

# **Treatability Study Report of Green Mountain Laboratories, Inc.'s Bioremediation Process**

## **Treatment of PCB Contaminated Soils, at Beede Waste Oil/Cash Energy Superfund Site, Plaistow, New Hampshire**

by

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Technical Project Manager

Vicente Gallardo  
Land Remediation and Pollution Control Division  
National Risk Management Research Laboratory  
Cincinnati, Ohio 45268

National Risk Management Research Laboratory  
Office of Research and Table Development  
U.S. Environmental Protection Agency  
Cincinnati, Ohio 45268

## **NOTICE**

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Sally Guiterrez, Acting Director  
National Risk Management Research Laboratory

## **ABSTRACT**

In 1998, under the sponsorship of the New Hampshire - Department of Environmental Services (NHDES), Green Mountain Laboratories, Inc. (GML) and the USEPA agreed to carry out a Superfund Innovative Technology Evaluation (SITE) project to evaluate the effectiveness of GML's Bioremediation Process for the treatment of PCB contaminated soils at the Beede Waste Oil/Cash Energy Superfund site in Plaistow, New Hampshire (hereinafter referred to as the Beede site). The treatment process involved inoculation/augmenting of the PCB contaminated soils with bulk microbial inoculum and nutrients, and allowing the microbes to aerobically degrade the PCBs. The bulk inoculum was produced on-site by the developer using animal feed-grade oatmeal as the substrate, shredded pine needles that provided certain specific co-metabolite compounds, nutrients and a proprietary consortium of microorganisms capable of degrading the PCBs to their eventual endpoints - carbon dioxide and mineral halides.

The results of the field evaluation of the technology, which are based on the data collected from the treatability study conducted in the third quarter of 1998, indicate no removal/degradation of the PCBs. In earlier laboratory tests, GML had used concentrated pine extract to provide the co-metabolite compounds, whereas, for the field study it used shredded pine needles. At the end of the field treatability study, based on its own observations and data, GML concluded that it may have inadvertently made some fundamental errors in the production and application of the bulk inoculum. Subsequently, the EPA SITE program and the NHDES agreed to give GML another opportunity to demonstrate its technology's capability in degrading PCB in the Beede site soil, but at a much smaller laboratory scale. In September 2000, GML carried out a limited number of preliminary bench-scale tests, at the Middlebury College in Middlebury, Vermont to reestablish the viability of its process. At the conclusion of the bench-scale tests, GML conceded that, at best the tests were inconclusive and at worst had failed. The project was terminated at that time.

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## **Section 1.0 INTRODUCTION**

In 1980, the U.S. Congress passed the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), also known as Superfund, committed to protecting human health and the environment from uncontrolled hazardous waste sites. CERCLA was amended by the Superfund Amendments and Reauthorization Act (SARA) in 1986. These amendments emphasize the long term effectiveness and permanence of remedies at Superfund sites. SARA mandates implementing permanent solutions and using alternate treatment technologies or resource recovery technologies, to the maximum extent possible, to clean up hazardous waste sites.

State and Federal agencies, as well as private parties, are now exploring a growing number of innovative technologies for treating hazardous waste. The sites on the National Priorities List total more than 1,200 and comprise a broad spectrum of physical, chemical, and environmental conditions requiring varying types of remediation. The U.S. Environmental Protection Agency (EPA) has focused on policy, technical, and informational issues related to exploring and applying new remediation technologies applicable to Superfund sites. One such initiative is EPA's Superfund Innovative Technology Evaluation (SITE) Program, which was established to accelerate development, demonstration, and use of innovative technologies for site cleanups.

In 1998, a bioremediation process developed by Green Mountain Laboratories, Inc. (GML), Middlesex, Vermont was field-tested under the SITE program. Through its research in the laboratory and some limited involvement in PCB remediation projects, GML believed that it had put together a process combining engineering, chemistry, and microbiology to remediate PCBs in soil. GML believed that its unique on-site processing technique using indigenous microbes, co-metabolites and nutrients could drive the bioremediation of PCBs to their ultimate endpoint of carbon dioxide and mineral halides.

A pilot-scale treatability study of the GML PCB Bioremediation process was performed at the Beede Waste Oil/Cash Energy site (hereafter referred to as Beede site) in Plaistow, New Hampshire, over an eight week period, from August through October 1998.

The results of the field study indicated that the GML process was unsuccessful in degrading the PCBs. In earlier laboratory tests, GML had used a concentrated extract of pine needles to provide the co-metabolite compounds to facilitate PCB degradation, whereas for the field study, it used shredded pine needles. At the end of the field study, based on its own observations and data GML concluded that it may have inadvertently made a fundamental error in the production of the bulk inoculum by using the shredded pine needles instead of the concentrated extract. Consequently, the EPA SITE program and the NHDES agreed to give GML another opportunity to demonstrate the technology's capability in degrading PCBs in the Beede site soil. However, GML was asked to first reestablish the viability of the process on a much smaller laboratory scale before conducting another field demonstration. In September 2000, GML conducted a limited number of preliminary bench-scale tests at the Middlebury College in Middlebury, Vermont to examine its process and the processing protocols. At the conclusion of the bench-scale tests, GML conceded that, at best the tests were inconclusive and at worst had failed to establish the capability of its PCB bioremediation process. The project was terminated at that time.

This treatability study report, organized into six sections, describes the GML technology, provides



information pertaining to the staging of the treatability tests, and analyzes data from the field trial. **Section 1** presents background on the SITE program, the selection of the GML technology into the Demonstration Program, and lists points of contact for GML, NHDES and the SITE program. **Section 2** describes the fundamentals of the GML PCB bioremediation process, and the research and development work that led to the technology's current design. **Section 3** summarizes the treatability study from the planning stage through the field trial. **Section 4** analyzes the data and discusses the results. **Section 5** reviews quality assurance/quality control (QA/QC) issues. **Section 6** lists technical references used in developing this report.

Although the GML technology did not meet its treatment objectives, publishing the results of the field treatability study is still worthwhile. By carefully documenting the experimental design of the project and describing its results, researchers can advance the technology by exploring new approaches.

This report represents the only published EPA document resulting from this SITE Program-sponsored project.

## **1.1 SITE Program**

The SITE Program is a formal program established by the EPA's Office of Solid Waste and Emergency Response (OSWER) and Office of Research and Development (ORD) in response to the Superfund Amendments and Reauthorization Act of 1986 (SARA). The SITE Program promotes the development, demonstration, and use of new or innovative technologies to clean up Superfund sites across the country.

The SITE Program's primary purpose is to maximize the use of alternatives in cleaning hazardous waste sites by encouraging the development and demonstration of new, innovative treatment and monitoring technologies. It consists of three major elements:

- the Demonstration Program,
- the Consortium for Site Characterization Technologies (CSCT), and
- the Technology Transfer Program.

The objective of the Demonstration Program is to develop reliable performance and cost data on innovative technologies so that potential users can assess the technology's site-specific applicability. Technologies evaluated are either available commercially or close to being available for full-scale remediation of Superfund sites. SITE demonstrations usually are conducted at hazardous waste sites under conditions that closely simulate full-scale remediation conditions, thus assuring the usefulness and reliability of the information collected. Data collected are used to assess: (1) the performance of the technology; (2) the potential need for pre- and post-treatment of wastes; (3) potential operating problems; and (4) the approximate costs. The demonstration also provides opportunities to evaluate the long term risks and limitations of a technology.

Existing and new technologies and test procedures that improve field monitoring and site characterizations are explored in the CSCT Program. New monitoring technologies, or analytical methods that provide faster, more cost-effective contamination and site assessment data are supported by this program. The CSCT Program also formulates the protocols and standard

operating procedures for demonstration methods and equipment.

The Technology Transfer Program disseminates technical information on innovative technologies in the Demonstration and CSCT Programs through various activities. These activities increase awareness and promote the use of innovative technologies for assessment and remediation at Superfund sites. The goal of technology transfer activities is to develop interactive communication among individuals requiring up-to-date technical information.

## **1.2 The SITE Demonstration Program and Reports**

For the first ten years in the history of the SITE program, technologies had been selected for evaluation through annual requests for proposals. EPA reviewed proposals to determine the technologies with promise for use at hazardous waste sites. Several technologies also entered the program from current Superfund projects, in which innovative techniques of broad interest were identified under the program.

In 1997 the program shifted from a technology driven focus to a more integrated approach driven by the needs of the hazardous waste remediation community. The SITE program now annually solicits applications for participation in the Demonstration program from parties responsible for clean up operations at hazardous waste sites. A team of stakeholders led by SITE program personnel will select sites and work with site representatives in bringing technologies for demonstration to their respective sites.

Once the EPA has accepted an application, cooperative arrangements are established among EPA, the developer, and the stakeholders to set forth responsibilities for conducting the demonstration and evaluating the technology. Developers are responsible for operating their innovative systems at a selected site, and are expected to pay the costs to transport equipment to the site, operate the equipment on site during the demonstration, and remove the equipment from the site. EPA is responsible for project planning, sampling and analysis, quality assurance and quality control, preparing reports, and disseminating information. Typically, results of Demonstration Projects are published in three documents: the SITE Demonstration Bulletin, the Technology Capsule, and the Innovative Technology Evaluation Report( ITER). The Bulletin describes the technology and provides preliminary results of the field demonstration. The Technology Capsule provides more detailed information about the technology and emphasizes key results of the SITE field demonstration. An additional report, the Technology Evaluation Report (TER), is available by request only. The TER contains a comprehensive presentation of the data collected during the demonstration and provides a detailed quality assurance review of the data.

However, with the GML study, the technology did not advance to a full Demonstration, thus only a treatability study report will be published.

## **1.3 Selection into the Demonstration Program**

In the past, technologies were selected for the Demonstration Program from a pool of responses to SITE's annual request for proposals (RFP). EPA reviewed proposals to search for innovative technologies that offered either reduced risk or cost or provided a treatment solution where none had existed previously. In 1997, the program shifted from a technology-driven focus to a more integrated approach shaped by the needs of the hazardous waste remediation community. The

annual RFP was discontinued, and instead a team of stakeholders matches technologies with a particular site that has been selected for study by the SITE program. The stakeholders and EPA solicit and evaluate proposals from technology developers interested in working at the chosen site.

In its "Host Site Application (HSA) to the SITE Demonstration Program" the State of New Hampshire - Department of Environmental Services (NHDES), on February 26, 1998, identified one such innovative technology. The technology identified is a bioremediation process for the treatment of soils contaminated with polychlorinated biphenyls (PCBs) developed by Green Mountain Laboratories, Inc. of Middlesex, Vermont (hereinafter also referred to as the "Developer"). In the HSA, the NHDES also identified a candidate site for demonstrating and evaluating the GML technology; the Beede Waste Oil/Cash Energy Superfund Site in Plaistow, New Hampshire (hereinafter also referred to as the Beede site).

#### **1.4 Points of Contact**

Additional information on the GML Technology, the Beede site and the SITE Program can be obtained from the following sources:

##### ***The GML PCB Bioremediation Technology:***

Raul Sanchez  
Green Mountain Laboratories, Inc.  
27 Cross Road  
Middlesex, Vermont 05602  
Telephone: (802) 223-1468  
Fax: (802) 223-8688  
Email: gml@together.net

##### ***The Beede Site:***

Robert P. Minicucci, II  
Innovative Technology Coordinator  
New Hampshire Department of Environmental Services (NHDES)  
P.O. Box 95  
6 Hazen Drive  
Concord, New Hampshire 03301  
Telephone: (603) 271-2941  
Fax: (603) 271-2456  
Email: rminicucci@des.state.nh.us

##### ***The SITE Program:***

Annette M. Gatchett, Acting Director  
Land Remediation and Pollution Control Division  
U.S. Environmental Protection Agency  
26 West Martin Luther King Drive  
Cincinnati, OH 45268  
Telephone: (513) 569-7697  
Fax: (513) 569-7620  
Email: gatchett.annette@epa.gov

Vicente Gallardo  
EPA SITE Technical Project Manager  
U.S. Environmental Protection Agency  
26 West Martin Luther King Drive  
Cincinnati, OH 45268  
Telephone: (513) 569-7176  
Fax: (513) 569-7620  
Email: gallardo.vincente@epa.gov

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

- The SITE Home Page ([www.epa.gov/ORD/SITE](http://www.epa.gov/ORD/SITE)) provides general program information, current project status, technology documents, and access to other remediation home. Note: URL is case sensitive.
- The OSWER CLU-In electronic bulletin board (<http://www.clu-in.org>) contains information on the status of SITE technology demonstrations.

Technical reports may be obtained by writing to USEPA/NSCEP, P.O. Box 42419, Cincinnati, Ohio 45242-2419, or by calling 800-490-9198.

## **Section 2.0**

### **TECHNOLOGY DESCRIPTION**

#### **2.1 Introduction**

Polychlorinated biphenyls (PCBs) are mixtures of synthetic organic chemicals that possess useful industrial characteristics. They are chemically stable, have low vapor pressure, low flammability, high heat capacity, low electrical conductivity, and high dielectric constant. Based on these properties commercial PCB mixtures were used in many industrial applications, especially in capacitors, transformers, and other electrical equipment. They were also used, but to a lesser extent, as plasticizers, hydraulic fluids and lubricants, carbonless copy paper, heat-transfer fluids and petroleum additives. The unique chemical properties, also contribute to the persistence of PCBs after they are released into the environment. Evidence that PCBs persist in the environment and may cause environmental and health hazards stopped the domestic manufacture of commercial mixtures in 1977. In 1976, US Congress enacted the Toxic Substance Control Act (TSCA), which directed the EPA to control the manufacture, processing, distribution, use, disposal, and labeling of PCBs. Regulations under TSCA govern all forms and combinations of chemicals which contain the biphenyl molecule with one or more chlorine atom substitutions. They also apply not only to the PCB chemicals themselves, but to items and materials which have been in contact with PCBs.

In the environment, PCBs also occur as mixtures of congeners, but their composition may differ from the commercial mixtures. After release into the environment, the composition of PCB mixture can change over time through partitioning, chemical transformation, and preferential bioaccumulation. PCBs adsorb to organic materials, sediments, and soils. PCBs are widespread in the environment, and humans can be exposed through multiple pathways. Levels in air, water, sediment, soil, and foods vary over several orders of magnitude, often depending on proximity to a source of release into the environment.

Based on the 1990 EPA Superfund guidelines, the NHDES has adopted the stringent soil cleanup standard of total PCBs < 1 mg/kg or ppm for residential areas. In the NHDES policy, the S-1 and S-2 standard is 1 ppm, while the S-3 standard is 2 ppm. The S-1 and S-2 standards apply to situations where soil is more accessible, whereas the S-3 standard is for fairly inaccessible soil. The current cleanup goals for PCB Superfund sites in EPA Region 1 are determined on a case-by-case basis and determined by the risks posed by each site.

In 1998, from August through October, over an eight-week period, a pilot-scale field treatability study of the GML technology was conducted at the Beede site in Plaistow, New Hampshire. The primary purpose of the treatability study was to provide an initial assessment of the effectiveness of the GML process in achieving or approaching either of these goals (the DES S1/S2 standards) under actual and/or simulated field conditions (soil character, other contaminants, weather, etc.).

Prior to entering the SITE program, GML conducted laboratory scale experiments on Beede soils using a unique combination of indigenous microbes, co-metabolites and nutrients. Based on the findings of these preliminary laboratory experiments, GML believed that it had developed the know-how that could be used to successfully degrade the full range of PCB congeners to innocuous final products, particularly carbon dioxide and mineral halides. GML claimed that it had designed its proprietary microbial consortium so that it would be able to successively degrade the various

intermediates that may be produced as well as the PCB congeners that may be present at a site. GML also claimed that the technology was designed to degrade PCBs in the presence of other contaminants such as oils (petroleum hydrocarbons) and heavy metals. The Beede site provided a unique opportunity to evaluate all these aspects of the process.

Information from the treatability study was to be used to design a larger scale system for treating a larger range and quantity of the contaminated soil at the site. Information from the study was also used to provide initial assessment of the applicability of GML bioremediation technology to other sites with similar waste characteristics.

## **2.2 The GML Bioremediation System**

### **2.2.1 Technology Description**

The GML PCB Bioremediation process involves bioaugmentation of the PCB contaminated soil with a customized microbial inoculum and a proprietary nutrient formulation. The bulk inoculum is built on food or animal grade grain (in the case of this study GML used oatmeal) that serves as the substrate as well as the food source for the proprietary consortium of PCB degrading microorganisms. The GML designers of the inoculum have indicated that in addition to the oatmeal and the microbial consortium, the extract of pine (specifically Spruce pine) needles forms a key constituent of the inoculum. According to GML, the Spruce pine extract supplies the microorganisms with terpenes (naturally occurring compounds found in Spruce pine needles) which serve as co-metabolites for the PCB degrading microbes. As per GML, as the microbes get acclimated towards the terpenes and other pine constituents they develop a greater affinity for PCB molecules, and thereby end up consuming/degrading the PCBs.

### **2.2.2 Process Development - As Described by GML**

As originally conceived, this project was intended to provide an inexpensive, natural and efficient method for the biodegradation of Polychlorinated Biphenyls (PCB). Of particular interest were those of higher molecular weight, such as Aroclor, 1254 and 1260 that had proven recalcitrant to biodegradation. This recalcitrance is based on the difficulty of dechlorinating highly substituted biphenyls and by the accumulation of by-products toxic to organisms capable of initiating the dehalogenation and cleavage of the biphenyl rings. Only naturally occurring organisms were to be employed and ideally the process was to be aerobic, as anaerobic conditions are logistically harder to maintain. It was also realized that no one organism was likely to carry out all necessary steps in the process, so a synergistic consortium of microbes was sought.

Isolation of microorganisms capable of tolerating the presence of and ultimately degrading PCBs was achieved through conventional methods. Coverslips coated with either Aroclor 1254 or 1260 were inserted into a dish containing soil known to be contaminated with Aroclors. A broth containing mineral salts, ammonium and trace metals intended to make up for deficiencies in the soil was added. Coverslips were then withdrawn at intervals; washed to remove loose debris and transferred to vials containing mineral salts, ammonium, trace metals and an Aroclor. Samples were transferred from vials demonstrating growth to a second vial, again containing mineral salts, ammonium, trace metals and Aroclor. Growth from this second set of vials was transferred to a third set from which regular passages were maintained in the same fashion as eukaryotic cell cultures, as well as being streaked onto an assortment of agar based solid media to facilitate isolation of the organisms. In this way, two aerobic, gram-negative rods were isolated which

demonstrated the potential to exist in tandem in or on minimal media with Aroclor 1254, 1260, or a combination of both as sole carbon source. It was also determined that one of these organisms was a methylotroph, as survival in Aroclor was aided by the addition of small amounts of methanol or formaldehyde.

After the initial isolation was achieved, the microbial pair was screened for its ability to degrade Aroclor 1254 and 1260 in aerobic culture. As no standard chromatographic method existed which could simultaneously separate PCBs from the major expected by-products, chlorocatechols and chlorobenzoates, a non-traditional approach was adopted. Thin layer chromatography, utilizing silica gel as the stationary phase and a water, methanol and acetic acid mobile phase proved capable of clearly separating all target compounds from chloroform extracts of test cultures. Target bands were plainly visible under ultraviolet light, and test cultures routinely exhibited evidence of the generation of the expected by-product not seen in uninoculated or killed control cultures.

Since the degradation of PCBs is stimulated through the use of a co-metabolite a search was made for a suitable structural analog. (In this application, GML defines a co-metabolite as a compound similar in structure but more easily degraded than the PCBs) The compound most commonly used experimentally, biphenyl, was eliminated since it is considered a hazard. The terpenes, a class of isoprenoid compounds common in the oils and waxes of plants, was selected, and crude extracts of balsam fir, pine and various herbs were tried. Balsam fir needles were settled upon as a source, being plentiful and easily available.

After selecting the organisms, media and a co-metabolite, a method of delivery to the soil was investigated. The delivery mechanism needed to be biodegradable, absorbent and economically feasible. Ease of storage, handling and transport were also considered. A process was developed in which oatmeal was saturated with mineral salt broth and an extract of balsam fir. This mixture, when dried and ground, resembled coarse sand. When needed, an appropriate amount could be inoculated, allowed to grow for 72 hours, fed with MSB and mixed with the soil. The goal was to insure sufficient cell density within the inoculum to allow the introduced cells to grow and thrive when introduced into soil. This amendment could be added to the soil and land farmed for aeration, and sprayed with MSB and fir extract until PCB levels in the target soil fell to acceptable levels.

## **Section 3.0**

### **TREATABILITY STUDY**

#### **3.1 Treatability Study Overview**

Based on the 1990 EPA Superfund guidelines the NHDES has adopted the soil cleanup standard of Total PCBs < 1 mg/kg or ppm for residential areas. In the NHDES policy, the S-1 and S-2 standard is 1 ppm, while the S-3 standard is 2 ppm. The S-1 and S-2 standards apply to situations where soil is more accessible, whereas the S-3 standard is for fairly inaccessible soil. The current cleanup goals for PCB Superfund sites in EPA Region 1 are determined on a case-by-case basis and determined by the risks posed by each site.

According to GML, prior to entering the SITE Program, they performed laboratory scale experiments with the unique combination of indigenous microbes, co-metabolites, nutrients and processing techniques that successfully degraded the full range of PCB congeners to innocuous final products. GML designed its microbial consortium so that it was able to successively degrade the various intermediates that may be produced as well as the PCB congeners that may be present at a site. GML claimed that the technology was particularly designed to degrade PCBs in the presence of other contaminants such as oils (hydrocarbons) and heavy metals. The Beede site provided a unique opportunity to evaluate all these aspects of the process.

Thus, the primary purpose of this treatability study was to determine how effective the GML bioremediation process was in achieving or approaching either of the NHDES treatment goals under actual and/or simulated field conditions (soil character, other contaminants, weather, etc.).

If successful, information generated from this treatability study was to be used to design a larger scale system for treating a larger range and quantity of the contaminated soils at the site. Information from this study was to provide initial assessment of the applicability of GML's bioremediation technologies at other sites that may contain similar waste constituents.

In early June of 1998, GML conducted an experiment with six test plots at its Middlesex, Vermont facility on PCB-contaminated soils from the Beede site. Two soils, one from location S-109 and the other from S-43 were used. [Note: Locations S-109 and S-43 refer to the general locations where soil samples S-109 and S-43 had been collected, respectively, during an earlier remedial investigation study at the Beede site.] Each soil was thoroughly homogenized and split into three parts. For each type of soil, one part was set aside as untreated control, while the other two were subjected to two different treatments (in terms of the consortium selected). At the end of two weeks of treatment the four treated and two control samples were analyzed for PCBs. Results of these analyses are presented in Table 3.1-1. Although limited, these results confirmed for GML that some extent of PCB degradation was achieved. It also suggested that high concentrations of total petroleum hydrocarbons (TPHs) may have interfered or retarded PCB degradation. Therefore, for the subsequent treatability study GML selected a consortium based on these results that performed best with high and low TPH soils and extended the treatment period to eight weeks.



Table 3.1-1 Summary Results of the June 1998 Bench-Scale Tests

Experiment #	Treatment or Control	Soil Source Location	Reference Data		June 1998 Bench-Scale Test Data		
			Concentrations based on Remedial Investigation		PCB Concentrations for Bench-Scale Test Samples (Post Treatment Experiments 1-4)		Percent Reductions in <b>Total Aroclors</b> based on Control Soil concentrations
			Total Aroclors (mg/kg)	TPHs (mg/kg)	<b>Total Aroclors</b> (mg/kg)	Total Congeners (mg/kg)	
1	Treatment Consortium 1	S-109	250	8800	<b>234</b>	306	<b>37.3</b>
2	Treatment Consortium 1	S-43	260	600	<b>45.0</b>	48.8	<b>62.2</b>
3	Treatment Consortium 2	S-109	250	8800	<b>376</b>	489	<b>-0.8</b>
4	Treatment Consortium 2	S-43	260	600	<b>49.6</b>	56.3	<b>58.3</b>
5	Control	S-109	250	8800	<u><b>373</b></u>	508	
6	Control	S-43	260	600	<u><b>119</b></u>	140	

### 3.2 Waste/Soil Selected for Testing

Two locations on the Beede site with relatively high PCB concentrations (i.e.  $\geq 100$  mg/kg total PCBs) were selected based on previous analytical results. The selection of the two source locations also depended on the concentrations of other relevant contaminants (TPHs and metals), and on availability and accessibility of the soil. Based on initial RI data provided by NHDES, the two candidate sources were identified as the locations S-109 and S-43, which had shown total PCB concentrations of 250 and 260 mg/kg, respectively, in the surface soil. Based on the needs of the experimental design (i.e., the various treatment and control plots) discussed in Section 3.4, it was estimated that about 10 cubic yards of PCB-contaminated soil (excluding gross debris and material larger than 1 inch in size) would be needed to stage the treatability study; approximately 7 cu. yd. from location S-109 and 3 cu. yd. from location S-43.

### 3.3 Objectives and Scope of the Treatability Study

The primary objectives of this field treatability study were (1) to establish the applicability of the GML treatment process to the Beede site, and (2) to determine its effectiveness in biodegrading the primary target contaminants, PCBs, in soil which also contained lead and other organic contaminants including TPHs.

For the treatability study, GML proposed to set up and test a series of treatments and controls for a period of eight weeks using PCB-contaminated soil from two distinct locations on the Beede site. Details of the experimental design are discussed in Section 3.4. The data and information generated from this study was to provide a better understanding of the treatment process and allow the developer to identify the candidate treatments and controls that would be used subsequently in a pilot-scale demonstration study. The data from the treatability study was to allow the developer to determine the optimal treatment conditions and parameters, for customizing the treatment to the Beede site soil. In addition, the data generated through this treatability study would enable the developer to clearly define the primary and secondary objectives for the demonstration study and also allow the SITE Program to develop a comprehensive site-specific demonstration test plan and a Quality Assurance Project Plan (QAPP).

The objectives for the treatability study were:

1. To determine if the GML process could work under field conditions to degrade total PCBs from an initial concentration of 100 mg/kg or more to less than 1 mg/kg of total PCBs in soil, using the congener specific EPA Method 8082 or its equivalent. To accomplish this objective, for each plot, the pre- and post-treatment average total PCB Aroclor concentrations was to be computed (along with their corresponding upper and lower 90% confidence limits) and reported along with the corresponding congener specific total PCB data. In addition, the average percent reduction in total PCBs was to be determined for each plot (i.e., treatment or control) based on the baseline (Day 0) and the final (Day 56) total PCB Aroclor concentrations.
2. To estimate the PCB degradation rates for the various treatments and control by plotting the average total PCB Aroclor and the congener specific total PCB concentrations against the treatment time. This analysis was to provide the basis for selecting the candidate treatments and controls for the demonstration tests. In addition, it was to provide an initial estimate of the treatment duration for the demonstration.
3. To determine the impact of TPH concentrations on the process' ability to degrade PCBs in soil. This was to be accomplished by comparing the overall reductions as well as the degradation rates (i.e., the concentration versus time profiles) observed in the plots with identical treatments but on different soils (i.e., on S-109 with high and S-43 with low TPH concentrations).
4. To determine the reproducibility of the treatment performance data. This will be accomplished by comparing the performance data (in terms of overall reductions and degradation rates) obtained from duplicated experiments.
5. To determine if toxic by-products are being produced as a result of PCB degradation. This determination was to be based on analyses of intermediate and post-treatment soil samples for chlorobenzoates, chlorocatechols, vinyl chloride and other chlorinated compounds.
6. To examine the toxicological impact of the GML treatment on the soil. This was to be accomplished to a limited degree by examining toxicological data from the analysis of the pre- and post-treatment soil from a given treatment plot.
7. To examine the nature and size of microbial populations in the various treatments and

controls through the course of the demonstration. This examination was to be based on selected microbial analyses that would be performed on soils from the same five plots (treatments and controls) at the start, midpoint and end of the study.

### 3.4 Experimental Design

In order to determine the optimal processing parameters and conditions that could be later used to demonstrate the effectiveness of its PCB treatment process through a pilot-scale demonstration, GML staged a series of treatments and controls which are presented in the Experimental Design Matrix in Table 3.4-1. The design included a total of eight distinct experiments plus two duplicates to determine reproducibility of performance data. Thus, a total of ten ex-situ plots were set up for this treatability study. GML designed, constructed and maintained the treatment plots. SAIC provided oversight during construction.

Each ex-situ plot was approximately 4' x 3.5' in size and lined on the insides and bottom with two layers of 10-mil (or higher) polyethylene liner to prevent escape of contaminated leachate, if generated. Of the ten plots, nine were protected by a roof-like cover (in the form of a removable, raised but slanted plywood sheet cover), and one was left exposed to the elements. As the GML treatment process was intended to be aerobic in nature, seven of the ten ex-situ plots were equipped with a passive aeration system in the form of perforated corrugated PVC piping.

Surface soils (i.e., top 12 inches) from two selected locations (primarily from the locations of RI samples S-109 and S-43) on the Beede site were excavated, screened, analyzed on-site (using a gas chromatograph (GC) with an ECD detector) to determine PCB concentrations, and homogenized. The homogenized soils were then blended with GML's proprietary inoculum (consisting of a substrate and a custom designed consortium of microbes) or just the substrate (i.e., without the specific microbes) and placed in the appropriate treatment or control plots as per the experimental design matrix shown in Table 3.4-1. In the non-amended control plot only the homogenized soil (i.e., without the inoculum or the substrate) was placed. Soil was placed (loosely packed) in these plots to a depth of approximately 18 inches. Plot covers were installed in a manner that allowed easy removal and reinstallation to facilitate sample collection.

Through the course of the study, GML routinely monitored soil moisture levels and irrigated the plots as needed. A brief description of each plot is presented below.

- |        |   |
|--------|---|
| Plot 1 | Ex-situ covered (rain and direct sunlight sheltered) plot equipped with corrugated perforated piping that provided passive aeration and received inoculum at about 5% by weight of the soil within the plot. The soil used for this experiment was obtained from source location S-109. It did not receive methanol as a co-metabolite. |
| Plot 2 | Ex-situ covered plot with no corrugated perforated piping but received inoculum at about 5% by weight of the soil within the plot. The soil used for this experiment was obtained from source location S-109. It did not receive methanol as a co-metabolite.   |
| Plot 3 | Ex-situ covered plot equipped with corrugated perforated piping that provided passive aeration and received uninoculated substrate at about 5% by weight of the soil within the plot. The soil used for this experiment was obtained from source location S-109. It did not receive methanol as a co-metabolite.                        |
| Plot 4 | Ex-situ covered plot equipped with corrugated perforated piping that provided   |

passive aeration and received inoculum at about 5% by weight of the soil within the plot. The soil used for this experiment was obtained from source location S-109. In addition a liter of methanol was poured into the corrugated piping at the start of the experiment and every 14 days thereafter. After adding methanol, the manifold was plugged for a day or two to prevent off gassing. Methanol was added as the starter source of carbon for the inoculum. It did not function as a co-metabolite in the sense that it was a compound similar in structure and more easily degraded than the PCBs.

- Plot 5            Prepared and maintained identically as Plot 4. The experiment in Plot 5 was a duplicate to Plot 4.
- Plot 6            Ex-situ covered plot equipped with a corrugated perforated piping that provided passive aeration and received inoculum at about 5% by weight of the soil within the plot. The soil used for this experiment was obtained from source location S-43. In addition, a liter of methanol, which served as a carbon source, was poured into the corrugated piping at the start of the experiment and every 14 days thereafter. After adding methanol, the manifold was plugged for a day or two to prevent off gassing.
- Plot 7            Prepared and maintained identically as Plot 6. The experiment in Plot 7 was a duplicate to Plot 6.
- Plot 8            Ex-situ covered plot equipped with corrugated perforated piping that provided passive aeration but did not receive inoculum. The soil used for this experiment was obtained from source location S-109. In addition, no methanol was added through the course of the study.
- Plot 9            Ex-situ covered plot with no corrugated perforated piping and did not receive inoculum. The soil used for this experiment was obtained from source location S-109. In addition, no methanol was added through the course of the study.
- Plot 10          Ex-situ experiment in a plot with liner but no corrugated perforated piping for passive aeration, and was not covered. The S-43 soil mixed with 5% inoculum was used for this experiment. One liter of methanol, diluted in water, was sprayed on the plot initially and every 14 days thereafter.

Table 3.4-1 Experimental Design Matrix

Plot No.	Experimental Parameters					
	Source <sup>1</sup> Location of Soil Used	Plot Type	Plot Sheltered/ Covered?	Corrugated perforated piping Used?	Inoculum or Substrate Application, in % of weight of soil	Methanol <sup>2</sup> Application
1	S-109	Ex-situ	Yes	Yes	5% (inoculum)	No
2	S-109	Ex-situ	Yes	No	5% (inoculum)	No
3	S-109	Ex-situ	Yes	Yes	5% (Non-microbial substrate)	No
4	S-109	Ex-situ	Yes	Yes	5% (inoculum)	Yes
5	S-109	Ex-situ	Yes	Yes	5% (inoculum)	Yes
6	S-43	Ex-situ	Yes	Yes	5% (inoculum)	Yes
7	S-43	Ex-situ	Yes	Yes	5% (inoculum)	Yes
8	S-109	Ex-situ	Yes	Yes	0	No
9	S-109	Ex-situ	Yes	No	0	No
10	S-43	Ex-situ	No	No	5% (inoculum)	Yes

Notes:

<sup>1</sup> Source location S-109 is known to contain higher levels of TPH (on the order of 8,800 ppm), and source location S-43 is known to contain lower levels TPH (on the order of 600 ppm).

<sup>2</sup> Methanol was applied to ex-situ plots 4, 5, 6 & 7 by pouring one liter into the piping manifold on day 0 and every 14 days thereafter. Methanol was applied to Plot # 10 by diluting one liter with a few gallons of water and then spraying it over the soil.

### 3.5 Field Operations

The major components involved in the staging of this treatability study were as follows:

- design and construction of treatment and control plots in which the defined experiments were carried out. Subsection 3.5.1 describes the plot design and its construction.
- production of the bulk inoculum. Subsection 3.5.2 describes the procedures that GML implemented to produce the inoculum on-site.
- prescreening sampling, analysis and preparation of the PCB contaminated test soils, blending with bulk inoculum or substrate and placement in the respective treatment or control plots. Subsection 3.5.3 discusses the soil presampling and analysis, preparation and inoculation/blending procedures used.
- plot maintenance. Subsection 3.5.4 describes the manner in which GML maintained the experimental plots through the course of the study.
- equipment and facilities needed to support the treatability study, which are discussed in

Subsection 3.5.5.

- decontamination and waste disposal activities which are discussed in Subsection 3.5.6, and
- collection and analysis of representative samples and collection of physical and operational data. Section 3.6 presents a discussion on sample/data collection and analysis.

### **3.5.1 *Plot Design and Construction***

The ten ex-situ treatment and control plots were staged on top of the paved area immediately adjacent to the main building (the enclosed warehouse type structure) near the main entrance, on the Beede site. The following procedures was used to construct the ex-situ plots.

On the paved surface two layers of 10-mil (or thicker) polyethylene liner was laid out over an area of about 15 feet by 60 feet. The ex-situ plots were constructed using five discarded but clean chipboard (wooden) crates that were available at the Beede site. Each wooden crate was about 4 feet wide, 3 feet tall and 6.7 feet long. First the top cover of each crate was removed. Next, wooden (chipboard) planks were used to divide each of the five 6.7-foot long open boxes into two compartments, each approximately 4 feet by 3.3 feet, thus creating a total of ten experimental chambers. The inside surfaces of each chamber were then secured with two layers of 10-mil plastic. In each plot that was designated to receive passive aeration two lengths (approximately 12 feet) of 4 inch diameter corrugated perforated pipe were laid across the plot in a manner that formed a “U” shape, with the two ends of the “U” raised enough so as not to be buried under the soil that was later placed over the pipe. The bottom of the “U” rested on top of the plastic liner at the bottom of the plot. To facilitate free cross-ventilation, multiple rectangular openings were cut about 20 inches above the plot bottom (i.e., 2 inches above the top of the soil surface). For plots with the passive aeration system, the top ends of the “U-shaped” perforated pipe were allowed to stick out of the rectangular openings in the side of the plots to facilitate improved air exchange. Plot # 10 (which was to be left exposed to precipitation) had a solid 4 inch diameter PVC pipe open at the top and slotted and covered on the bottom with perforated landscape material and placed vertically in the lower corner so that it could be used to remove leachate, if necessary. A sloped cover made from chipboard and lumber was constructed for each box to shed rainfall for all plots except for #10 which was left uncovered. The original design which called for greenhouse type covers made with polyethylene sheeting was redesigned using solid chipboard which made for a stronger roof structure. This redesign, however, resulted in less solar heat gain over the duration of the test. The boxes, which were not originally designed to hold the pressure of soil and water, were reinforced by wrapping each box with two steel bands. Each box was then placed on two pallets and placed on two layers of 10 mil polyethylene sheeting.

### **3.5.2 *Inoculum Production***

Based on information from GML, at its Middlesex, Vermont facility, GML took a liter of its proprietary nutrient fortified suspension and inoculated it with a few microliters of its customized consortium of microbes. After allowing the one liter suspension to incubate for about 48 hours, it was further split into six to eight equal parts and used in turn to inoculate six to eight five-gallon carboys filled with the same nutrient fortified suspension. After allowing the suspensions to incubate for about 48 hours, the 30 or 40 gallons of inoculum suspension were ready to be transported to the Beede site. The carboys were transported to the site in a refrigerated truck by road.

On August 8, 1998, personnel from Green Mountain Labs began the preparation of the microbial substrate. Two 150 gallon open top livestock watering basins were used to prepare the substrate. The key ingredients were rolled oats, ground pine needles, nutrients and bacterial culture. A perforated garden hose that was placed in the bottom of each tub and covered with perforated landscape fabric was attached to an air compressor to serve as an aerator to help maintain aerobic conditions in the substrate mixture. Next, both tubs were filled with a total of 700 pounds of dry rolled oats and wetted with 30-40 gallons of water to which nutrients including ferric chloride had been added. The two basins were inoculated with a solution of the microbial consortia at approximately 16:00 on August 8, 1998. On August 9, approximately 600 pounds of pine needles were ground up using a bagging lawn mower and were then mixed into the inoculated wetted oats in the two basins using hand held garden hoes. The pine needles had been sitting in closed trash cans in the sun for several days and had begun to compost. The mixture was covered with plywood. Observation of the emergence of the bubbles at the substrate surface indicated that aeration was occurring but the distribution throughout the basins was not uniform suggesting that both aerobic and anaerobic regions may have existed within the basins contents. Care was taken to mix the substrate thoroughly prior to adding it to the soil.

In addition to the inoculated substrate, a separate uninoculated batch of substrate consisting of rolled oats, ground pine needles, and nutrients and ferric chloride solution was mixed in a plastic trash can on August 9 to be used in test plot #3.

### **3.5.3 Soil Preparation and Inoculation**

On August 3, 1998, under the supervision of SAIC field personnel, excavation of soil from the previously identified candidate source area was initiated. The soil excavation and screening and the sampling of the excavated soil piles was completed on August 4, 1998. The excavation was performed by personnel from Sanborn, Head & Associates' (engineering contractor for NHDES) a subcontractor, TWM. Soil sampling was conducted by SAIC personnel. A field engineer from Sanborn Head & Associates was also present at the site to help coordinate the soil excavation activities.

#### ***Soil Selected for Excavation***

Based on data from the Beede Waste Oil site remedial investigation two sites had been selected to serve as the source of the soil to be used in the treatability test. These two site were each associated with a specific soil sample. These soil samples are designated as "S-109" and "S-43." The following is a description of the soil excavation and preparation procedure.

#### ***Preparation of S-109 Soil***

An area roughly 20 ft by 20 ft (centered on the S-109 sample location) was marked off for excavation. A polyethylene liner was placed adjacent to the excavation area and a Reed shaker screen with one-inch openings was placed on the liner. A backhoe was then used to dig soil, initially from within 18 inches of the surface, and dropped into the shaker. The goal was to screen discrete batches of approximately two cubic yards of soil and then stop excavation and transport the soil to separate polyethylene liners laid out nearby. The soil was dropped into separate piles of roughly one half of the backhoe bucket per pile using the large loader bucket on the back of the backhoe. Due to initial communication problems, the first batch was larger than planned and consisted of approximately five cubic yards placed in 11 piles. A total of 15 batches were screened and placed on three 20 ft by 100 ft liners. Batches 12 through 15 were excavated from a depth of approximately 18 to 24 inches within the same hole. The piles were then covered and

marked with stakes that identified the batch with a number starting with "1" and then a letter starting with "A" to designate individual piles within each screened batch. A total of 62 individual piles were prepared in this manner.

### ***Preparation of S-43 Soil***

An area roughly 12 ft by 12 ft (centered on the S-43 sample location) was marked off for excavation. The soil was excavated, screened, and placed on a single 20 ft by 100 ft polyethylene liner in the same manner as for the S-109 soil except that the piles were increased in size to the full contents of large backhoe bucket. A total of five screened batches were processed. The batches were numbered 16 through 20 and resulted in a total of 15 soil piles. These piles were marked, sampled, and covered in a similar manner as the S-109 soil was.

### ***Pre-Screening Soil Sampling***

Separate soil samples were collected from each pile. Grab samples were collected from several locations around the surface of the soil pile using a stainless steel spoon. The grab samples were mixed by hand in a stainless steel bowl. A representative sample of the composited sample was then placed in a small plastic container for subsequent analysis using an on-site gas chromatograph. After the sampling was completed the soil piles were then covered with a sheet of polyethylene to protect the soil from rainfall.

### ***On-Site Soil Analysis***

A gas chromatograph was set up in the on-site trailer to be used by Raul Sanchez of GML to perform a field screening of the samples collected from the soil piles for PCB Aroclor 1248. The PCB Aroclor 1248 data (based on the on-site analysis) for the staged soil piles was examined by SAIC field staff. Piles with the greatest concentrations of PCBs were selected for further processing. Piles were selected to ensure that sufficient soil volume was available to conduct the treatability test.

### ***Final Soil Preparation and Inoculation***

On August 10 and 11, 1998 a field crew from TWM returned to the site to perform the final soil handling activities. On August 10, 1998, the selected S-109 soil piles were picked up with the backhoe and placed on a polyethylene liner and were then mixed using the small bucket of the backhoe. A cement mixer was placed on the liner adjacent to the soil pile and used in order to obtain more thorough mixing. Once the soil was mixed with the backhoe, the soil was then transferred to the cement mixer using shovels. In order to estimate the soil volume per mixer batch a five gallon bucket was filled with soil and placed in the mixer. A total of eight buckets (40 gallons) was needed to fill the mixer to the maximum operating volume. In order to simplify operating procedures it was decided that each soil plot would receive three mixer volumes for an estimated total volume of 120 gallons or 0.6 cubic yards. Note that a rough measurement of the soil after placement resulted in a volume of 150 gallons or 0.74 cubic yards. In order to ensure that the controls were handled in a similar manner as the other test plots, the soil was also placed in the cement mixer and mixed prior to placement in the test plots even though no additional materials were added. To prevent cross contamination in the mixer, the soil to be placed in the control soil plots was processed first. Once the two controls, plots #8 and #9, were filled, soil preparation operations ceased for the day.

On August 11, 1998, soil preparation and placement in the soil plots was resumed. No substrate had been used to this point. At the request of Green Mountain personnel, the volume of substrate added to each plot was set at a volume that would maximize the volume added, ensuring that most of the prepared substrate was utilized. This was determined to be equivalent to one and one



half five-gallon buckets full per mixer batch which as described above was estimated to be 40 to 50 gallons in volume. Using a scale, a five gallon bucket of soil was determined to weigh 66 pounds. Assuming that the substrate has a density near that of water, the substrate application rate was estimated to be 12.7% by weight and 18.8% by volume on a wet basis. This was two to three times the application rate of 5% that was specified in the treatability study test plan. The developer, during setup, produced more substrate than needed and did not want to waste any. The test plots were filled in a sequence that minimized cross-contamination of the microbial organisms. Because the S-43 soil was not included in any of the uninoculated plots, this soil was processed last in the sequence.

Plot #3 was filled with soil that had been mixed with the uninoculated substrate. The procedure for mixing the substrate was to first put 1.5 5-gallon bucket fulls of substrate into the cement mixer and then fill the remaining volume of the mixer with soil. The mixer was operated long enough to ensure that the material was well mixed. Observation of the soil substrate mixture indicated that the substrate which consisted of a sticky oat and pine needle mixture, was broken into clumps of 1-2 inches or less in diameter. The remaining plots containing inoculated substrate and S-109 soil were then filled in this manner using three full cement batches per plot. In order to prevent cross contamination of plot #3 with inoculated soil, a plywood cover with plastic sheeting draped over the edge was placed over plot #3 when the adjacent plot was filled.

At the request of NHDES upon completion of the filling of the plots needing S-109 soil, a 55-gallon drum was filled with some of the remaining S-109 soil and set aside for potential use by another party (Ttanks). The remaining S-109 soil was returned to the excavation hole and the selected soil piles from the S-43 area were transferred to the liner for processing. As was done with the S-109 soil the S-43 soil was first mixed using the small bucket of the backhoe and was then mixed with inoculated substrate in the cement mixer and placed in the remaining soil plots. The last plot (#10) was filled on August 10, and the plots were watered by GML staff. Shortly thereafter, one liter of pure methanol was added to plots #4, #5, #6, #7, and #10. For plots #4, #5, #6, and #7 one half of each liter was poured down into each of the two corrugated vent pipes using a funnel with a three foot tube attached to ensure that the methanol was placed near the bottom. For plot #10 the methanol was dribbled across the surface of the soil.

#### **3.5.4 Plot Maintenance**

Once the treatment and control plots were setup, the only maintenance required was periodic irrigation of the soils. GML was solely responsible for this activity and irrigated the plots on an as-needed basis at a frequency that it deemed necessary to ensure that the plots are maintained in proper conditions. A logbook was kept by GML to document visits, observations, moisture content, irrigation time and rate, etc.

#### **Water Source**

Water to be used for cleaning of equipment and watering of the soil plots was obtained from an on-site potable water well. Water was obtained by placing a submersible pump in the well and pumping the water through garden hoses into two 150 gallon basins used to prepare the inoculated substrate. In order to prevent cross contamination with live microorganisms the basins were first washed and then filled with a dilute chlorine solution and left to stand overnight. The basins were then drained, rinsed, and then filled again with well water. After several hours they were drained and then filled with well water and were then ready for use. Water was drawn from the basins using a submersible sump pump placed in one of the basins. When needed, water was pumped from

the well to the basins using a submersible pump.

### **3.5.5 Support Equipment and Facilities**

Typical support equipment and facilities that were required during the course of the treatability study at the Beede site were:

- 110V electrical power supply,
- potable water, for plot irrigation and decontamination of earth handling and sampling equipment,
- a trailer, to set up a temporary on-site analytical laboratory for analysis of screened soil samples, as well as for the SAIC and GML field staff to conduct administrative and clerical activities, and to take rest breaks,
- portable toilet, and
- earth handling equipment, such as a backhoe with a front-end loader, a mechanical shaker screen (like a Reed-Screen-All) and a cement mixer.

NHDES, through its on-site contractor, SHA, provided for or facilitated the availability of support equipment and facilities. SAIC, with the assistance of SHA, installed a pump at the deep well (6" O.D.) existing at the site for potable water.

### **3.5.6 Decontamination, Waste Disposal and Contingency Plan**

During and at the completion of the treatability study, equipment was decontaminated by washing with water/alconox or other suitable means (e.g. steam or pressure washing) before being moved off-site.

All investigation derived waste (IDW) such as, personal protective gear and other solid hazardous waste generated during the treatability study (e.g., cinder blocks, plastic liners, manifold PVC) was placed in approved 55 gallon drums, labeled appropriately and stored on-site where indicated by NHDES. Similarly, liquid wastes (e.g., washwater, methanol and hexane used in sample preparation) were placed in separate 55 gallon drums, labeled appropriately, and staged as directed by NHDES for ultimate disposal.

Analytical and toxicity data generated for the soils in the various plots remaining at the end of the treatability study were used by NHDES to determine the suitability of the soils for return to their respective original source locations on the site. If it was deemed suitable, then NHDES, through its on-site contractor, SHA, provided for or facilitated the return of the soil to its original source area or any other staging area on the site it determined to be appropriate. However, if NHDES determined that the soils were NOT suitable for return to their respective original source locations, then it arranged for these soils to be drummed in appropriate 55-gallon drums and staged on-site for subsequent disposal and/or processing.

## **3.6 Sample Collection and Analysis**

The primary objective of this treatability study was to measure the changes in the PCB concentrations in the various treatments and controls over the course of the study by obtaining soil samples from each plot biweekly. SAIC was responsible for obtaining and preparing all samples necessary to accomplish the objectives stated in Section 3.3 and obtaining all the supporting analyses.

The Test Plan (for this treatability study) dated July 1998, provided a detailed sampling plan, which included the number and type of samples to be collected, collection frequencies, and the analyses required. Based on the prescribed test plan, five soil grab samples from each plot per each of the five sampling events (for a total of 250, excluding duplicate and QC samples) were collected for total PCB-Aroclor and other analyses. The five sampling events were conducted on Days 0, 14, 28, 42 and 56. The SITE program offered to split samples with GML during each sampling event. However, GML requested split soil samples only from the Day 0 (or the baseline) and Day 14 event. The following discussion provides a summarized account of the sample collection effort that was undertaken for the treatability study.

### ***Sample Sequence***

Within each sample plot five grab samples were collected; one was from the center and four from near each corner approximately 6 inches from either side and in a location that would not be impeded by the buried passive air vents. The first grab sample was collected from the westernmost corner (closest to the drum storage area). The sequence then proceeded from corner to corner in a clock-wise manner and with the fifth or last grab from the plot being from the center.

### ***Plot Sequence***

In order to minimize cross-contamination during sampling, the plots were sampled in a sequence of less treatment to more treatment and secondly less soil contaminants to more soil contaminants. Following this rule the plots were sampled in the following order:

- Plot 9 (Control - no treatment)
- Plot 8 (Control - no treatment)
- Plot 3 (uninoculated substrate added)
- Plot 6 (inoculated substrate added, soil with lower PCB and TPH)
- Plot 7 (inoculated substrate added, soil with lower PCB and TPH)
- Plot 10 (inoculated substrate added, soil with lower PCB and TPH)
- Plot 1 (inoculated substrate added, soil with higher PCB and TPH)
- Plot 2 (inoculated substrate added, soil with higher PCB and TPH)
- Plot 2 (inoculated substrate added, soil with higher PCB and TPH)
- Plot 4 (inoculated substrate added, soil with higher PCB and TPH)
- Plot 5 (inoculated substrate added, soil with higher PCB and TPH)

### ***Grab Samples***

Each soil grab sample was collected from the soil plots using a 2 ¾ inch diameter coring sampler with a cone shaped nose. The samples were collected by pushing the sampler downward until the sampler touched the liner at the bottom of the plot or when the sampler failed to move farther with direct pressure but no twisting. The sampler was then withdrawn so as not to damage the liner. Sufficient sample volume was collected from each grab except for plot 10 which is discussed separately below. On subsequent sampling episodes, more than one core sample was collected at each grab sample location when additional sample volume was needed.

### ***Plot #10***

Due to heavy rainfall the night before collection of Day 0 samples, Plot #10 had become filled with water that extended to the top of the soil. The consistency of the Plot #10 samples was of a slurry and during the initial attempt to use the 2 ¾ inch sampler with the cone tip, the sample would drop out of the sampler as soon as the tip broke the water surface upon retrieval. Attempts to use the same sampler with a butterfly tip also failed. Attempts to use other diameter samplers and covering the vent hole in the top of the samplers (at the time of retrieval) also failed. Ultimately, the Plot #10 grab samples were collected using a stainless steel spoon by digging down no more than 6 inches.

### ***Grab Sample Processing***

The contents of the sampler were deposited in a decontaminated stainless steel bowl and was then thoroughly mixed using a decontaminated stainless steel spoon. Once mixed, a portion was transferred into sample bottles including a split for Green Mountain Lab.

### ***Composite Sample***

After all appropriate grab sample bottles were filled, a composite sample was produced in a decontaminated bowl by combining equal amounts of material from each of the remaining grab samples left in their respective bowls. An unused sample bottle served as the measuring device to ensure equal volumes were collected from each grab. This was done starting with the bowl that contained the least sample and if this first volume did not fill the bottle completely then the volume collected from all of the other grabs was reduced accordingly. This combined soil became the composite sample for the plot. The composite sample was then thoroughly mixed using a decontaminated stainless steel spoon. The mixed composite sample was then transferred to the appropriate sample containers. All remaining sample was then returned to the appropriate treatment plot and the sample boreholes were physically collapsed using one of the stainless steel spoons. All sampling equipment was decontaminated between the sampling effort for each plot.

### ***Sampling Equipment Decontamination Procedures***

In response to advice from Green Mountain Labs personnel, a modification of the first washing step involving a dilute chlorine solution was added to the sampling equipment decontamination procedures to reduce contamination with any live microorganisms growing in the sampled media. The modification involved replacing the first soak with tap water with a 15 -minute soak in a solution of 25% chlorine bleach in tap (well) water. In addition the first step of scraping off the equipment with a brush was modified to a rinsing step using well water and pressurized garden sprayers. The following sequential procedure was used to decontaminate sampling equipment and utensils:

- Rinse off gross soil particles with tap water using pressure sprayer,
- soak and scrub inside and out, for 15 minutes using a 25% chlorine bleach solution in the first tub,
- soak and scrub inside and out, withalconox solution in the second tub,
- rinse with tap water in the third tub,
- final rinse distilled water from a squeeze bottle,
- rinse with methanol from a squeeze bottle,
- finally, rinse with hexane from a squeeze bottle, and then,
- allow it to air dry on top of a flat surface covered with clean aluminum foil.

### ***Decontamination of Sample Boring Device Between Grab Samples Within the Same Plot***

Due to the limited number of sampling devices available and time required to perform the full decontamination procedure between each grab, it was decided that, for grab samples within a given soil plot, only a gross decontamination would be performed on the sample boring device used to collect the soil sample. This decision is based on the knowledge that the contents of each soil plot were well mixed at the beginning of the test. The gross decontamination steps used on the sample boring device between grabs within each soil plot are as follows:

- Rinse off gross soil contamination using pressure sprayer and tap water,
- wipe the inside and outside of the boring device with a clean paper towel.

### ***Water in Treatment Plots***

On August 12, 1998 water was observed covering approximately one half of the surface of plot #10. Plot #10 was the only uncovered treatment plot and it had rained approximately 2 inches (as measured in the 5-gallon bucket left out the night before) during a heavy thunderstorm the night

before. It was suspected that additional rainwater was driven by the wind off the side of the roof of the adjacent plot, into plot #10. Raul Sanchez of GML was informed of this development. He said there were no plans to remove the water in plot #10 and so it was left as is.

While sampling plot #4 water was also observed at a depth of approximately 4 inches from the bottom. The observation was made by looking down into the holes left behind when the grab sampler was removed. Raul Sanchez of GML was also informed of this observation.

### ***Temperature Measurement***

On August 12 the first treatment plot temperature measurements were made using a bi-metal type temperature gauge in which the temperature sensitive portion is encased in a approximately 3-foot long thin metal rod. Two temperature measurements were made per plot. The first was made by inserting the rod straight down into the center of the treatment plot. The gauge was allowed to stabilize for approximately two minutes before reading. The second reading was made by inserting the rod straight down approximately 4-6 inches in from the side of the treatment plot in the middle of the side between the location of the first and second grab samples.

### **DAY "14" SAMPLING**

On Day 13, August 25, 1998, SAIC staff arrived on-site to prepare for the Day 14 sampling to be conducted the next day.

The following day, August 26, 1998 (Day 14) soil sampling was conducted following the same procedures established on August 12 (Day 0). As specified in the sampling plan the list of analyses to be conducted on the composite samples was less than those analyzed for Day 0 samples. The following changes to the Day 0 sampling procedures were made:

- Due to water in the plot, the coring device would not retain sufficient volume while sampling plot #4. Grabs 2-5 were collected using a spoon in a similar manner as was done for plot #10.
- At the request of GML staff, the number of split samples collected was reduced from five to one to one per plot. This change was made to determine the treatment effectiveness, modify nutrient addition and because of budget constraints. The split sample was taken from the composite sample for each plot.

### **Day "28" Sampling**

On Day 27, September 8, 1998, SAIC staff arrived on-site to prepare for the Day 28 sampling to be conducted the next day.

The following day, September 9, 1998 (Day 28), soil sampling was conducted following the same procedures established on August 12 (Day 0). As specified in the sampling plan the list of analyses to be conducted on the composite samples was the same as those analyzed for Day 0 samples with the exception for metals and toxicological analyses.

### ***Soil Gas Sampling***

Measurements of the oxygen content of the soil gas in the treatment plots were conducted on September 11, 1998. The measurements were made using a thin stainless steel tube attached to an oxygen gas meter using vinyl tubing. The stainless steel tube was equipped with a threaded rod that could be inserted into the tube its entire length. Appendix A presents the procedures for soil gas sampling that were used. With the exception of the control plots #8 and #9, all of the treatment

plots exhibited reduced levels of oxygen with the lowest reading being 5.8%.

#### *Leachate Sample Collection*

Samples of the water were collected from Plots #1, #4, #5, #6, and #7. However, the shipping cooler was damaged during shipment and resulted in the breakage and loss of both sample bottles from Plot #1. In between sample collection at each plot several liters of tap water were flushed through the sample tubing to prevent gross cross contamination of the samples.

#### **Day “56” Sampling**

On Day 55, October 6, 1998, SAIC staff arrived on-site to prepare for the Day 56 sampling to be conducted the next day.

The following day, October 7, 1998 (Day 56), soil sampling was conducted following the same procedures established on August 12 (Day 0). As specified in the sampling plan the list of analyses to be conducted on the composite samples was the same as those analyzed for Day 0 samples with the exception for metals. Upon completion of the sampling effort the air compressor was turned off and disconnected.

## **Section 4.0**

### **Results and Discussion**

#### **4.1 Introduction**

As noted in Section 1, this project was not carried to completion (i.e., through a field demonstration) due to strong indications that the technology did not perform effectively or as anticipated by GML, the developer, in spite of efforts by all stakeholders to overcome the problems. Nevertheless, data collected through the course of the eight-week field treatability study (August through October 1998) is summarized and discussed in this section.

#### **4.2 Soil Characterization**

As discussed in Section 3, based on the soil PCB concentration data available from previously conducted remedial investigation (RI) at the Beede site, two areas that were known to be potentially contaminated with PCBs, RI sample locations S109 and S43, were chosen as the soil source areas for the treatability tests. However, in order to ensure that a sufficient quantity of soil with reasonably high PCB concentration was available from both areas, the soils from locations S109 and S43 were carefully excavated, screened and staged in small piles, sampled and analyzed on-site, as discussed in Section 3.

Based on the results of on-site analysis of the soil excavated from the S109 area, the PCB Aroclor 1248 concentrations ranged from 24 mg/Kg to 2,350 mg/Kg, with an average concentration of 669 mg/Kg and a median concentration of 238 mg/Kg. Of the S109 derived soil piles used to constitute the bulk of the test soil used for the study the PCB Aroclor 1248 concentrations ranged from 154 mg/Kg to 2,350 mg/Kg, with an average concentration of 977 mg/Kg and a median concentration of 1,003 mg/Kg.

Based on the results of on-site analysis of the soil excavated from the S43 area, the PCB Aroclor 1248 concentrations ranged from 47 mg/Kg to 288 mg/Kg, with an average concentration of 118 mg/Kg and a median concentration of 105 mg/Kg. Of the S43 derived soil piles used to constitute the bulk of the test soil used for the study the PCB Aroclor 1248 concentrations ranged from 93 mg/Kg to 288 mg/Kg, with an average concentration of 154 mg/Kg and a median concentration of 112 mg/Kg.

#### **4.3 Performance Data**

##### **4.3.1 *PCB Data for Treatments and Controls***

As stated in Section 3.3, the primary objectives of this field treatability study were 1) to establish the applicability of the GML treatment process to the Beede site and (2) to determine its effectiveness in biodegrading the primary target contaminants, PCBs, in soil which also contained lead and other organic contaminants including TPHs. To accomplish these objectives, a series of experiments, as described in Section 3.4, consisting of a few variations of GML's biotreatment process and controls were carried out over an eight-week period from August through October of 1998 at the Beede site. The key evaluation criteria for these objectives was the total PCB concentrations in the soil that was subjected to the various treatments and controls. Due to budgetary constraints the majority of the soil samples collected through the course of the study were analyzed for the PCB Aroclor contents using the EPA Method 8082. However, a limited

number of composite samples from the Days 0, 28 and 56 sampling events were analyzed for congener specific PCBs using a combination of EPA Methods 1668 and 680. Congener specific analyses were performed to determine if biodegradation altered the Aroclor 1248 pattern. If PCBs were to undergo biodegradation, the original Aroclor patterns would no longer prevail and thereby mislead one to believe that all PCBs have undergone biodegradation when in fact only a few of the more readily degradable congeners may have biodegraded. The rationale was, that if one or more of the GML treatments did indeed achieve or came significantly close to achieving the treatment goal of reducing the total PCB concentration to less than 1 mg/Kg, then the congener-specific PCB analysis of the composite samples from the respective treatment plot would provide a more definitive confirmation of such treatment performance.

Summary results of PCB Aroclor analysis are presented in Table 4.2-1. These results, which are based on at least five grab samples per plot per each of the five sampling events, clearly indicate that irrespective of the treatment or control applied to the test soil the PCB concentrations (as Aroclor 1248) remained unchanged after eight weeks of treatment duration. Furthermore, the PCB concentration ranges (or the 90% confidence intervals for each plot per event) were found to be fairly tight, thus reducing the uncertainties of the analytical findings (of the lack of performance). The relative tightness of the confidence intervals could be attributed to fact that the target soils and the bulk inoculum (or the non-inoculated substrate) were thoroughly homogenized prior to use in the respective plots, thus reducing the variability in PCB concentrations across a given plot.

Summary results of the congener-specific PCB analyses are presented in Table 4.2-2 and the homologue-specific analyses are presented in Table 4.2-3. These results also suggest that no specific or noteworthy treatment effect could be attributed to any of the treatments or controls.

GML was provided with split samples from the baseline event Day 0, and Day 14, however GML only analyzed samples from the baseline event. In addition, the SITE Program made all of its analytical results available to GML for review. Based on the results of their own sample analysis (for PCB Aroclor) and the SITE Program results, Mr. Raul Sanchez of GML (the President) conceded during an October 1998 teleconference with the EPA Technical Project Manager (Dr. Ronald Lewis), the NHDES Innovative Technology Coordinator (Mr. Robert P. Minicucci, II), and the SAIC Work Assignment Manager (Mr. Dan Patel) that their efforts (through the field treatability tests) had failed to demonstrate the treatment effect of their PCB Bioremediation process. Mr. Sanchez stated that he and his technical team (including Dr. Tony Rutkowski, the chief microbiologist) had carefully reviewed the treatment procedures and methods employed prior to and during the course of the treatability testing. Although they could not pin point the exact cause of the system's non-performance, GML believed that the primary reason may have been the method in which the co-metabolite source material was applied. Mr. Sanchez elaborated that during their earlier laboratory trials, they had use concentrated pine needle extract as the source for terpenes - the desired co-metabolite compounds. However, for the field tests, they had used shredded pine needles. According to Mr. Sanchez, the lack of readily available co-metabolite compounds to the treatment system within the inoculated soil may have been a key detrimental factor.

Subsequently, the EPA SITE program and the NHDES agreed to give GML another opportunity to demonstrate its technology's capability in degrading PCB in the Beede soil, but at a much smaller laboratory scale. In September 2000, GML carried out a limited number of preliminary bench-scale tests, at the Middlebury College in Middlebury, Vermont to reestablish the viability of its process. At the conclusion of the bench-scale tests, GML conceded that, at best the tests were inconclusive and at worst had failed. The project was terminated at that time.



Table 4.2-1 Summary of PCB Aroclor Data for the Various Treatments and Controls

Source Soil	Plot# Description of Experiment	Statistic	PCB Aroclor 1248 Concentrations				
			Day 0	Day14	Day28	Day 42	Day 56
S-109	Plot 1 with • Inoculum at 5 % weight of soil, • passive aeration through corrugated perforated piping, • sloped plywood cover, and • <b>no</b> methanol	Average	<b>220</b>	<b>254</b>	<b>250</b>	<b>267</b>	<b>231</b>
		90% Confidence Interval	187 - 253	238 - 270	239 - 261	238 - 296	202 - 260
		Median	208	249	254	269	221
S-109	Plot 2 with • Inoculum at 5 % weight of soil, • <b>no</b> passive aeration through corrugated perforated piping, • sloped plywood cover, and • <b>no</b> methanol	Average	<b>207</b>	<b>225</b>	<b>256</b>	<b>267</b>	<b>216</b>
		90% Confidence Interval	183 - 231	222 - 228	239 - 272	193 - 341	200 - 232
		Median	213	225	264	239	210
S-109	Plot 3 with • Non-microbial substrate at 5 % weight of soil, • passive aeration through corrugated perforated piping, • sloped plywood cover, and • <b>no</b> methanol	Average	<b>224</b>	<b>223</b>	<b>240</b>	<b>261</b>	<b>205</b>
		90% Confidence Interval	210 - 238	207 - 239	205 - 275	210 - 312	188 - 222
		Median	222	223	223	230	211
S-109	Plot 4 with • Inoculum at 5 % weight of soil, • passive aeration through corrugated perforated piping, • sloped plywood cover, and • methanol	Average	<b>254</b>	<b>219</b>	<b>229</b>	<b>222</b>	<b>207</b>
		90% Confidence Interval	203 - 305	210 - 228	214 - 244	213 - 231	191 - 223
		Median	235	221	222	224	205
S-109	Plot 5 (a duplicate of Plot 4) with • Inoculum at 5 % weight of soil, • passive aeration through corrugated perforated piping, • sloped plywood cover, and • methanol	Average	<b>214</b>	<b>247</b>	<b>237</b>	<b>240</b>	<b>228</b>
		90% Confidence Interval	191 - 237	226 - 268	227 - 247	227 - 253	220 - 236
		Median	205	255	232	236	233

Table 4.2-1 Summary of PCB Aroclor Data for the Various Treatments and Controls (continued...)

Source Soil	Plot# Description of Experiment	Statistic	PCB Aroclor 1248 Concentrations				
			Day 0	Day14	Day28	Day 42	Day 56
S-43	Plot 6 with • Inoculum at 5 % weight of soil, • passive aeration through corrugated perforated piping, • sloped plywood cover, and • methanol	Average	<b>77</b>	<b>89</b>	<b>81</b>	<b>87</b>	<b>77</b>
		90% Confidence Interval	72 - 82	74 - 104	75 - 87	83 - 91	73 - 81
		Median	77	84	78	89	78
S-43	Plot 7 (a duplicate of Plot 6) with • Inoculum at 5 % weight of soil, • passive aeration through corrugated perforated piping, • sloped plywood cover, and • methanol	Average	<b>72</b>	<b>80</b>	<b>74</b>	<b>81</b>	<b>72</b>
		90% Confidence Interval	62 - 82	74 - 86	67 - 81	77 - 85	68 - 76
		Median	73	81	74	81	71
S-109	Plot 8 with • <b>No</b> inoculum or substrate, • passive aeration through corrugated perforated piping, • sloped plywood cover, and • <b>no</b> methanol	Average	<b>223</b>	<b>236</b>	<b>249</b>	<b>237</b>	<b>225</b>
		90% Confidence Interval	190 - 255	224 - 248	207 - 291	208 - 266	211 - 239
		Median	212	233	236	237	230
S-109	Plot 9 with • <b>No</b> inoculum or substrate, • <b>no</b> passive aeration through corrugated perforated piping, • sloped plywood cover, and • <b>no</b> methanol	Average	<b>190</b>	<b>227</b>	<b>237</b>	<b>246</b>	<b>230</b>
		90% Confidence Interval	154 - 226	184 - 270	202 - 272	227 - 265	209 - 251
		Median	196	217	227	257	223
S-43	Plot 10 with • Inoculum at 5 % weight of soil, • <b>no</b> passive aeration through corrugated perforated piping, • <b>no</b> shelter or cover, and • methanol	Average	<b>68</b>	<b>69</b>	<b>77</b>	<b>81</b>	<b>79</b>
		90% Confidence Interval	62 - 74	60 - 78	73 - 81	73 - 89	72 - 86
		Median	65	71	76	80	82

Table 4.2-2 Summary of PCB Congener Data for the Various Treatments and Controls

PCB Congener ID #	PCB Concentration (ug/Kg)					
	Plot # 1			Plot # 2		
	Day 0	Day 28	Day 56	Day 0	Day 28	Day 56
1	14	11	7	15	12	13
3	1	1	1	1	4	1
8	1,600	1,600	1,200	1,600	450	1,400
15	8,000	8,900	7,400	6,600	3,800	7,100
18	30,000	14,000	13,000	27,000	13,000	11,000
28	57,000	42,000	18,000	51,000	34,000	20,000
37	9,200	6,000	4,800	7,800	6,900	5,700
44	170	16,000	15,000	14,000	12,000	15,000
49	10,000	9,800	14,000	8,800	8,500	14,000
52	19,000	15,000	17,000	18,000	13,000	17,000
66	14,000	14,000	14,000	12,000	8,500	13,000
70	18,000	16,000	16,000	13,000	17,000	17,000
74	9,600	7,900	7,800	6,600	15,000	7,700
77	1,200	1,300	1,300	980	1,500	1,400
81	110	140	130	95	130	110
87/115	2,300	2,600	2,980	1,500	3,100	2,990
90/101	4,500	6,000	2,400	3,200	6,900	3,000
99	2,200	2,400	1,500	1,700	2,700	1,600
110	4,100	4,600	2,200	3,300	6,600	2,300
119	120	130	89	110	130	69
118	3,000	3,400	2,900	2,600	3,100	2,500
123	460	420	110	370	380	39
105	2,400	2,000	1,900	1,800	2,300	1,500
114	240	220	170	200	200	150
126	19	14	18	14	11	12
151	170	130	130	100	86	120
128/167	240	260	248	210	230	211
138/158	1,200	1,100	1,130	1,000	1,100	1,110
149	730	760	149	690	580	660
153/168	560	580	901	540	570	881
156	130	110	120	110	110	98
157	30	26	26	26	24	22
169	ND	ND	ND	ND	ND	ND
170	130	100	100	110	110	86
177	20	15	20	16	15	16
180	210	180	200	180	170	160
183	49	40	42	43	38	34
184	ND	ND	ND	ND	ND	ND
187	100	78	81	92	70	63
189	420	4	4	4	4	3
201	38	28	4	3	31	34
202	8	7	8	7	6	7
194	22	18	18	18	17	15
195	10	9	10	8	7	8
206	17	16	18	13	17	16
207	2	1	1	1	2	1
209	5	4	4	4	4	4
Total Congener Concentration	218,155	177,901	147,118	185,461	162,408	148,133

Table 4.2-2 Summary of PCB Congener Data for the Various Treatments and Controls  
(continued...)

PCB Congener ID #	PCB Concentration (ug/Kg)					
	Plot # 3			Plot # 4		
	Day 0	Day 28	Day 56	Day 0	Day 28	Day 56
1	13	5	9	9	30	5
3	1	1	1	1	2	1
8	1,200	1,300	110	1,300	4,500	1,200
15	5,100	8,600	6,300	5,100	25,000	7,100
18	25,000	19,000	12,000	30,000	19,000	11,000
28	25,000	47,000	16,000	50,000	50,000	20,000
37	6,400	74,000	6,000	7,800	7,700	5,200
44	12,000	21,000	14,000	13,000	15,000	14,000
49	7,600	15,000	13,000	8,800	11,000	12,000
52	14,000	23,000	16,000	16,000	17,000	15,000
66	11,000	13,000	15,000	12,000	9,000	15,000
70	110,000	24,000	13,000	14,000	17,000	18,000
74	6,700	21,000	7,600	7,600	15,000	6,600
77	860	2,000	1,300	980	1,300	1,300
81	84	180	110	37	130	110
87/115	1,300	3,800	2,860	1,300	3,000	3,000
90/101	2,600	7,000	2,900	2,500	5,400	2,800
99	1,400	3,500	1,600	1,400	2,700	1,700
110	2,600	7,500	2,200	2,500	5,800	2,200
119	95	160	79	77	120	80
118	2,200	4,800	2,400	2,300	3,300	3,100
123	400	560	80	490	400	68
105	1,600	3,300	1,700	1,700	2,200	1,700
114	190	310	150	180	200	150
126	13	18	15	14	14	15
151	87	140	120	71	100	120
128/167	190	310	213	190	200	191
138/158	750	1,500	1,070	720	1,100	1,100
149	480	880	620	440	640	710
153/168	370	820	831	330	570	871
156	97	170	98	100	100	94
157	23	35	23	24	24	23
169	ND	ND	ND	ND	ND	ND
170	99	160	89	96	110	79
177	15	23	16	14	14	16
180	160	280	160	160	170	170
183	37	58	34	38	38	36
184	ND	ND	ND	ND	ND	ND
187	74	110	64	72	72	71
189	3	6	3	4	4	3
201	29	41	34	31	31	29
202	7	10	7	7	7	8
194	16	27	15	16	17	15
195	8	12	8	8	8	9
206	12	28	15	13	16	15
207	1	2	1	1	1	1
209	4	6	4	4	4	4
Total Congener Concentration	141,000	238,000	138,000	181,000	218,000	145,000

Table 4.2-2 Summary of PCB Congener Data for the Various Treatments and Controls  
(continued...)

PCB Congener ID #	PCB Concentration (ug/Kg)					
	Plot # 5			Plot # 6		
	Day 0	Day 28	Day 56	Day 0	Day 28	Day 56
1	9	16	6	1	1	0
3	1	1	1	ND	ND	ND
8	1,400	2,700	1,500	74	160	55
15	6,200	17,000	8,900	1,850	7,450	2,000
18	36,000	22,000	13,000	4,600	2,700	2,100
28	60,000	66,000	21,000	16,000	15,000	7,450
37	9,100	12,000	5,300	2,750	2,850	2,750
44	17,000	26,000	12,000	5,550	6,350	8,250
49	10,000	16,000	14,000	3,800	4,800	6,550
52	19,000	2,600	18,000	3,905	7,350	8,200
66	14,000	14,000	16,000	6,000	4,150	7,600
70	17,000	28,000	17,000	6,250	8,000	8,500
74	9,200	24,000	8,300	3,400	7,000	4,350
77	1,100	2,200	1,500	450	630	705
81	43	190	110	28	57	59
87/115	1,300	4,000	3,640	555	1,300	1,480
90/101	2,800	9,900	4,300	1,250	3,250	1,600
99	1,400	3,800	2,000	590	1,200	770
110	2,600	82,000	2,700	1,200	2,800	1,305
119	120	170	94	26	61	313
118	2,600	5,100	3,000	1,200	1,700	1,750
123	450	600	70	110	180	44
105	1,900	3,100	2,000	795	1,020	1,100
114	200	310	160	58	92	78
126	16	19	17	5	7	9
151	86	140	140	68	61	87
128/167	200	310	236	105	155	173
138/158	860	1,500	1,220	560	730	920
149	540	920	690	315	430	475
153/168	400	820	1,001	255	395	691
156	110	160	110	57	74	84
157	26	33	26	14	16	19
169	ND	ND	ND	ND	ND	ND
170	110	140	95	59	71	71
177	17	22	19	8	9	13
180	180	240	180	91	105	130
183	40	58	41	21	23	29
184	ND	ND	ND	ND	ND	ND
187	81	100	79	41	39	23
189	4	5	3	2	3	3
201	30	38	34	10	16	21
202	7	10	8	2	3	5
194	17	24	18	8	9	10
195	8	11	10	3	4	6
206	15	28	20	6	8	9
207	1	2	1	0	1	1
209	4	6	4	2	2	2
Total Congener Concentration	216,000	272,000	159,000	62,000	78,000	68,000

Table 4.2-2 Summary of PCB Congener Data for the Various Treatments and Controls  
(continued...)

PCB Congener ID #	PCB Concentration (ug/Kg)					
	Plot # 7			Plot # 8		
	Day 0	Day 28	Day 56	Day 0	Day 28	Day 56
1	ND	ND	ND	13	4	2
3	ND	ND	ND	1	1	ND
8	22	45	32	1,600	1,400	780
15	1,300	2,900	1,900	7,800	8,000	4,000
18	3,100	1,500	1,200	26,000	20,000	22,000
28	13,000	11,000	3,600	54,000	44,000	19,000
37	2,400	2,500	2,000	7,400	7,300	6,300
44	3,900	5,200	4,600	13,000	18,000	18,000
49	2,700	4,000	4,500	8,000	13,000	22,000
52	4,800	6,200	4,200	15,000	20,000	15,000
66	5,200	3,500	5,200	11,000	9,800	22,000
70	3,700	6,600	4,500	14,000	19,000	16,000
74	2,200	6,000	1,900	7,400	17,000	11,000
77	390	520	510	1,000	1,500	1,500
81	28	54	34	120	120	96
87/115	370	1,200	1,050	1,000	2,800	2,240
90/101	900	2,800	1,000	2,200	7,000	2,000
99	470	1,000	670	1,000	2,600	1,400
110	930	2,200	800	2,100	5,700	1,700
119	22	55	29	60	120	77
118	850	1,500	1,100	2,500	3,400	2,700
123	90	140	29	210	400	57
105	660	860	680	2,000	2,300	1,900
114	48	75	51	180	200	210
126	4	6	6	9	13	12
151	53	44	63	120	100	120
128/167	80	120	115	160	210	201
138/158	460	610	710	940	1,100	1,088
149	270	380	410	570	670	630
153/168	220	320	541	440	600	881
156	45	58	53	90	110	94
157	10	13	12	20	23	23
169	ND	ND	ND	ND	ND	ND
170	46	57	47	97	110	81
177	6	7	8	15	16	17
180	70	82	85	160	170	160
183	16	16	18	37	39	37
184	ND	ND	ND	ND	ND	ND
187	31	32	1	76	81	70
189	2	2	2	3	4	3
201	7	16	12	23	28	28
202	2	3	3	5	7	7
194	6	7	6	16	18	15
195	3	3	4	7	8	9
206	4	8	6	14	16	16
207	ND	1	ND	1	1	1
209	1	2	1	4	4	3
Total Congener Concentration	48,000	62,000	42,000	180,000	207,000	173,000

PCB Congener ID #	PCB Concentration (ug/Kg)					
	Plot # 9			Plot # 10		
	Day 0	Day 28	Day 56	Day 0	Day 28	Day 56
1	14	4	2	ND	ND	ND
3	1	1	ND	ND	ND	ND
8	1,200	1,700	710	44	46	31
15	5,800	9,800	4,000	2,600	2,600	1,400
18	27,000	21,000	22,000	4,700	2,000	2,800
28	56,000	48,000	15,000	21,000	11,000	6,400
37	7,900	7,100	6,500	3,800	2,200	2,000
44	14,000	18,000	30,000	6,400	5,500	6,900
49	8,100	13,000	24,000	4,000	3,900	5,000
52	16,000	20,000	22,000	7,300	6,100	4,600
66	12,000	9,900	23,000	7,000	3,200	5,900
70	15,000	20,000	18,000	6,900	6,300	4,600
74	8,300	16,000	13,000	4,000	5,500	3,400
77	1,000	1,500	1,600	540	520	550
81	40	140	99	45	52	36
87/115	1,200	3,200	2,000	530	1,000	840
90/101	2,500	7,600	2,100	1,200	2,500	880
99	1,300	2,900	1,300	600	900	670
110	2,300	6,100	1,600	1,200	2,000	720
119	86	140	86	46	50	29
118	2,900	3,600	2,800	160	1,300	1,100
123	400	440	58	20	150	23
105	2,100	2,200	1,800	1,100	800	710
114	180	210	150	99	72	56
126	13	12	14	7	6	7
151	160	98	130	100	45	64
128/167	210	220	202	150	120	117
138/158	920	1,200	1,090	720	570	590
149	600	740	620	410	340	310
153/168	460	640	891	300	310	471
156	100	110	98	83	61	54
157	25	24	24	19	13	12
169	ND	ND	ND	ND	ND	ND
170	100	110	82	75	56	45
177	16	17	18	11	7	8
180	180		170	120	81	82
183	40		39	26	18	18
184	ND		ND	ND	ND	ND
187	78		75	50	33	31
189	4		3	3	2	2
201	26		28	16	12	12
202	6		7	4	3	3
194	18		16	10	7	6
195	7		9	5	3	4
206	15		16	8	8	6
207	1		1	1	1	0
209	4		4	2	2	1
Total Congener Concentration	188,000	216,000	195,000	75,000	59,000	50,000

Table 4.2-3 Summary of PCB Homologue Data for the Various Treatments and Controls

PCB Homologue	PCB Concentration (ug/Kg)														
	Plot # 1			Plot # 2			Plot # 3			Plot # 4			Plot # 5		
	Day 0	Day 28	Day 56	Day 0	Day 28	Day 56	Day 0	Day 28	Day 56	Day 0	Day 28	Day 56	Day 0	Day 28	Day 56
Total monoCB	16	12	8	17	17	15	14	5	11	10	35	5	11	18	7
Total diCB	17,000	18,000	22,000	16,000	14,000	15,000	13,000	17,000	13,000	12,000	48,000	12,000	14,000	31,000	15,000
Total triCB	190,000	120,000	90,000	170,000	110,000	97,000	150,000	140,000	97,000	170,000	140,000	95,000	180,000	180,000	110,000
Total tetraCB	150,000	130,000	140,000	130,000	120,000	140,000	100,000	150,000	140,000	120,000	130,000	130,000	150,000	220,000	140,000
Total pentaCB	29,000	31,000	21,000	24,000	35,000	21,000	21,000	54,000	20,000	20,000	32,000	22,000	23,000	50,000	25,000
Total hexaCBI	4,000	3,700	3,900	3,500	3,400	3,600	2,700	4,900	3,500	2,500	3,400	3,700	3,000	4,800	4,000
Total heptaCB	720	580	670	610	560	540	540	900	560	540	550	570	600	820	640
Total octaCB	120	96	110	99	98	99	94	140	99	98	100	94	96	130	110
Total nanoCBI	24	22	25	19	24	22	17	38	20	18	22	20	20	37	260
Total Homologues	391,000	303,000	278,000	344,000	283,000	277,000	287,000	358,000	274,000	325,000	354,000	263,000	371,000	487,000	295,000

  

PCB Homologue	Plot # 6			Plot # 7			Plot # 8			Plot # 9			Plot # 10		
	Day 0	Day 28	Day 56	Day 0	Day 28	Day 56	Day 0	Day 28	Day 56	Day 0	Day 28	Day 56	Day 0	Day 28	Day 56
Total monoCB	1	1	0	ND	ND	ND	15	4	2	15		2	ND	ND	ND
Total diCB	3,900	6,250	2,450	1,800	3,600	2,200	17,000	17,000	9,200	15,000		9,100	3,500	3,300	1,800
Total triCB	38,000	37,000	27,500	29,000	28,000	20,000	160,000	120,000	130,000	170,000		130,000	54,000	28,000	26,000
Total tetraCB	45,000	57,000	70,500	36,000	48,000	39,000	110,000	280,000	150,000	130,000		210,000	56,000	47,000	45,000
Total pentaCB	8,000	16,500	11,350	6,300	14,000	7,400	17,000	33,000	19,000	20,000		19,000	11,000	13,000	6,600
Total hexaCBI	1,800	2,350	2,850	1,500	1,900	2,200	3,000	3,500	3,600	3,300		3,600	2,400	1,800	1,900
Total heptaCB	310	345	455	240	280	290	540	600	560	580		590	390	270	280
Total octaCB	30	49	64	25	45	38	78	93	87	88		90	53	39	36
Total nanoCBI	8	11	12	6	11	8	19	23	21	21		22	10	10	8
Total Homologues	97,000	120,000	115,000	75,000	96,000	71,000	308,000	454,000	312,000	339,000		372,000	127,000	93,000	82,000



## Section 5.0

### QUALITY ASSURANCE

#### 5.1 QA Summary

Quality Assurance (QA) may be defined as a system of activities the purpose of which is to provide assurance that defined standards of quality are met with a stated level of confidence. A QA program is a means of integrating the quality planning, quality assessment, quality control (QC), and quality improvement efforts to meet user requirements. Included are all actions taken by project personnel, and the documentation of laboratory and field performance. Typically, project-specific QA/QC requirements are specified in a Quality Assurance Project Plan (QAPP). The objective of the quality assurance program is to reduce measurement errors to agreed upon limits and to produce results of acceptable and known quality. The QAPP specifies the necessary guidelines to ensure that the measurement systems remain in control and provides detailed information on the analytical approach to ensure that data of acceptable quality is obtained to achieve project objectives.

For the preliminary evaluation of the GML PCB Bioremediation Process, a Treatability Test Plan (TP), instead of a QAPP, was developed, approved and implemented. The following sections provide information on the use of data quality indicators, limitations on data use and a summary of the QC analyses associated with project measurements.

#### 5.2 Data Quality Indicators

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

Precision is generally measured by laboratory/matrix spiked sample duplicates and field sample duplicates. In the case of duplicates, precision is evaluated by expressing, as a percentage, the difference between results of the sample and sample duplicate results. The relative percent difference (RPD) is calculated as:

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

For three or more measurements, precision is evaluated by the standard deviation of the multiple measurements relative to the mean, i.e. the relative standard deviation (RSD), according to the following equation:

$$RSD = (SD/X_{avg}) \times 100$$

Where SD is the standard deviation and  $X_{avg}$  is the average of the multiple concentrations.

To determine and evaluate accuracy, known quantities of select target analytes were spiked into selected field samples. Equipment used to provide data for this project was tested for accuracy through the analysis of calibration check standards and laboratory control samples. To determine matrix spike recovery, the following equation was applied:

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

where  $C_{ss}$  = Analyte concentration in spiked sample  
 $C_{us}$  = Analyte concentration in unspiked sample  
 $C_{sa}$  = Analyte concentration added to sample

To determine the % recovery of LCS analyses or spiked blanks, the equation below was used:

$$\% \text{ Recovery} = \frac{\text{Measured Concentration}}{\text{Theoretical Concentration}} \times 100$$

Another important aspect of assessing data quality is completeness. Completeness is a measure of the amount of valid data produced from the total effort compared to the total amount of data determined to be necessary to meet project objectives.

To determine if a measurement is valid, it must be reproducible and comparable. Comparability expresses the extent to which one data set can be compared to another. To generate comparable results, standard methods that are widely accepted along with strict analytical and field protocols were used. These methods were clearly specified in the TP and reviewed before samples or data were collected.

While several precautions were taken to generate data of known quality through the control of the measurement system, the data must also be representative of true conditions. Representativeness refers to the degree with which analytical results accurately and precisely reflect actual conditions present at the locations chosen for sample collection.

### 5.3 Conclusions and Data Quality Limitations

A review of the critical sample data and associated QC analyses was performed to determine whether the data collected were of adequate quality to provide proper evaluation of the project's technical objectives. The only critical measurement for this technology demonstration was the PCB Aroclor analysis of the soil grab samples from the various treatment and control experiments. These samples were collected on Days 0, 14, 28 42 and 56 of the field treatability testing. The results of the measurements designed to assess the data quality objectives for these analyses are summarized and discussed below.

Accuracy for the analysis of PCB Aroclor in the soil grab samples collected from the various treatment and control plots was assessed by the analysis of Matrix Spike Duplicate Samples (MS/MSD). As it turned out, the predominant PCB constituent in the target soil matrix was Aroclor 1248. Therefore, the MS/MSD samples were spiked with Aroclor 1248. For the most part (i.e., for 22 out of the 24 pairs of MS/MSDs) the critical compounds met the data quality objectives with average % recovery values ranging between 71 - 123%, which was within the established control limits of 70 - 130 % for the Aroclor analysis.

Precision was evaluated through the Aroclor 1248 analysis of field duplicate samples as well as laboratory paired matrix spiked duplicates (MS/MSDs). For the most part (i.e., for 22 out of the 25 pairs of field duplicate samples) the % RPD values were within specified control limit of 30%, with an average % RPD of 16.3. For the most part (i.e., for 22 out of the 24 pairs of laboratory MS/MSDs samples) the % RPD values were within specified control limit of 30%, with an average % RPD of 19.7.

Detection limits as reported met objectives as stated in the TP.

Comparability was achieved through the use of EPA approved analytical methods and protocols and verified by the validation of analytical data, which indicated that most TP and method-specified criteria were met.

Completeness objectives were met for the treatability study phase sampling and analytical program.

## **Section 6.0**

### **REFERENCES**

SITE Program - Test Plan (July 1998) for Treatability Study of Green Mountain Laboratories, Inc.'s Bioremediation Process for Treatment of Soils Contaminated with PCBs at the Beede Waste Oil/Cash Energy Superfund Site, Plaistow, New Hampshire.

Rutkowski, Anthony A., U.S. Patent - 6,096,531, August 1, 2000, Methods and composition for bioremediation.