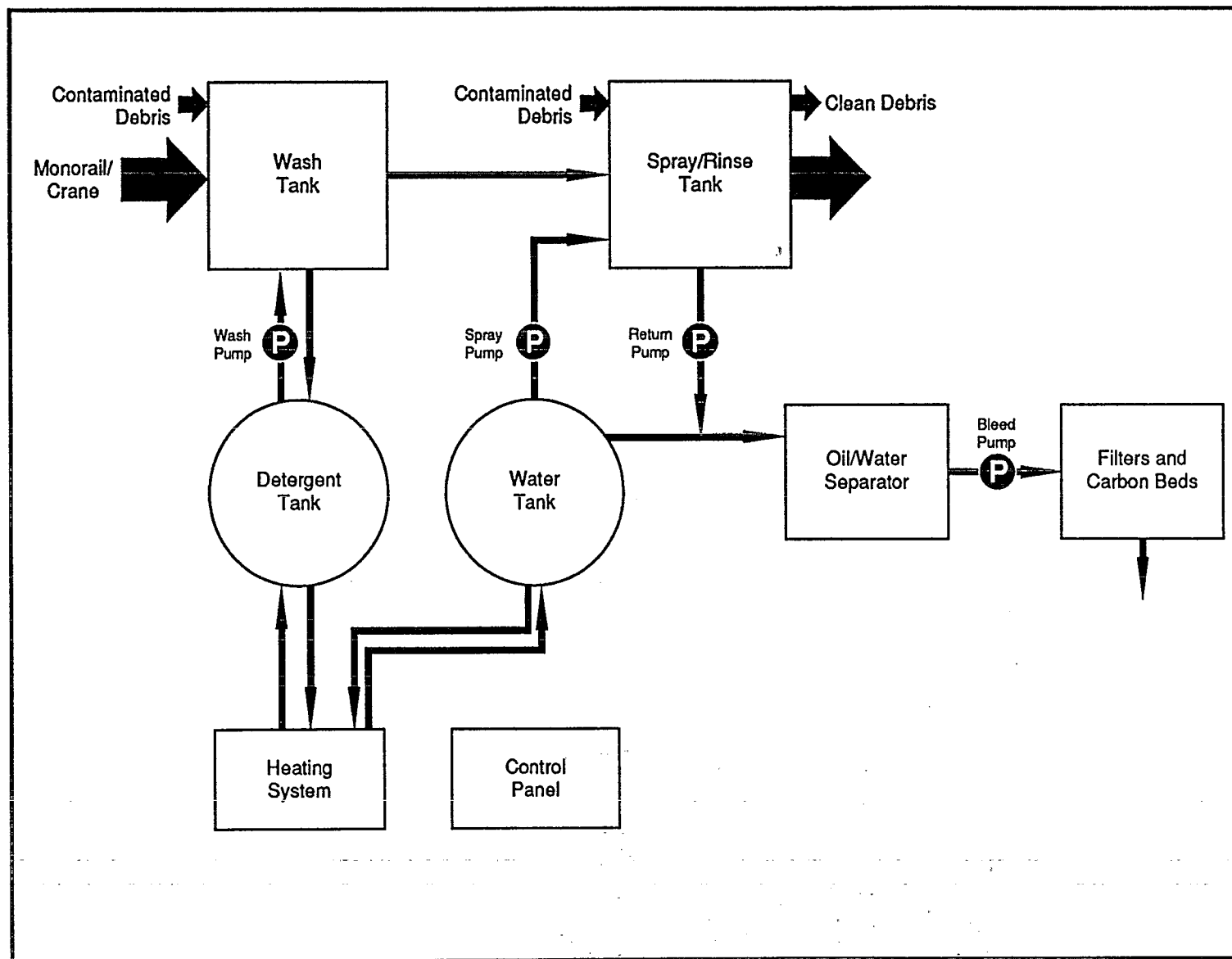


Figure 9. Schematic diagram of a full-scale DWS.

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APPENDIX A
QA/QC DATA
GRAY PCB SITE DEMONSTRATION

**TABLE A-1 SUMMARY OF MATRIX SPIKE RECOVERIES
(METHOD 8080 - WIPE SAMPLES)**

Sample ID: (MS/MSD)	Matrix Spike (C1)	Matrix Spike Duplicate (C2)	Precision ^a	Range ^b
9001557/9001558	90	106	16%	b
9001534/9001535	67	89	28%	b
9001284/9001285	62	62	0%	b
9001264/9001265	60	57	5%	b
9000739/9000740	93	77	19%	b
9000664/9000665	73	77	5%	b
9000709/9000710	67	80	18%	b
9000677/9000678	64	86	29%	b
9000689/9000690	67	69	3%	b

^a Precision estimated by calculation of relative percent difference (RPD) using the following equation:

$$RPD = \frac{\frac{|C_1 - C_2|}{C_1 + C_2}}{2} \times 100$$

^b Range established in QAPjP as 30%

TABLE A-2. FIELD AND REAGENT BLANKS (METHOD 8080 - WIPES SAMPLES)
($\mu\text{g}/100\text{cm}^2$)

Batch No.	Field Blank	Reagent Blank
1	<0.1	<0.1
2	<0.1	<0.1
3	<0.1	<0.1
4	<0.1	<0.1
5	<0.1	<0.1
6	<0.1	<0.1
7	<0.1	<0.1
8	<0.1	<0.1
9	<0.1	<0.1
10	<0.1	<0.1
11	<0.1	<0.1
12	<0.1	<0.1
13	<0.1	<0.1
15	<0.1	<0.1
16	<0.1	<0.1
18	<0.1	<0.1
19	<0.1	<0.1
20	<0.1	<0.1
21	<0.1	<0.1
22	<0.1	<0.1
23	<0.1	<0.1
24	<0.1	<0.1

APPENDIX B
QA/QC DATA
SHAVER'S FARM SITE DEMONSTRATION_

**TABLE B-1. SUMMARY OF SURROGATE RECOVERIES OF PRETREATMENT
WIPE SAMPLES (METHOD 8270)
(PERCENT)**

Surrogate	Number of Data Points	Mean Percent Recovery	Range	Standard Deviation
2-Fluorobiphenyl	18	71	9-114	34
2-Fluorophenol	19	67	4-102	27
Nitrobenzene-d5	19	65	8-94	31
Phenol-d5	18	71	0-99	29
Terphenyl-d14	19	100	15-184	43
2,4,6-Tribromophenol	18	67	4-130	36

**TABLE B-2. SUMMARY OF SURROGATE RECOVERIES OF POSTTREATMENT
WIPE SAMPLES (METHOD 8270)
(PERCENT)**

Surrogate	Number of Data Points	Mean Percent Recovery	Range	Standard Deviation
2-Fluorobiphenyl	17	94	71-113	14
2-Fluorophenol	17	73	43-99	16
Nitrobenzene-d5	17	80	52-101	14
Phenol-d5	17	74	47-100	16
Terphenyl-d14	17	107	65-166	30
2,4,6-Tribromophenol	17	78	23-138	22

**TABLE B-3. SUMMARY OF SURROGATE RECOVERIES OF PRETREATMENT
METAL STRIP SAMPLES (METHOD 8270)
(PERCENT)**

Surrogate	Number of Data Points	Mean Percent Recovery	Range	Standard Deviation
2-Fluorobiphenyl	9	74	16-120	40
2-Fluorophenol	9	58	12-85	23
Nitrobenzene-d5	9	60	10-115	34
Phenol-d5	9	78	9-116	33
Terphenyl-d14	9	108	22-187	50
2,4,6-Tribromophenol	9	55	10-120	34

**TABLE B-4. SUMMARY OF SURROGATE RECOVERIES OF POSTTREATMENT
METAL STRIP SAMPLES (METHOD 8270)
(PERCENT)**

Surrogate	Number of Data Points	Mean Percent Recovery	Range	Standard Deviation
2-Fluorobiphenyl	9	100	78-116	12
2-Fluorophenol	9	62	46-91	17
Nitrobenzene-d5	9	81	57-92	11
Phenol-d5	9	72	56-90	12
Terphenyl-d14	9	118	80-199	41
2,4,6-Tribromophenol	9	62	39-75	10

**TABLE B-5. SUMMARY OF MATRIX SPIKE RECOVERIES
(METHOD 8270 - WIPE SAMPLES)**

	Matrix Spike (C1)	Matrix Spike Duplicate (C2)	Precision ^a	Range ^b
Sample ID: MS & MSD-8/2 Benzonitrile	118	150	24%	50%
Sample ID: MS & MSD-7/20				
Acenaphthene	65	65	0%	b
4-Chloro-3-methylphenol	64	82	25%	b
2-Chlorophenol	58	89	42%	b
1,4-Dichlorobenzene	41	81	66%	b
2,4-Dinitrotoluene	74	66	11%	b
N-Nitrosodipropylamine	37	54	37%	b
4-Nitrophenol	51	55	8%	b
Pentachlorophenol	84	86	2%	b
Phenol	60	84	33%	b
Pyrene	75	78	4%	b
1,2,4-Trichlorobenzene	65	90	32%	b

^a Precision estimated by calculation of relative percent difference (RPD) using the following equation:

$$RPD = \frac{\frac{|C_1 - C_2|}{C_1 + C_2}}{2} \times 100$$

^b Range established in QAPjP as 50%

TABLE B-6. FIELD AND REAGENT BLANKS (METHOD 8270 - WIPE SAMPLES)
($\mu\text{g}/100\text{cm}^2$)

	Field Blank (Batch #2)	Field Blank (Batch #6)	Field Blank (Batch #10)	Reagent Blank	Reagent Blank	Reagent Blank
Benzonitrile	ND ^a (5.0) ^b	ND (5.0)	ND (5.0)	ND (5.0)	ND (5.0)	ND (5.0)
2,4-Dichlorophenol	ND (5.0)	ND (5.0)	ND (5.0)	ND (5.0)	ND (5.0)	ND (5.0)
2,6-Dichlorophenol	ND (5.0)	ND (5.0)	ND (5.0)	ND (5.0)	ND (5.0)	ND (5.0)
1,2,4-Trichlorobenzene	ND (5.0)	ND (5.0)	ND (5.0)	ND (5.0)	ND (5.0)	ND (5.0)

^a Not detected at specified detection limit.

^b Numbers in parenthesis indicate the minimum detectable concentration of the analyte.

TABLE B-7. REAGENT BLANK (METHOD 8270 - METAL STRIP SAMPLES)
($\mu\text{g}/\text{g}$)

	Reagent Blank
Benzonitrile	ND ^a (0.50) ^b
2,4-Dichlorophenol	ND (0.50)
2,6-Dichlorophenol	ND (0.50)
1,2,4-Trichlorobenzene	ND (0.50)

^a Not detected at specified detection limit.

^b Numbers in parenthesis indicate the minimum detectable concentration of the analyte.

**TABLE B-8. SUMMARY OF SURROGATE RECOVERIES OF PRETREATMENT
WIPE SAMPLES (METHOD 8150)
(PERCENT)**

Surrogate	Number of Data Points	Mean Percent Recovery	Range	Standard Deviation
2,4-Dichlorophenylacetic acid	13	99	33-132	26

**TABLE B-9. SUMMARY OF SURROGATE RECOVERIES OF POSTTREATMENT
WIPE SAMPLES (METHOD 8150)
(PERCENT)**

Surrogate	Number of Data Points	Mean Percent Recovery	Range	Standard Deviation
2,4-Dichlorophenylacetic acid	13	104	98-107	3

**TABLE B-10. SUMMARY OF SURROGATE RECOVERIES OF PRETREATMENT
METAL STRIP SAMPLES (METHOD 8150)
(PERCENT)**

Surrogate	Number of Data Points	Mean Percent Recovery	Range	Standard Deviation
2,4-Dichlorophenylacetic acid	7	103	90-125	15

**TABLE B-11. SUMMARY OF SURROGATE RECOVERIES OF POSTTREATMENT
METAL STRIP SAMPLES (METHOD 8150)
(PERCENT)**

Surrogate	Number of Data Points	Mean Percent Recovery	Range	Standard Deviation
2,4-Dichlorophenylacetic acid	7	106	90-122	11

**TABLE B-12. SUMMARY OF MATRIX SPIKE RECOVERIES
(METHOD 8150 - WIPE SAMPLES)**

	Matrix Spike (C1)	Matrix Spike Duplicate (C2)	Precision ^a	Range ^b
Sample ID: MS1-7/20 Dicamba	90	95	5%	50%
Sample ID: MS1-8/2 Dicamba	90	82	9%	50%

^a Precision estimated by calculation of relative percent difference (RPD) using the following equation:

$$RPD = \frac{\frac{|C_1 - C_2|}{C_1 + C_2}}{2} \times 100$$

^b Range established in QAPjP as 50%

**TABLE B-13. SUMMARY OF MATRIX SPIKE RECOVERIES
(METHOD 8150 - METAL STRIP SAMPLES)**

	Matrix Spike (C1)	Matrix Spike Duplicate (C2)	Precision ^a	Range ^b
Dicamba	137	150	9%	50%

^a Precision estimated by calculation of relative percent difference (RPD) using the following equation:

$$RPD = \frac{\frac{|C_1 - C_2|}{C_1 + C_2}}{2} \times 100$$

^b Range established in QAPjP as 50%

TABLE B-14. FIELD AND REAGENT BLANKS (METHOD 8150 - WIPE SAMPLES)
($\mu\text{g}/100\text{cm}^2$)

	Field Blank (Batch #4)	Field Blank (Batch #8)	Field Blank (Batch #9)	Reagent Blank	Reagent Blank
Dicamba	ND ^a (0.27) ^b	ND (0.27)	ND (0.27)	ND (0.27)	ND (0.27)
2,4-D	ND (1.2)	ND (1.2)	ND (1.2)	ND (1.2)	ND (1.2)
2,4,5-T	ND (0.20)	ND (0.20)	ND (0.20)	ND (0.20)	ND (0.20)

^a Not detected at specified detection limit.

^b Numbers in parenthesis indicate the minimum detectable concentration of the analyte.

TABLE B-15. REAGENT BLANK (METHOD 8150 - METAL STRIP SAMPLES)
($\mu\text{g}/\text{g}$)

	Reagent Blank	Reagent Blank
Dicamba	ND ^a (0.27) ^b	ND (0.27)
2,4-D	ND (1.2)	ND (1.2)
2,4,5-T	ND (0.20)	ND (0.20)

^a Not detected at specified detection limit.

^b Numbers in parenthesis indicate the minimum detectable concentration of the analyte.

TABLE B-16. SUMMARY OF SURROGATE RECOVERIES OF PRE- AND POSTTREATMENT METAL STRIP SAMPLES (METHOD 8280) (PERCENT)

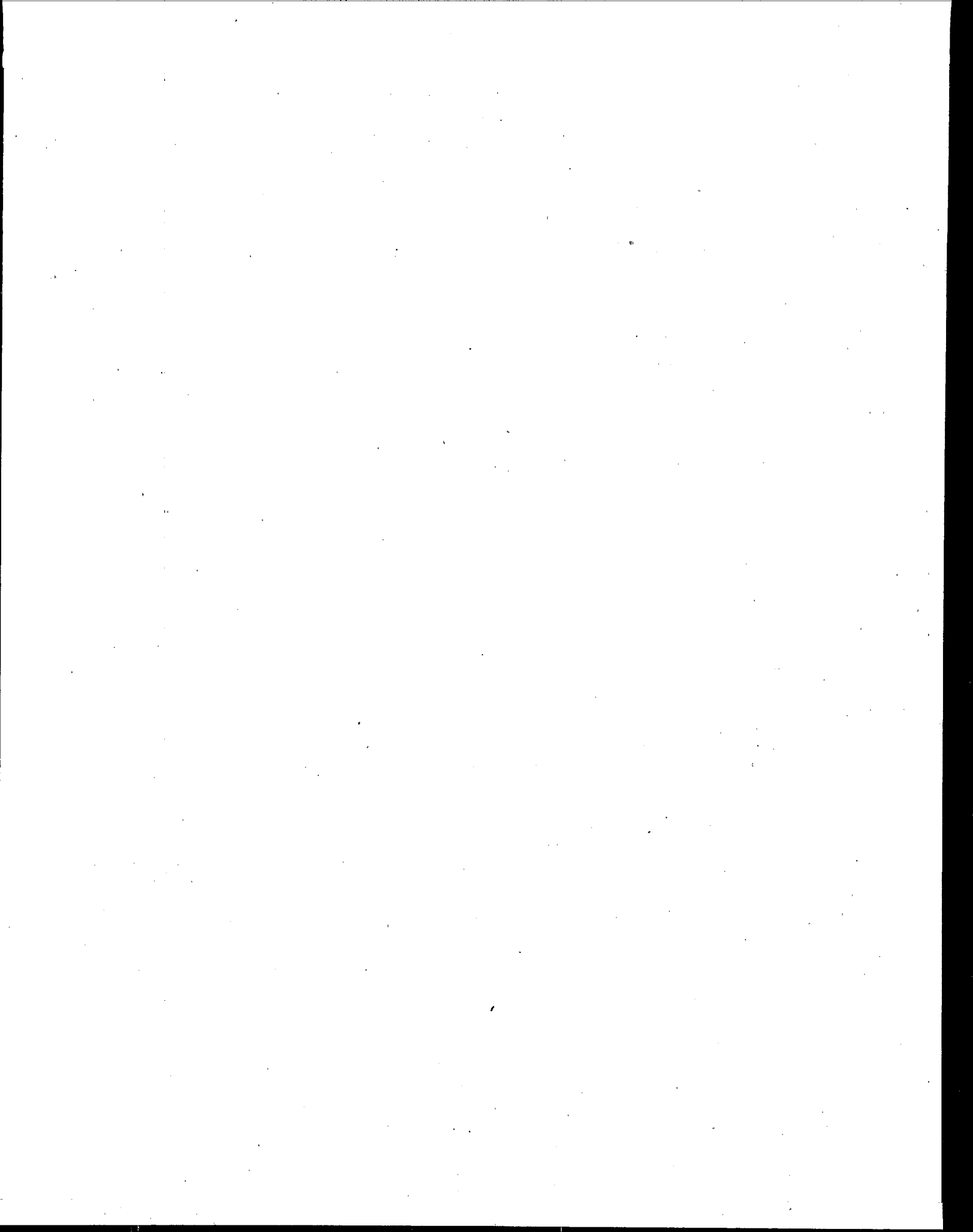
Surrogate	Number of Data Points	Mean Percent Recovery	Range	Standard Deviation
¹³ C-1,2,3,4,6,7,8-HpCDD	4	93	86-101	6
¹³ C-1,2,3,4,6,7,8-HpCDF	4	82	74-91	7
¹³ C-1,2,3,6,7,8-HxCDD	4	93	88-98	4
¹³ C-1,2,3,6,7,8-HxCDF	4	95	89-99	4
¹³ C-1,2,3,7,8-PeCDD	4	102	97-107	5
¹³ C-1,2,3,7,8-PeCDF	4	100	97-103	3
¹³ C-2,3,7,8-TCDD	4	98	94-102	4
¹³ C-2,3,7,8-TCDF	4	118	113-124	5
¹³ C-OCDD	4	85	69-107	16
¹³ C-OCDF	4	84	67-104	16

**TABLE B-17. REAGENT BLANK (METHOD 8280 - METAL STRIP SAMPLES)
(ng/g)**

	Reagent Blank
HpCDD	ND ^a (0.70) ^b
HpCDF	ND (0.50)
HxCDD	ND (0.36)
HxCDF	ND (0.21)
OCDD	ND (2.9)
OCDF	ND(0.97)
PeCDD	ND (0.18)
PeCDF	ND (0.10)
TCDD	ND (0.16)
2,3,7,8-TCDD	ND (0.16)
TCDF	ND (0.095)

^a Not detected at specified detection limit.

^b Numbers in parenthesis indicate the minimum detectable concentration of the analyte.



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