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Application of a Bimetallic Treatment System (BTS) for PCB Removal from Older Structures on DoD Facilities

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FINAL TECHNICAL REPORT FOR:
Application of a Bimetallic Treatment System (BTS) for PCB Removal from Older Structures on DOD Facilities

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LIST OF ACRONYMS

AMTS	Activated Metal Treatment System
ASTM	American Society for Testing and Materials
BAAP	Badger Army Ammunition Plant
Badger	Badger Army Ammunition Plant
BTS	Bimetallic Treatment System
CAS	Columbia Analytical Services
CCAFS	Cape Canaveral Air Force Station
CoC	Chain of Custody
Dem/Val	Demonstration/validation (demonstrate/validate)
DoD	Department of Defense
DOT	Department of Transportation
DQI	Data Quality indicators
DQO	Data Quality objectives
ECD	Electron Capture Detector
EDD	Electronic Data Deliverables
EPA	Environmental Protection Agency
ESTCP	Environmental Security Technology Certification Program
ft	Feet
ft^2	Square feet
g	gram
GC	Gas Chromatography
HASP	Health and Safety Plan
hr	hour
HVLP	High Volume/Low Pressure
ID	Identifier
ITL	Integrated Transfer Launch
JEGS	Japan Environmental Governing Standard
KSC	Kennedy Space Center
LUT	Launch Umbilical Tower
MDL	method detection limit
mg/kg	Milligram per kilogram
mg/L	milligrams per liter
Mg/Pd	Magnesium/Palladium
mL	Milliliters
MS	Mass spectrometry
MSFC	Marshall Space Flight Center
NASA	National Aeronautics & Space Administration

LIST OF ACRONYMS (CONT'D)

NAVFAC	Naval Facilities Engineering Command
NFESC	Naval Facilities Engineering Service Center
NM	Non metal
OSHA	Occupational Safety and Health Administration
PARCC	Precision, Bias, Accuracy, Representativeness, Completeness, and Comparability
PCB	Polychlorinated Biphenyl
PPE	Personal Protective Equipment
psi	Pounds per square inch
QA/QC	quality assurance / quality control
QAPP	Quality Assurance Project Plan
RPD	Relative percent difference
SAP	Sampling and Analysis Plan
SIL	Silicone roof sealant
SRF JRMC	US Naval Ship Repair Facility and Japan Regional Maintenance Center
TA	Treatment Area
TSCA	Toxic Substances Control Act
UCF	University of Central Florida
µg/L	Micrograms per liter
USAF	United States Air Force
VIB	Vertical Integration Building
VOC	Volatile Organic Compounds
VP	Vinyl Polymer
wt	weight

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Executive Summary:

Polychlorinated biphenyls (PCBs) are a group of synthetic aromatic compounds with the general formula $C_{12}H_{10-x}Cl_x$ that were historically used by industry because of their excellent dielectric properties and their resistance to heat and chemical degradation. PCBs were commonly used as additives in paints and asphaltic-based adhesives that were subsequently applied to Department of Defense (DoD) structures. Prior to 1979, PCBs were extensively used in industrial paints, caulking material and adhesives, as their properties enhanced structural integrity, reduced flammability and boosted antifungal properties. Numerous DoD facilities have older metal structures upon which paints containing PCB were applied. These painted structures may present risks to human health or the environment because of inhalation or ingestion concerns as the paint degrades and becomes brittle and can become air-born or impact soil, surface water or groundwater. To date, no reliable methods are available that allow for the removal of PCBs from painted structures/equipment without damaging the coating or the structures/equipment itself.

The overall objective of this project was to refine and deploy a safe, cost-effective, *in situ* treatment method for the removal and destruction of PCBs found on DoD structures. This overall project objective was addressed by the following specific objectives:

1. Determine the protocol for formulating bimetallic treatment system (BTS) for site-specific conditions to enhance applicability to various PCB-containing materials found across numerous DoD facilities while maximizing safety and efficacy with the ultimate goal of reducing PCB concentrations to less than 50 mg/kg.

2. Demonstrate the effectiveness of BTS on a wide range of actual contaminated structures at three DoD facilities. Evaluate the relationships between dose applied, repeated applications and reaction kinetics with the intention of specifically identifying the factors influencing treatment and limiting reaction rates for a specific media (e.g. different painted structures). Evaluate environmental condition effects (temperature and humidity; weathering) and impact of BTS on material appearance and adhesion.

Research and development work at the National Aeronautics and Space Administration Kennedy Space Center NASA-KSC and University of Central Florida (UCF) has led to the development of a bimetallic treatment system (BTS) comprised of elemental magnesium (Mg) coated with a small amount of palladium (Pd) that is utilized in conjunction with a solvent solution capable of donating hydrogen atoms. BTS as a treatment technology has two functions: 1) to extract the PCBs from weathered coating materials and other PCB containing materials such as insulation, rubber gaskets and asphaltic compounds; and 2) to degrade the extracted PCBs. The chemical reductant and catalyst system has been optimized for use in BTS and typically consists of 0.1% Pd on zero-valent or metallic Mg. It is hypothesized that the interaction of the bimetallic Mg/Pd system with a solvent containing available hydrogen moieties (i.e., alcohols) results in the

generation of atomic hydrogen at particular sites on the metal surface. The bound atomic hydrogen is available for reaction with PCB molecules in solution yielding a reductive dehalogenation reaction.

The BTS technology demonstrations were conducted at two DoD facilities: 1) the Vertical Integration Building (VIB) at the Cape Canaveral Air Force Station (CCAFS), Florida; and 2) the Badger Army Ammunition Plant (Badger), Sauk County, Wisconsin. Primary and secondary performance objectives were developed that were evaluated using either qualitative or quantitative performance criteria to determine success. These performance criteria included:

Distribution and Adherence of the BTS One of the qualitative performance objectives is that the BTS applicator is able to evenly distribute the paste on the surface to be treated. The metric was evaluated by assessing the adherence of the BTS to an object in a 0.25 to 0.5 inch layer over the time period of exposure to treated surfaces. BTS was applied using a spray applicator and hand trowel application method. This objective was met although the spray application did not work well in the cold weather implementation.

Adherence of Sealants The metric was evaluated by assessing the adherence of the sealant to the BTS, the ability to apply the sealant evenly over the surface of the paste, and its ability to dry to a non-tacky, non-porous layer that reduced volatilization of BTS solvent. Two sealants were tested: 1) a vinyl polymer (VP) truck bed liner; and 2) a silicone-based roof sealant (Sil). This objective was met using both sealants.

Ease of implementation The ease of use of this technology was evaluated based on our experience in the field. This objective was met with respect to both the ease of handling and applying both the paste and sealant on the various surfaces and locations that were treated.

Reduction in PCB concentrations in treated paint to less than 50 mg/kg A key performance objective is the reduction of PCB concentrations in the treated material to less than 50 mg/kg. This objective was partially met. One application of paste was effective in achieving this target after only one week of treatment in all cases where the starting concentration in the paint was less than approximately 500 mg/kg, especially if the surface being treated was metal and not concrete. In cases where the starting concentrations in the paint were greater than 500 mg/kg, significant reductions (93%) in PCB concentrations were achieved but more than one application of paste is necessary to reduce concentrations below 50 mg/kg.

Reduction in PCB concentrations in BTS paste to less than 50 mg/kg The reduction of PCB concentrations in the paste to less than 50 mg/kg is another key performance objective. This objective was partially met. For the active paste (metal in the paste) if the starting paint concentrations were below roughly 2,500 mg/kg then the concentrations in the paste were less than 50 mg/kg. If the pre-treatment paint concentrations were very high (>20,000 mg/kg) then the active metal paste was not able to degrade all of the PCBs in the paste to below 50 mg/kg although degradation did occur in the paste. Even when Mg/Pd and additional ethanol was added in the laboratory to the active paste that had been exposed to the very high starting concentrations it was not possible to get the concentrations in the paste to below 50 mg/kg after 21 days. For the non-metal paste, which was activated in the lab after removal from the field by the addition of ethanol and the active metal (Mg and acid or Mg/Pd), the concentrations were reduced to below 50 mg/kg for all samples using the acidified ethanol and Mg and/or ethanol and the Mg/Pd.

Follow-on work

Studies conducted at UCF after the project was initiated have shown that the Pd catalyst can be removed from the BTS paste and a small amount of acid added to make a paste that is both less expensive and more reactive. The addition of a small amount of acetic acid to the ethanol significantly increased the rate of PCB degradation. These studies also showed that acidified ethanol with Mg particles was as effective or in some cases more effective than the Mg/Pd particles in non-acidified ethanol at degrading PCBs.

1 INTRODUCTION

1.1 Background

Polychlorinated biphenyls (PCBs) are a group of synthetic aromatic compounds with the general formula $C_{12}H_{10-x}Cl_x$ that were historically used by industry because of their excellent dielectric properties and their resistance to heat and chemical degradation. Although the United States Environmental Protection Agency (EPA) has banned the manufacture of PCBs since 1979, they have been found in at least 500 of the 1,598 National Priorities List (Superfund) sites identified by the EPA. Prior to the EPA's ban on PCB production, PCBs were commonly used as additives in paints and asphaltic-based adhesives that were subsequently applied to Department of Defense (DoD) structures. Prior to 1979, PCBs were extensively used in industrial paints, caulking material and adhesives, as their properties enhanced structural integrity, reduced flammability and boosted antifungal properties. Numerous DoD facilities have older metal structures upon which paints containing PCB were applied. These painted structures may present risks to human or the environment and are considered TSCA-level (Toxic Substances Control Act) waste if disposal is required. Some of the affected structures could be refurbished and utilized for new programs, but because the paint currently on the structures contains PCBs, the DoD may be unable to reuse or even discard these structures without significant cost. To date, no reliable methods are available that allow for the removal of PCBs from painted structures/equipment without damaging the coating or the structures/equipment itself.

In 2004, National Aeronautics and Space Administration (NASA) Kennedy Space Center (KSC) began investigating the potential of using a solvent-based system to remove PCBs found in paints located on a number of structures at three of their operating Centers. This innovative research was initially funded by NASA's Environmental Program Office and NASA's Office of Space Flight. The funding included the preliminary proof of concept laboratory research and the demonstration of a Bimetallic Treatment System (BTS) on parts set aside from NASA's Launch Umbilical Tower at KSC. Additionally, main engine test stands at Marshall Space Flight Center (MSFC) were tested for PCB removal and destruction using BTS. Both of these structures were utilized during the Apollo Program and the test stands at MSFC could potentially be reused by NASA for its future launch vehicle.

This project seeks to demonstrate the application of a BTS to remove and degrade PCBs found on structures and equipment at DoD facilities. The project involved the collection of PCB-containing materials from various DoD structures, followed by the laboratory evaluation of the PCB removal efficiency by BTS for each location. This included determining the number of BTS applications required to reach pre-determined cleanup goals stipulated by the various installations. The work then proceed into the field where the BTS was applied to PCB-containing structures at the Vertical Integration Building (VIB) at the Cape Canaveral Air Force

Station, FL and the Badger Army Ammunition Plant (Badger), Sauk County, Wisconsin. Both sites had PCBs identified in the paint on the structures and equipment, appropriate site conditions and a suitable on-site support network for execution of the Demonstration Validation (Dem/Val).

1.2 Objectives of the Demonstration

The overall objective of this project was to refine and deploy a safe, cost-effective, *in situ* treatment method for the removal and destruction of PCBs found on DoD structures. This overall project objective was addressed by four specific objectives:

1. Determine the protocol for formulating BTS for site-specific conditions to enhance applicability to various PCB-containing materials found across numerous DoD facilities while maximizing safety and efficacy with the ultimate goal of reducing PCB concentrations to less than 50 mg/kg.

2. Demonstrate the effectiveness of BTS on a wide range of actual contaminated structures at three DoD facilities. Evaluate the relationships between dose applied, repeated applications and reaction kinetics with the intention of specifically identifying the factors influencing treatment and limiting reaction rates for a specific media (e.g. different painted structures). Evaluate environmental condition effects (temperature and humidity; weathering) and impact of BTS on material appearance and adhesion.

3. Review BTS application and handling process and develop appropriate on-site safety protocols for institutions to implement during its application, including the handling of any site-specific waste products generated by BTS and the application process. The BTS process itself produces non-toxic waste.

4. Develop full-scale cost and performance reports for DoD facility end-users to utilize when addressing PCBs found on existing structures.

1.3 Regulatory Drivers

Although the EPA has banned the manufacture of PCBs since 1979, they have been found in at least 500 of the 1,598 National Priorities List (Superfund) sites identified by the EPA. Prior to the EPA's ban on PCB production, PCBs were commonly used as additives in paints and asphaltic-based adhesives that were subsequently applied to DoD structures. DoD facilities constructed as early as 1930 utilized PCB-containing binders or PCB-containing paints, which are now leaching into the environment and posing ecological and worker health concerns. In order to dispose of materials containing PCBs in a non-hazardous waste landfill the concentrations must be below 50 mg/kg as per the requirements of TSCA. Disposal of materials containing >50 mg/kg of PCBs in a hazardous waste landfill is expensive and also has the additional and potentially expensive complication of the long-term liability of those wastes. In order for the materials to be recycled and/or reused, which is a preferable option to landfilling from an environmental and economic standpoint, elevated concentrations of PCBs must be reduced in order to allow the materials be handled safely. For instance, scrap metal recovery ovens operating in conformance with 40 CFR 761.72(a) could be used for structures containing PCB concentrations greater than 500 mg/kg. However, the ovens are typically very small, and structures would have to be cut into small sections and a special permit for cutting the structures would be required due to the potential environmental and human exposure risks.

There are few viable options, other than the use of the BTS technology, for removing and degrading PCBs from structures and equipment with coatings such as paint and adhesives utilizing an *in situ* approach. With BTS, PCBs are removed from the structure within hours and are degraded on site to benign end-products. There are no future environmental liabilities associated with off-site disposal and no potential loss of PCB-containing materials to the environment during the removal or treatment process.

2 TECHNOLOGY DESCRIPTION

The following sections provide: an overview of the technology (Section 2.1); technology development (Section 2.2); and advantages and limitations of the technology (Section 2.3).

2.1 Technology Overview

Research and development work at NASA-KSC and UCF has led to the development of a BTS comprised of elemental magnesium (Mg) coated with a small amount of palladium (Pd) that is utilized in conjunction with a solvent solution capable of donating hydrogen atoms. BTS as a treatment technology has two functions: 1) to extract the PCBs from weathered coating materials and other PCB containing materials such as insulation, rubber gaskets and asphaltic compounds; and 2) to degrade the extracted PCBs (Figure 2-1). BTS can be used in a one step process with the paste, including the active metal reductant, being applied to the surface of the material to be treated. The PCBs are extracted from the material being treated by the solvent paste and the PCBs are degraded by the metal reductant within the paste. The BTS can also be use in a two-step process where the solvent paste is applied to the surface to be treated but the paste does not contain the active metal. PCBs are extracted from the material being treated, the paste is removed, and the active metal reductant is added to the paste in a separate container to perform the degradation step. Figures 2-1 and 2-2 are schematics that show these two methods.

One Step Process of Extraction and Treatment in Applied Paste

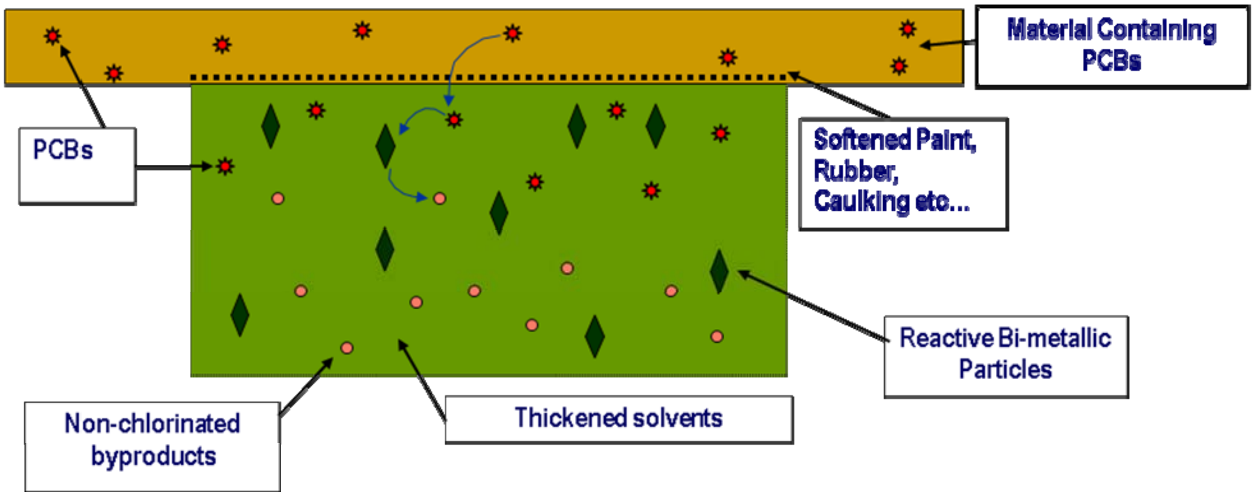


Figure 2-1. Schematic showing the one step process method of applying BTS. The extraction and degradation of the PCBs occurs in this one step using an active (metal-in) paste.

Two Step Process of Extraction of PCBs into Solvent Paste and then Treatment of the Solvent Paste

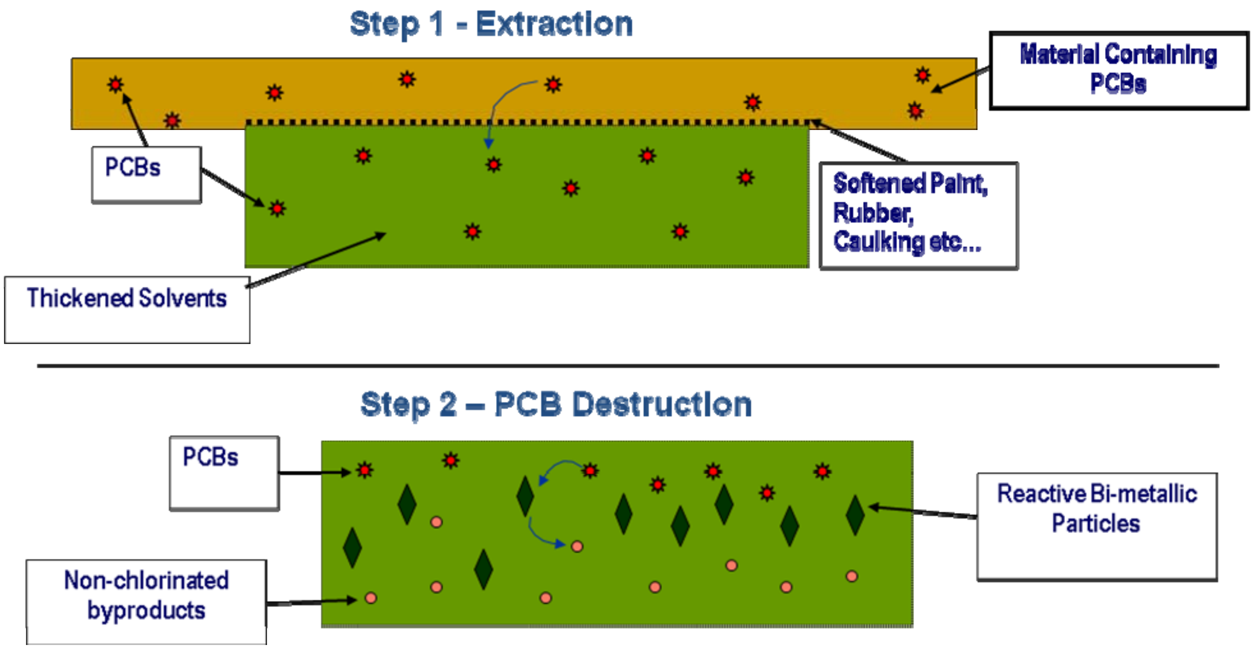


Figure 2-2. Schematic showing the two step process method of applying BTS. In this method the extraction is the first step using an inactive (no metal) paste that extract the PCBs. The paste is then removed and put into a container where reactive metal is mixed into the inactive paste and the degradation step proceeds.

The process for removing PCBs from any type material is accomplished as an independent step to the degradation process. With painted material, the goal is to remove the PCBs from the paint without destroying the paint and collect the PCBs in an environmentally friendly solvent. For some materials being treated, the removal of PCBs may cause a degradation of the materials (e.g., loss of flexibility in caulking materials) making reuse of the material difficult. The research conducted prior to this demonstration indicated that the removal of PCBs can usually be accomplished within 24 hours of application of the solvent to the material to be treated. PCBs are extremely hydrophobic (organophilic) and will partition strongly into the BTS from hardened paint or binder material. The solvent assists in opening, but not destroying, the paint’s polymeric lattice structure, allowing pathways for PCB movement out of the paint and partition into the solvent. A number of solvent systems were tested by NASA-KSC and UCF and can be used in the BTS formulation.

Aroclor is a PCB mixture produced from approximately 1930 to 1979. It is one of the most commonly known trade names for PCB mixtures and is believed to be the most common mixture of PCBs found in coating materials. During the 1950 to 1960 time frame, PCBs were added to paint formulations as drying oils and plasticizers or softening agents in concentrations that range from 10–12% PCBs (100,000– 120,000 mg/kg) to 20–30% PCBs (200,000–300,000 mg/kg). Data provided to EPA indicate that PCBs have been found in dried paint at concentrations that range from <1 mg/kg to 97,000 mg/kg (EPA 1999).

The chemical reductant and catalyst system developed by the team is manufactured using a mechanical alloying method. The chemical reductant and catalyst system has been optimized for use in BTS and typically consists of 0.1% Pd on zero-valent or metallic Mg. The Mg/Pd bimetallic reductant and catalyst system is a potent dechlorination reagent capable of removing the chlorine from high concentration solution of chlorocarbons in minutes (Engelmann, 2003). Literature suggests the degradation end-product for the dehalogenation of all PCB Aroclor mixtures is a non-chlorinated biphenyl molecule, which is a benign end-product (Doyle, 1998). The magnesium metal is a powerful reducing agent and reacts with water to form hydrogen gas (H_2) and magnesium hydroxide (Avedesian, 2005). Palladium is a well-documented hydrogenation catalyst that chemisorbs molecular hydrogen, weakening the bond between the hydrogen atoms, forming atomic hydrogen bound to the Pd surface (Tsuji, 2004). It is hypothesized that the interaction of the bimetallic Mg/Pd system with a solvent containing available hydrogen moieties (i.e., alcohols) results in the generation of atomic hydrogen at particular sites on the metal surface. The bound atomic hydrogen is available for reaction with PCB molecules in solution yielding a reductive dehalogenation reaction. The proposed reaction mechanism is shown below:

$$\mathrm{Mg} + 2\ \mathrm{H_2O} \rightarrow \mathrm{Mg\ (OH)_2} + \mathrm{H_2}$$

$$\mathrm{H_2\ (Pd\ catalyst)} + \mathrm{RCl} \rightarrow \mathrm{RH} + \mathrm{HCl}$$

In this reaction, RCl represents the PCB molecule and one of its chlorine atoms, and the RH represents the PCB molecule with the chlorine removed and replaced by a hydrogen atom.

2.2 Technology Development

In 2004, NASA KSC began investigating the potential of using a solvent-based system to remove PCBs found in paints on a number of structures at three of their operating Centers. This innovative research was initially funded by NASA's Environmental Program Office and NASA's Office of Space Flight. The funding included preliminary proof of concept laboratory research and the demonstration of a bimetallic treatment system (BTS) on painted parts containing PCBs set aside from NASA's Launch Umbilical Tower (LUT) at KSC. LUT paint chips were immersed in the water-in-solvent emulsion containing BTS for various periods of time ranging from two hours to four days. These test evaluated the effective rate of PCB removal from the paint, and the rate of degradation of the PCBs once inside the emulsion droplet and in contact with the bimetallic reactant. Data indicated that significant if not complete PCB extraction and degradation from paint chips containing as high as 11,000 mg/kg total PCBs could be achieved with as little as 48 hours of exposure.

Additionally, main engine test stands at Marshall Space Flight Center (MSFC) were tested for PCB removal and destruction using BTS.

The original BTS formulation developed NASA-KSC and UCF was intended for application to structures that were dismantled and could be treated by immersion of parts of the structures into a liquid treatment bath. In order to treat structures that were not scheduled to be demolished, a treatment system capable of being 'painted-on and wiped off' was needed. In response to this need, further formulation work was conducted in 2004/2005 and an improved formulation of BTS was developed and tested in the laboratory at Marshall Space Flight Center (MSFC) in Huntsville, AL using LUT components. This formulation used a thickened paste BTS system rather than a liquid emulsion-based system to allow the BTS paste to be applied directly to structures in their original location. The data from testing of BTS paste suggests that for painted structures, the paste: 1) is an easier and safer form of BTS to manufacture; 2) can be 'painted-on and wiped-off'; and 3) is effective in removing PCBs from the coating material.

Researchers at NASA and UCF demonstrated rapid and complete dechlorination of PCBs in liquid based BST systems described above. Table 2-1 presents results achieved in a liquid BTS system comprised of water and 10% methanol. The formulation was modified by increasing the percentage of methanol to make the solution more organic in nature increase the solubility of PCBs in the solution. Due to safety concerns associated with the generation of hydrogen gas when the Mg/Pd is added to the water, solutions with 100% methanol and ethanol were tested, resulting in similar rates of reduction to previous formulations as shown in Tables 2-2 and 2-3.

As is evident from the data found in Tables 2-1 – 2-3, the degradation of PCBs can be achieved rapidly and completely in the presence of the reductant and catalyst system developed by the NASA/UCF team.

TABLE 2-1: EXPOSURE OF AROCLOR 1260 IN 10% METHANOL-WATER SOLUTION TO 1.0 G MG/PD CATALYST.

Sample Identification	Aroclor 1260 (mg/l)	% PCB Degradation
Extracted Standard (no Mg/Pd added)	5.9	nil
Standard exposed to Mg/Pd 1.0 hr	0.4	92%
Standard exposed to Mg/Pd 4.0 hr	<0.1	>98%
Standard exposed to Mg/Pd 4.0 hr dup	<0.1	>98 %

TABLE 2-2: EXPOSURE OF AROCLOR 1254 IN METHANOL ONLY TO 0.5 g MG/PD CATALYST.

Sample Identification	Aroclor 1254 (mg/l)	% PCB Degradation
Extracted Standard (no Mg/Pd added)	5.5	nil
Standard exposed to Mg/Pd 0.5 hr	0.3	95 %
Standard exposed to Mg/Pd 1.0 hr	<0.1	>98 %
Standard exposed to Mg/Pd 2.0 hr	<0.1	>98 %
Standard exposed to Mg/Pd l 4.0 hr	<0.1	>98 %

TABLE 2-3: EXPOSURE OF AROCLOR 1260 IN ETHANOL-GLYCERIN SOLUTION CONTAINING 1.0 G MG/PD CATALYST.

Sample Identification	Aroclor 1260 (mg/l)	PCB Degradation
Extracted Standard (no Mg/Pd added)	10.6	nil
Standard exposed to Mg/Pd 24 hr	0.4	92 %
Standard exposed to Mg/Pd 4.0 hr	<0.1	>98%
Standard exposed to Mg/Pd 4.0 hr dup	<0.1	>98 %

A variety of solvent formulations can be used to make up the BTS based on the specific type of surface material (i.e., paint, gasket material) being treated. However, the optimal formulation of the BTS from most of the testing done to date uses ethanol as the solvent. The data presented in Tables 2-4 and 2-5, illustrate the ability of BTS to remove PCBs and degrade them from painted structures.

TABLE 2-4. LAUNCH UMBILICAL TOWER (LUT) PAINT BEFORE AND AFTER TREATMENT WITH BTS CONSISTING OF MG/PD, GLYCERIN AND METHANOL (EXPOSURE TIME 24 HRS).

Sample Identification	Initial Concentration Aroclor 1260 (mg/Kg)	After BTS Aroclor 1260 (mg/Kg)	% PCB Removal and Degradation
4696 F1 Stand	110	0.8	83 %
4553 F1 Stand	260	9.7	96%
LUT Red 05/18/05	7.7	0.2	97%

TABLE 2-5. MARSHALL SPACE FLIGHT CENTER PAINT TREATED AND UNTREATED WITH BTS CONSISTING OF MG/PD, GLYCERIN AND ETHANOL (EXPOSURE TIME 24 HRS).

Sample Identification	Initial Concentration Aroclor 1260 (mg/Kg)	After BTS Aroclor 1260 (mg/Kg)	% PCB Removal and Degradation
4696 F1 Stand	4.6	0.8	83 %
4553 F1 Stand	6.3	<0.3	95 %

Additional BTS formulation properties that must be addressed for each site-specific application include viscosity and stability. If the BTS is to be applied to surfaces rather than having parts added to a treatment bath it must be viscous or thick enough to remain where it is applied. Several thickening agents have been tested. Adding a stabilizing agent ensures the solvent in the BTS will not evaporate and leave the Mg/Pd exposed to contact with air. Due to the extreme reactivity of Mg/Pd in the BTS, the choice of thickening agents and stabilizing agents is complex. During BTS formulation testing, a number of reagents were evaluated to ensure the rate of dehalogenation was not inhibited by its addition to the system. **Table 2-6** includes examples of the data obtained with the addition of glycerin as a stabilizing agent and thickener, showing no interferences of this additive on the PCB reduction potential.

TABLE 2-6: EXPOSURE OF AROCLOR 1260 IN ETHANOL WITH AND WITHOUT THE ADDITION OF GLYCERIN.

Sample Identification	Aroclor 1260 (mg/l)	% PCB Degradation
Extracted Standard (no Mg/Pd added)	10.6	N/A
Standard exposed to Mg/Pd 24 hr	<0.1	>99 %
Standard exposed to Mg/Pd 24 hr (duplicate)	<0.1	>99 %
Standard exposed to Mg/Pd with glycerin	<0.1	>99 %
Standard exposed to Mg/Pd with glycerin duplicate	< 0.1	>99 %

In summary, BTS is comprised of a bimetallic reductant and catalyst system containing Mg and Pd incorporated into a solvent that: 1) is capable of extracting PCBs from multiple, thick layers of decades-old paint and from caulking, insulation materials, rubber gaskets and other structural materials containing PCBs; and 2) functions as a hydrogen donation for the degradation of the PCBs in the presence of Mg/Pd. BTS has been shown to treat a wide range of PCB concentrations and achieve low cleanup levels, making the potential application to DoD structures very appealing.

2.3 Advantages and Limitations

A number of options can be considered for disposal of structures containing PCB-laden materials (such as paints). The following is a list of alternative remediation or disposal options that may be considered for PCB impacted structures:

1. Non-Hazardous State-Approved Landfill (performance-based disposal)

PCB concentrations must be less than 50 mg/kg. The concentrations of PCBs in the paint on many structures exceed this criterion. For example, the PCB concentrations on the NASA structures at KSC were much greater with some samples containing more than 50,000 mg/kg. This option can be used for material with < 50 mg/kg of PCBs but does not eliminate long-term potential environmental liability associated with the material.

2. Hazardous Waste Landfill (performance-based disposal)

Hazardous waste landfills can accept material with higher concentrations of PCBs than other landfills but the costs for disposal of material at hazardous waste landfills can be very high and this option may be cost-prohibitive for large structures. This option does not eliminate the long-term environmental liability associated with the material.

3. On-Site Landfill (risk-based disposal)

In some situations it may be possible to construct a landfill on-site near the structure or structures that require disposal. It would be necessary to obtain a State permit for a site-owned and operated landfill (open-ended, risk-based approval) and sufficient area must be available. This option has potential risks associated with leaching of contaminants into soil and groundwater, and still retains all its long-term environmental liability.

4. Smelters (performance-based disposal, decontamination provisions)

Metal may be recovered by recycling that material at metal smelters. The concentrations of PCBs on structures to be smelted must be less than 500 mg/kg. If the structures must be cut into manageable sizes, a special permit is required to perform the cutting operations.

5. Scrap Metal Recovery Ovens (decontamination provisions)

Metal may also be recovered by recycling to Scrap Metal Recovery Ovens operating in conformance with 40 CFR 761.72(a). This option can be used for material containing PCB concentrations greater than 500 mg/kg, however, the ovens are typically very small, and structures would have to be cut into extremely small sections. A special permit for cutting the structures would be required.

6. Physical Removal of Paint

Paint containing PCBs can be removed from surfaces using approaches such as: 1) sandblasting; 2) water blasting; or 3) chemical paint removers and physical scraping. Sandblasting and water blasting may be difficult to control in the open and could result in the release of PCBs into the air or onto surrounding natural media. Chemical paint removers typically contain hazardous chemicals. All these options still require disposal of the PCB-laden material removed from the surfaces.

The BTS technology that was tested in this study provides an effective process to remove PCBs from structures and degrade them without transportation to another location. The main advantages of the BTS technology over other treatment technologies include:

- potential for lower overall costs than alternative approaches in some situations;
- elimination of long-term liabilities because PCBs are destroyed rather than being transferred to another medium;
- ability to destroy PCBs on-site without the need to transport the PCB containing material from the site; and
- ability to treat PCBs without needing to destroy the building or structure so that the option exists for reuse of the building or structure.

The main limitations of using the BTS technology are:

- It may be difficult to effectively and quickly distribute the viscous BTS to all surfaces impacted with PCBs especially if they are on irregular surfaces. If applying to large vertical surfaces in cold temperature where the paste is more viscous it may not be possible to apply BTS using a spray application and will need to be applied using a trowel.
- The application of BTS to paints and binder materials containing PCBs does have the potential to alter the adhesive qualities of the material while removing and degrading the PCBs. It may not always be possible to leave paints in place and have them function as an effective coating material. Paints, caulking materials or binders containing PCBs, may require reapplication of a new coating after the application of BTS.

3 PERFORMANCE OBJECTIVES

Primary and secondary performance objectives were developed that were evaluated using either qualitative or quantitative performance criteria to determine success. The performance objectives are provided in Table 3-1. The primary objectives are discussed in detail in sections 3.1 to 3.5 and the secondary objectives are discussed in Section 3.6.

3.1 Distribution and Adherence of the BTS

One of the qualitative performance objectives is that the BTS applicator is able to evenly distribute the paste on the surface to be treated. The metric was evaluated by assessing the adherence of the BTS to an object in a 0.25 to 0.5 inch layer over the time period of exposure to treated surfaces. BTS paste was applied to surfaces to be treated using two types of application methods and its thickness estimated. The paste was visually inspected periodically (every two to 5 days for the first week and then weekly for up to a month) to determine if it was able to adhere to the surfaces (both vertical and horizontal treated surfaces) by inspecting for leaks, sections of paste pulling away from the surface or bubbling up off the surface.

This objective was met. BTS was applied using a spray applicator and hand trowel application method. In some cases the paste did not adhere well to the vertical painted surfaces but this was due to adhesion issues with the paint (old and friable paint on concrete surface) and not due to the pastes ability to adhere to the surface. The spray applicator did not perform well in the cold weather at Badger (paste thickened in the cold) and most of the treatments were done using a trowel application method. The spray applicator also resulted in a much thinner layer of BTS being applied compared with the trowel application which did affect the treatment ability of the BTS so preference was given to the hand application method. These results are discussed further in Section 6.1.

3.2 Adherence of Sealants

The BTS, once applied, needs to be sealed for the duration of the treatment. The purpose of the sealant is to keep the ethanol-limonene solvent from evaporating. Evaporation of the solvent must be prevented or at least the rate of evaporation reduced so that the paste remains moist. PCB removal from the paint as well as degradation stops if the paste dries. A serried of sealants were tested prior to the ESTCP Demonstrations including: plastic film and aluminum foil (too labor intensive and not effective for large surfaces); a series of water-based latex paints (ultimately formed cracks in the film upon drying and or aging); organic solvent based paints such as urethanes, epoxides, and enamels (performed better than the latex paint, but not effective in maintaining the moisture level in the BTS paste for more than three days); a silicone roof sealant (successful in preliminary tests); and a commercially available vinyl polymer truck bed

TABLE 3-1
PERFORMANCE OBJECTIVES

Performance Objective	Performance Metric	Data Requirements	Success Criteria	Results
Qualitative Performance Objectives				
Ease and versatility of application of BTS	Adhering to object in a 0.25 to 0.50 inch layer	Measuring layer thickness with a calibrated probe and visual imspection of adherence	Ability to apply BTS to different shaped surfaces using different application technique and have it adhere evenly	Criteria met - (see Section 6.1 for details).
Ability to seal BTS after application	Adhering to BTS and dries to a non-tacky, non-porous layer	Samples will be taken every week and or visual and manual inspections conducted to verify BTS solvent content of paste (See Table 3-24 for analytical methods)	Ability to apply surface coating over BTS such that volatilization is minimized	Criteria met - (see Section 6.2 for details).
Paint adherence post BTS application	Impact to paint adherence	Using an ASTM pull-test procedure and visual inspection of paint condition.	BTS will have minimal effect on painted surface. Structure of paint will maintain basic adhesive properties.	Criteria partially met - (see Section 6.3 for details).
Ease of use	Ability of a technician-level individual to use the technology	Feedback from the technicians on usability of the BTS paste and sealant	BTS will be relatively easy to handle and applly in the field with proper operator training	Criteria met - (see Section 6.3 for details).
Quantitative Performance Objectives				
Reduce PCB concentrations in paint	mg/kg PCBs. Remove samples and analyze for PCBs	Pre and post BTS application sampling and analysis (See Table 3-24 for analytical methods)	Reduction in PCB concentrations in treated paint to less than 50 mg/kg in no less than two applications of BTS.	Criteria partially met - dependent on starting PCB concnetrations (see Section 6.4 for details).
Reduce PCB concentrations in paste	mg/kg PCBs. Remove samples and analyze for PCBs	Pre and post BTS application sampling and analysis (See Table 3-24 for analytical methods)	Reduction of PCB levels within BTS to less than 50 mg/kg in no more than 30 days.	Criteria partially met - (see Section 6.5 for details).

liner (successful in preliminary tests). It was decided that a sprayable sealant would be the easiest to apply to cover large surface areas and uneven or complicated (multiple folds or layers such as pipes, pieces of equipment etc…) surfaces.

The metric was evaluated by assessing the adherence of the sealant to the BTS, the ability to apply the sealant evenly over the surface of the paste, and its ability to dry to a non-tacky, non-porous layer that reduced volatilization of BTS solvent. Two sealants were tested: 1) a vinyl polymer (VP) truck bed liner; and 2) a silicone-based roof sealant (Sil). The sealant and underlying BTS paste were inspected every one to two days, depending on location, for the first five days and samples were collected of the paste and sealant weekly for up to a month after application. The sealants ability to adhere to the BTS and to reduce volatilization of the ethanol in the BTS was evaluated by visual inspection (looking for leaks around the edges of the sealant, looking for bubbles or drooping of sealant away from BTS surface) and manual inspection by pressing lightly on the sealed surface to test the fluidity of the paste.

This objective was met using both sealants. Both sealants were able to provide an effective seal to minimize ethanol evaporation from the BTS paste for the three weeks of application. In the cases where there were issues with the condition of the paint (old and friable paint on concrete) there were issues with both the sealant and paste sticking to the surface and the seal failed. However, this had to do with the paint itself being unable to adhere to the surface and not the sealant. In addition, the cold temperatures during the Badger deployment did affect the ability of the sealant to remain flexible and some cracking of the sealant did occur. If possible, it would be preferable to apply BTS in above freezing conditions to avoid the risk of sealant failure. If applications must be done in below freezing temperatures, additional care must be taken to inspect and repair cracks in the sealant as they appear. These results are discussed further in Section 6.2.

3.3 Ease of implementation

The ease of use of this technology was evaluated based on our experience in the field. The implementability was evaluated by the qualitative assessment of the ease with which the operator was trained to handle and apply both the paste and the sealant in the various field situations.

This objective was met with respect to both the ease of handling and applying both the paste and sealant on the various surfaces and locations that were treated. These results are discussed in greater detail in Section 6.3.

3.4 Reduction in PCB concentrations in treated paint to less than 50 mg/kg

A key performance objective is the reduction of PCB concentrations in the treated material to less than 50 mg/kg. The metric was evaluated by collecting samples of the paint pre-BTS application and then weekly for up to a month for analysis of PCB concentrations. This was a destructive analysis and was done on one section of the test area on a weekly basis until all sections had been analyzed.

This objective was partially met. One application of paste was effective in achieving this target after only one week of treatment in all cases where the starting concentration in the paint was less than approximately 500 mg/kg, especially if the surface being treated was metal and not concrete. In cases where the starting concentrations in the paint were greater than 500 mg/kg, significant reductions (93%) in PCB concentrations were achieved but more than one application of paste is necessary to reduce concentrations below 50 mg/kg. These results are discussed further in Section 6.4.

3.5 Reduction in PCB concentrations in BTS paste to less than 50 mg/kg

The reduction of PCB concentrations in the paste to less than 50 mg/kg is another key performance objective. The metric was evaluated by collecting samples of the paste weekly for up to a month for analysis of PCB concentrations. This was a destructive analysis that was done on one section of the test area on a weekly basis until all sections had been analyzed. For BTS that did not contain the activated metals for degradation (non-active paste) a subset of the paste sample was analyzed for PCBs and then the activated metal and, if necessary, additional ethanol, were added to the non-active paste to promote degradation (post-application activated paste). Samples of the post-application activated paste were then analyzed for PCB concentrations.

This objective was partially met. For the active paste (metal in the paste) if the starting paint concentrations were below roughly 2,500 mg/kg then the concentrations in the paste were less than 50 mg/kg with the exception of the 3 weeks samples from Badger Building 6810-11 where concentrations were still slightly above the 50 mg/kg target. If the pre-treatment paint concentrations were very high (>20,000 mg/kg) then the active metal paste was not able to degrade all of the PCBs in the paste to below 50 mg/kg although degradation did occur in the paste. Even when Mg/Pd and additional ethanol was added in the laboratory to the active paste that had been exposed to the very high starting concentrations it was not possible to get the concentrations in the paste to below 50 mg/kg after 21 days.

For the non-metal paste, which was activated in the lab after removal from the field by the addition of ethanol and the active metal (Mg and acid or Mg/Pd), the concentrations were reduced to below 50 mg/kg for all samples using the acidified ethanol and Mg and/or ethanol and the Mg/Pd. Further discussion on this objective is presented in Section 6.5.

3.6 Impact to paint adherence

One of the secondary objectives was to evaluate the impact to the paint adherence after exposure to BTS. The metric was evaluated by visually assessing the condition of the paint after BTS application as well as by using the field test for pull-off strength of coatings (ASTM D3359-02).

When the paint at the Badger test sites was subjected to the ASTM D3359-02 procedure prior to application of BTS, the paint at all the test sites failed. Thus, the effect of the BTS on the integrity of the painted surfaces at Badger could not be assessed.

There were difficulties in using ASTM D3359-02 at the VIB on post-treatment samples due to preparation of the surface prior to testing and ASTM D4541-02 was also used to evaluate the paint adherence for the VIB panels.

This objective was not met in that the BTS negatively affected the adherence and strength of at least the surface layer of paint. These results are discussed further in Section 6.6.

4 FACILITY/SITE DESCRIPTION

The BTS technology demonstrations were conducted at two DoD facilities: 1) the Vertical Integration Building (VIB) at the Cape Canaveral Air Force Station (CCAFS), Florida; and 2) the Badger Army Ammunition Plant (Badger), Sauk County, Wisconsin.

A third demonstration site was evaluated, the ex-USS CHARLES F. ADAMS (hereafter referred to as ADAMS) in Philadelphia, which is part of the Navy Inactive Ships Program. Samples of painted metal, caulking, wire insulation material, and gaskets were collected in February 2007. Laboratory testing of the samples from the ADAMS was conducted to verify the existence of PCBs in the samples obtained and indicated that none of the materials tested contained PCBs at high enough concentrations to warrant a demonstration of the BTS technology. Therefore the funds that were to be used to perform the field demonstration at this site were instead used to develop a methodology to apply a variation of BTS to treat PCB-impacted oil from US Naval Ship Repair Facility and Japan Regional Maintenance Center (SRF-JRMC), located in Yokosuka, Japan.

Section 4.1 and 4.2 present descriptions of the two sites where BTS technology demonstrations were conducted. Section 4.3 presents a description of the site in Japan for which the methodology to modify BTS was developed.

4.1 Vertical Integration Building

The Vertical Integration Building (VIB) was located at the Cape Canaveral Air Force Station, Florida. Additional information on this site is presented below.

4.1.1 Facility/Site Location and Operations

Facility 70500 (VIB) was built in 1964 and was used in support of the United States Air Force (USAF) Titan Missile Program, which ceased launch operations in 2005. The facility is located on the west side of CCAFS, north of the NASA Causeway in the Integrated Transfer Launch (ITL) Area (Figure 4-1). Within the VIB, the primary and secondary stages of the Titan Missiles were mated to the mobile launch stand. The VIB Annexes were constructed in the late 1980s to provide additional facilities for the assembly of the Titan launch vehicles. The entire VIB Facility was scheduled to be dismantled in 2007.

As part of the dismantlement efforts, conventional and/or explosive demolition approaches were planned for the VIB, an approximately 98,990 square foot steel framed facility with an 18 level high bay extending to a height of over 230 feet. For USAF and Occupational Safety and Health Administration- (OSHA-) safe dismantlement, and in preparation for off-site disposal, the steel structure had to be cut into pieces sized to meet restrictions for packaging, transportation and disposal of metal debris. Portions of the paint on the steel in the VIB High Bay area were documented to contain PCBs greater than or equal to 50 mg/kg that were presumably added during manufacture and/or the original construction in 1964.

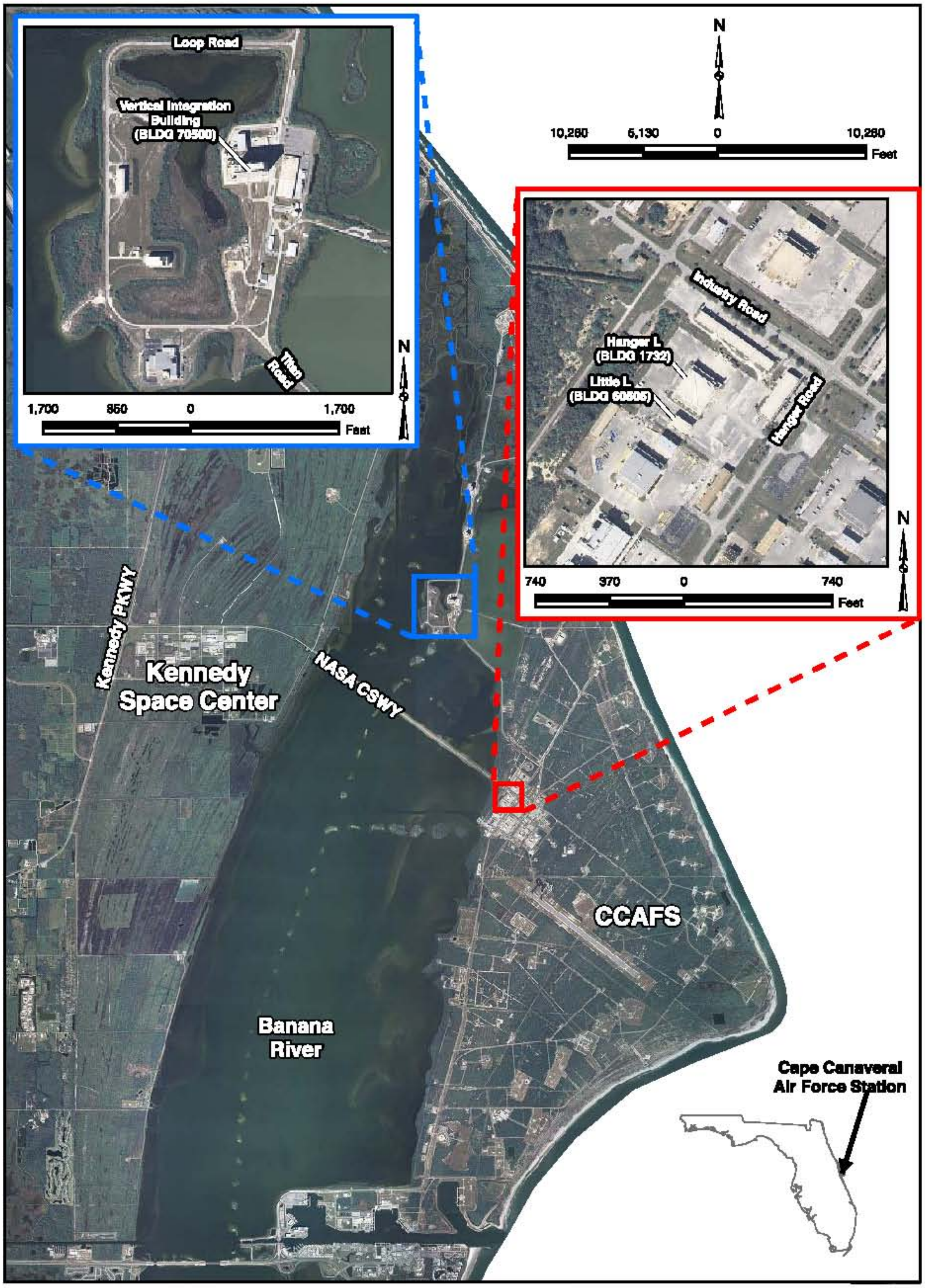
The VIB deactivation included the demolition, recycling, and/or disposal of the entire VIB Facility. As part of the demolition efforts, steel components with PCB paint concentrations in excess of 50 mg/kg were handled as bulk product waste in accordance with EPA TSCA requirements.

4.1.2 Facility/Site Conditions

The entire ITL Area is located on a dredge spoil island bordered by the Banana River to the West and mosquito control lagoons to the East. The building components that are to be treated are from inside the structure and as such have not been exposed to significant weathering due to sun or rain exposure. The components that were treated were removed from the VIB structure and treated within another building at CCAFS so that efforts were made to maintain similar environmental setting.

Prior to demolition activities and in order to determine the presence, distribution, and concentrations of the PCB-containing paints, paint samples were collected from various structures within the VIB and sent for laboratory analysis. PCB paint sampling and analysis was conducted at the VIB in 2005 through 2006 by AMEC for the USAF as part of the planning for demolition activities. The sampling was initiated to evaluate total PCB concentrations in paint throughout the facility prior to demolition and final disposition. The following is a summary of the site layout and sampling activities/PCB results at the VIB.

The VIB paint sampling was approached by areas, as the VIB was comprised of the main high bay and several annexes. Since PCB-laden paint was not used after 1980, the evaluation was confined to the original VIB building, as the annexes were constructed in the late 1980's. The first floor of the entire facility and the northern portion of the VIB between the annexes did not



2005 Aerial Source: Brevard County Property Appraiser

[illegible]

Site Location

VIB, Cape Canaveral Air Force Station, FL

Applications of BTS for PCB Removal from DoD Facilities

Geosyntec consultants

Figure **4-1**

Guelph | July 2007

site location.ai

have PCB concentrations greater than 50 mg/kg. Throughout the VIB high bay, there was no apparent pattern to the concentration distribution based on structure type, sample location, or paint color scheme except that each of the eight samples collected on the first level were all below 50 mg/kg. Approximately 9,622 tons of metal and steel from the VIB have paint with PCB concentrations greater than 50 mg/kg (CCAFS provided estimate).

The VIB was dismantled in 2007. The dismantling of the VIB was expedited and started in late May of 2007 due to concerns with building structural integrity in the upcoming hurricane season. Advance notice was provided to the research team to allow for the arrangement of sufficiently large sections of the painted structures to be removed and transported to Hangar S Storage Building #66232 at CCAFS /KSC for testing (Figure 4-1). Although the field demonstration was not conducted *in situ* in the actual VIB, care was taken when removing the structural components of the VIB that were tested to insure that no damage was done to the paint on the sections to be tested. The "*in situ*" testing was conducted on large sections of painted I-beams or similar structural sections that were removed from the VIB and transported to Hangar S Storage Building #66232 at CCAFS (approximately 5 miles from the VIB). Care was taken to maintain environmental conditions of the painted structures; sections of the structure were stored and worked on in a similar covered building, with similar temperatures and humidity.

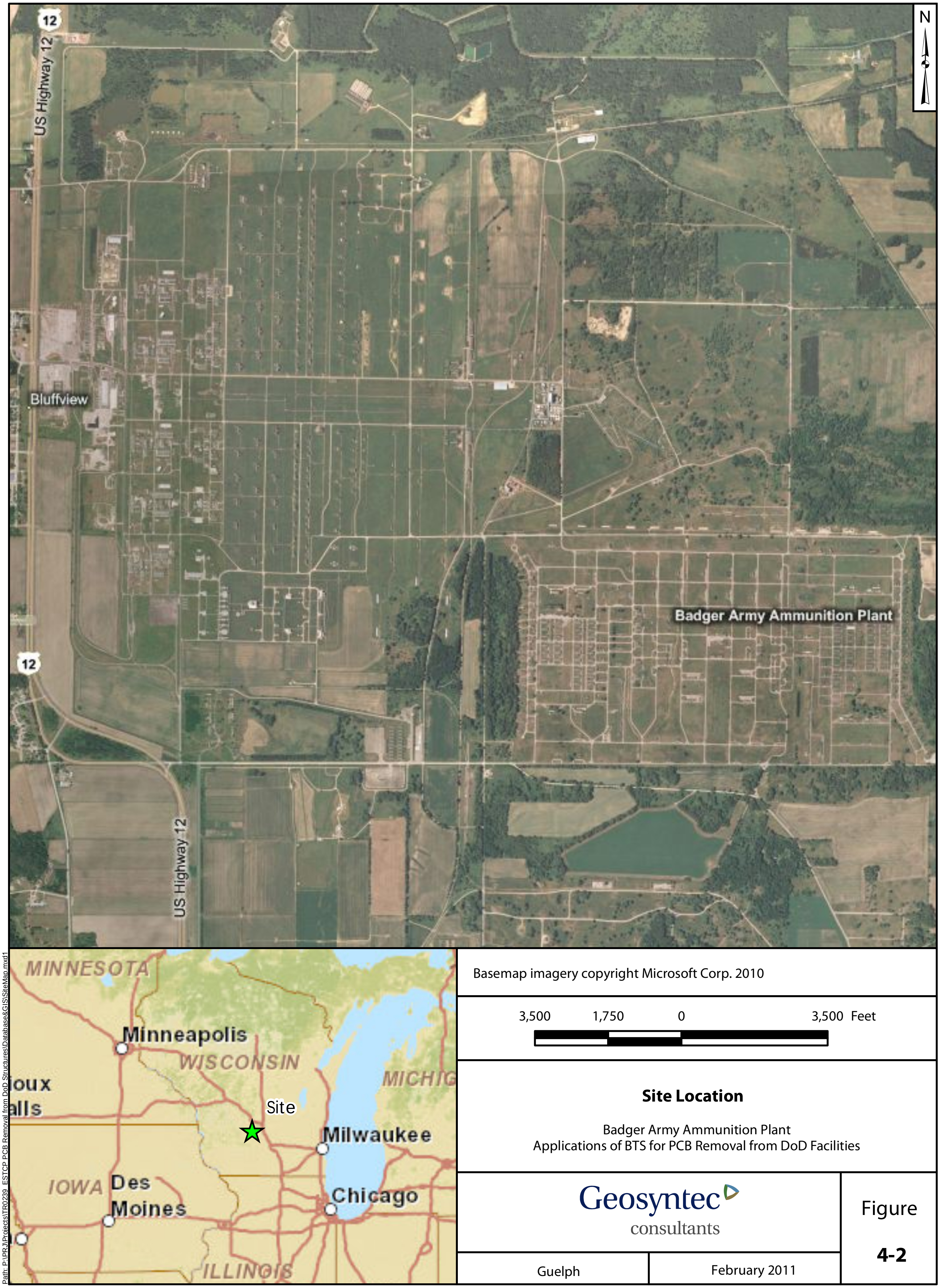
4.2 Badger Army Ammunition Plant

The Badger Army Ammunition Plant (Badger) is located in Sauk County, Wisconsin. Additional information on this site is presented below.

4.2.1 Facility/Site Location and Operations

Badger was established in 1942 and operated intermittently over a 55-year period to produce single and double-base propellants for cannon, rocket, and small arms ammunition (Figure 4-2). Plant operations were terminated in March 1975 and all production facilities and many support functions were placed on standby. In 1997 the Army determined that Badger was no longer necessary for national defense. Future reuse, ownership, and management of the Badger facility depends on the level and extent of explosive decontamination that can be achieved by the U.S. Army. The contamination that needs to be addressed is the removal, by open burning or "flashing", of explosive potential from the buildings that were exposed to open, uncontained explosives. Those explosives include nitrocellulose and nitroglycerine. The explosive potential is due to the residual explosives that fell on floors, collected in concrete pores, settled or condensed onto pipes, beams and equipment during production of ammunition (Plexus Scientific, 2002).

Further investigation into the buildings and equipment that needed treatment indicated the presence of PCBs in the paint on the structures and equipment. The concern with the PCBs is



Basemap imagery copyright Microsoft Corp. 2010

Site Location

Badger Army Ammunition Plant
Applications of BTS for PCB Removal from DoD Facilities

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consultants

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February 2011

Figure

4-2

Path: P:\PRJ\Projects\TR0239_ESTCP PCB Removal from DoD Structures\Database&GIS\SiteMap.mxd1

that the open burning will not produce a sufficiently hot enough flame to destroy the PCBs thus release PCBs into the atmosphere and possible leave residual PCBs in the burn residue.

4.2.2 Facility/Site Conditions

The Badger Army Ammunition Plant is located on the eastside of US12, south of Baraboo, and north of Sauk City in Wisconsin. The building components that were treated are from both inside and outside the structures at the site and as such some had not been exposed to significant weathering due to sun or rain exposure and other components were highly weathered.

Prior to the Badger facility being selected as one of the BTS test sites, the Army conducted an extensive testing program for the purpose of determining the level and location of heavy metal and PCB contamination. This surveillance program documented that high levels of PCBs were present at several locations at the Badger site. For example, the rocket propellant press house buildings had PCB levels as high as 11,000 mg/kg in the painted surfaces of the concrete structure. Some of the painted pipes and tanks of the nitroglycerine Nitrating and Separator houses had as much as 59,000 mg/kg of PCBs in the paint. The buildings and materials that were treated at Badger are no longer in use and are scheduled for demolition, recycling or disposal.

4.3 Japanese Machine Oil Site, Yokosuka, Japan

A Navy operation located in Yokosuka, Japan has a series of machines for operations in milling and cutting industrial materials. Over the years, oils containing polychlorinated biphenyls (PCBs) have been used for lubrication in these machines and during this time, PCBs have contaminated the interior of the surfaces of the equipment, including seals and gaskets. Attempts have been made to 'clean' the PCBs out of the equipment by replacing the contaminated oil with new (wash oil) oil but a PCB concentration above the regulatory limit (Japanese limit of 0.5 mg/kg) persists even in the new oil. Materials or equipment containing oils or fluids which exceed the maximum PCB concentration of 0.5 mg/kg will not be allowed into the Japanese economy and must be excessed or disposed via the United States economy. Facilities managers are exploring options to treat PCB-containing oils and fluids so excess equipment can be released locally rather than requiring costly shipment to United States jurisdiction.

The Yokosuka site is representative of at least four other Navy facilities or fleet activities in Japan housing equipment subject to Japan Environmental Governing Standard (JEGS) 14, which regulates PCB concentrations in fluids or oils in materials or equipment entering the Japanese economy. Materials or equipment containing oils or fluids which exceed the maximum PCB concentration of 0.5 mg/kg will not be allowed into the Japanese economy and must be excessed or disposed via the United States economy. Considering that some of the equipment slated for disposal weighs more than 100,000 pounds, managers at the facility are exploring options to keep the equipment in Japan rather than pay for shipping back to United States territories.

UCF obtained a limited amount of lubricating oil from several milling and cutting machines owned by the U.S. Navy. The funding from this project was used only to refine the methodology for treating the PCBs in the oil through laboratory testing. There was no field testing component to this portion of the project.

The development of the methodology for treatment of PCB-impacted oil was not part of the original project and therefore was not part of the Demonstration Plans. Rather than trying to fit the description of the workplan and results in the structure of the body of this report, the Final Report for Development a Field Deployable Methodology for Safely and Effectively Degrading PCB Contaminated Oils and Machine Shop Equipment Located at the U.S. Navy Shipyard in Yokosuka, Japan is included as Appendix A.

5 TEST DESIGN

This section provides the detailed description of the system design and testing conducted during the demonstration.

5.1 Conceptual Experimental Design

For this demonstration a set of baseline samples were collected from the demonstration sites to evaluate which materials contained PCBs and at what concentrations. Samples were collected of the PCB-impacted materials (painted metal, concrete, and wood) and taken back to the laboratory to test the BTS formulation and sealant options. Once laboratory testing was complete, the field demonstration was conducted. Pre-treatment samples were collected and then the BTS was applied to the painted surfaces and sealed. Both active BTS paste (one step process; paste containing metal; see Figure 2-1) and non-active BTS paste (two step process; no active metal in paste; see Figure 2-2) were used and two types of sealant were tested (vinyl polymer truck bed liner and a silicon roof sealant). Over a three week period sections of BTS were then removed and samples of the paint, BTS, sealant and concrete were collected and tested for PCBs.

5.2 Baseline Characterization and Treatability Study Results

5.2.1 Vertical Integration Building

The VIB deactivation included the demolition, recycling, and/or disposal of the entire VIB Facility. As part of the demolition efforts, steel components with PCB paint concentrations in excess of 50 mg/kg were handled as bulk product waste in accordance with EPA TSCA requirements.

The VIB is located approximately 10 miles from the NASA laboratory where the treatability testing was being conducted, so rather than collecting samples for work in the laboratory; the BTS was applied to small sections of the infrastructure at the VIB itself. Samples of paint were collected from the structural components (I-beams) and taken to the NASA laboratory for PCB testing. Samples of the structures to be treated, sections of painted materials, were used to determine the range of PCB concentrations. The BTS was then applied *in situ* on small sections of the painted metal beams to determine the optimal BTS formulation, and the residual PCB concentration after BTS was applied. Details of these experiments are presented in the Final Demonstration Plan (Demonstration Plan 1, V3, February 2009). The conclusions made based on the results of the laboratory treatability study are presented below.

- There is a large variability in the PCB concentrations in paint that is visually the same and collected from similar sections or structural components of the building.

- There were issues working in the building while it was under demolition (lack of power, access restrictions) and the BTS paste in the *in situ* tests was not completely sealed (the VP liner was applied in too thin a layer).

- Despite less than optimal seal, PCBs were extracted from the paints before the paste dried out.

- A series of sealant tests were also conducted in the KSC paint booth testing room, located behind the KSC laboratory. The tests were conducted using the silicone-based roof sealant (Sil) and this material was able to provide an adequate seal and maintain the moisture in the BTS paste for the duration of the test (over a week).

- Tests were conducted on the application of BTS paste using a textured spray apparatus similar to those used for drywall installation. Using this method an even ¼ to ½ inch layer of BTS paste could be applied on a vertical metal surface.

5.2.2 Badger Army Ammunition Plant

In November of 2006, NASA and the UCF team members traveled to Badger to collect samples from previously identified locations of PCB contamination. The purpose of the visit was to take samples of material that could be tested in the laboratory with the BTS treatment system to establish the optimum formulation and conditions for PCB degradation. During the November 2006 visit to Badger, samples were collected from six major PCB contaminated sites. These sites consisted of two press house buildings, 6810-11 and 6810-36; a wooden rest house, building 6815-08; a nitrating house building, 6657-02; a roll house building, 687-20 and various parts collected from presses located in the staging area.

Based upon analysis and treatment of the collected samples as described in the Demonstration Plan (Demonstration Plan 2, V2, February 2009), it was decided that field testing of the BTS technology would be focused on only four locations, press houses 6810-11 and 6810-36, the nitrating tanks in 6657-02, and the press staging area. Details of these experiments are presented in the Final Demonstration Plan (Demonstration Plan 2, V2, February 2009). The following conclusions were made based on the results of the laboratory treatability study.

- PCB concentrations in paint were higher than had been tested previously with the BTS.

- A sprayable sealant was selected because of ease of application and of the many tested; the VP truck bed liner was selected as the best sealant. The Sil was tested at the VIB and therefore not on the Badger samples.

- It was determined that applying BTS on porous surfaces such as wood and concrete that is not completely sealed with paint resulted in the loss of ethanol from the paste into the treated surface. This resulted in a modification of the paste to include an absorbent material, sodium polyacrylate and cellulose pulp, in the paste to hold extra ethanol but maintain the paste consistency.

- All of the previous tests with the BTS paste were conducted at moderate temperatures between 23 to 27 °C. Because the average temperature during the fall, winter, and spring at Badger are much lower than this, it was decided to conduct a series of tests on the Badger samples at lower temperatures. It was determined that the BTS could perform PCB extractions at lower temperatures but that the rate of extraction might be slower.

5.3 Design and Layout of Technology Components

The BTS used in the field was the same as that used in the laboratory treatability tests. The BTS was composed of 95% ethanol and 5% limonene by volume. The mass percentages for the formulations using an active paste are presented in Table 5-1. A maximum of 20 gal of BTS was applied on the structural components from the VIB and 20 gal at Badger. These estimate were based on the results of the treatability testing done (thickness of BTS applied), including a safety factor. The amount of BTS applied is a function of the covering rate, and is not a function of the reaction kinetics, with the exception of needing to apply a second coat if degradation is not complete after approximately 3 weeks. The BTS has to be applied to cover the paint, and prevent the ethanol from evaporating before the end of PCB extraction. The final amounts of BTS to be used was determined based on the size and types of structural components from the VIB that were transferred to the Hangar S Storage Building #66232 for testing and on the areas tested at Badger.

Table 5-1. BTS ingredients (including %) for active paste

Component	Weight %
Sodium Polyacrylate	3.4
Cellulose Pulp	3.4
Glycerin	14.8
Calcium Stearate	6.8
Polyethylene Glycol 8000	3.4
90% Ethanol - 10% Limonene	58.2
Mg/Pd Metal Powder	10

In preparation for field-scale deployment, the catalyst was manufactured at UCF for inclusion in the BTS active metal paste. The Mg/Pd reductant/catalyst was manufactured using mechanical alloying techniques. There is no commercial vendor currently identified to produce the reductant/catalyst that has been developed; however, there are a number of potential milling vendors that have this capability for future large-scale production. Once the reductant/catalyst had been manufactured the BTS reagent was prepared at KSC for the VIB demonstration. The BTS paste was prepared at UCF and shipped to Badger for application and evaluation by the research team. In order for PCB degradation to take place, a proton donor solvent such as ethanol must be present.

Structural components tested were oriented in both vertical and horizontal positions for application testing. BTS was applied using a texture sprayer and manually using a trowel to a minimum thickness of ¼ inch. Both the bedliner and roof coat sealants were available for sealing the BTS paste. For inactive paste, the BTS was removed at the end of three weeks, placed in a pail and shipped to UCF. Once in the lab at UCF the inactive paste was activated in pails by adding Mg/Pd in an ethanol solution. If moisture levels declined significantly, additional solvent was added to the BTS.

The number and location of BTS application area for each test structural sample varied based on the size and shape of the structural samples available for testing, and other site constraints.

5.3.1 Vertical Integration Building

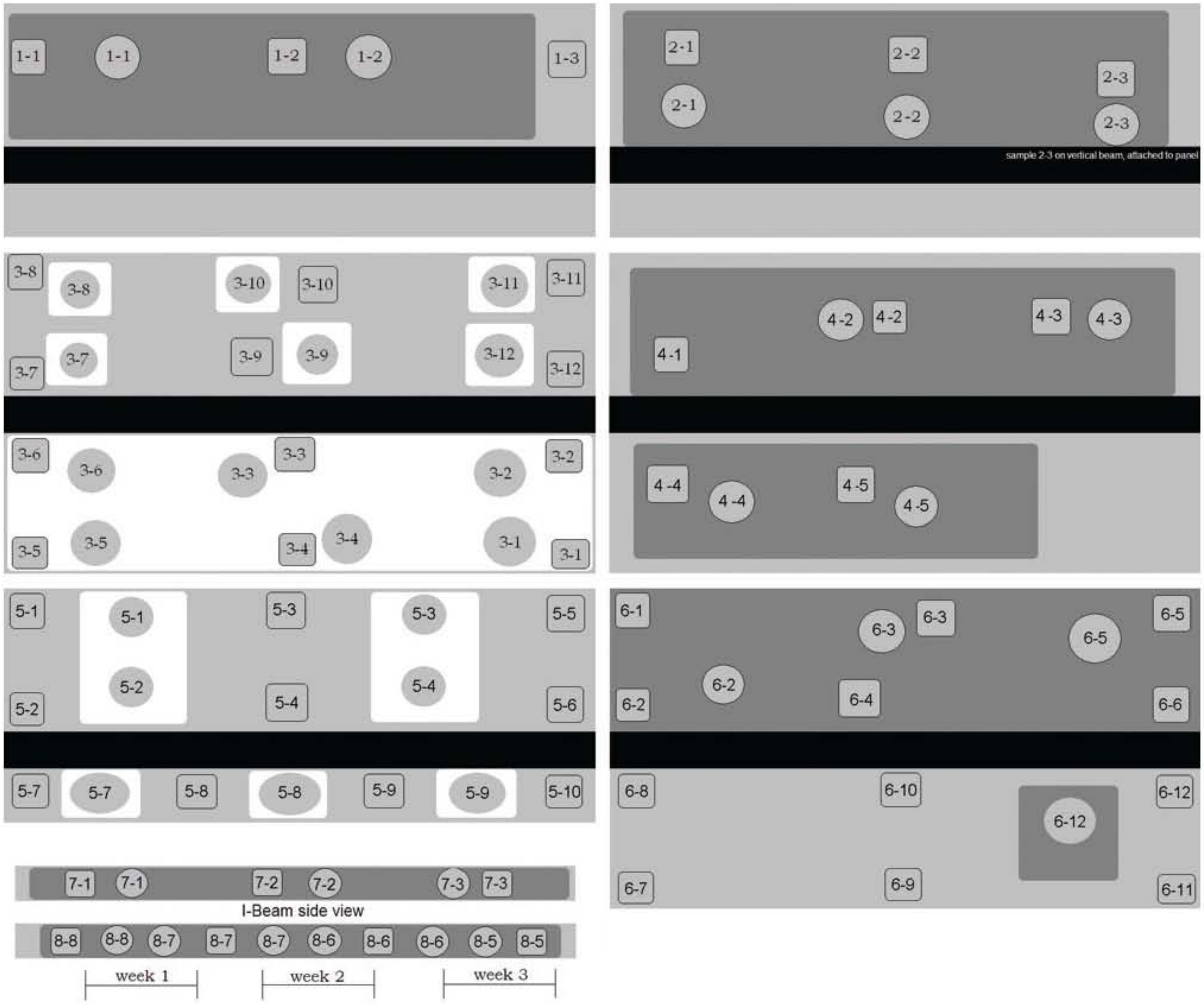
Based on the pre-demonstration laboratory testing that was conducted on paint samples from structural steel components within the VIB and on the painted I-beams themselves within the VIB it was determined that a number of painted surfaces could be used to validate the efficacy of BTS to remove and degrade PCBs. As described in Section 4.1.1, the dismantling of the VIB started before the Demonstration due to concerns with structural integrity of the building in the upcoming hurricane seasons. Based on the results of the pre-demonstration laboratory testing done on the materials in the VIB, a selection of large structural sections of the building that were suspected to contain PCBs were removed and transported to Hangar S Storage Building #66232 for use in this Dem/Val (Figure 4.1). These structural components were removed by dismantling and removing sections that were small enough for transport and cutting up other sections that were too large for transport. Care was taken to minimize any damage to the painted surfaces, to minimize any environmental exposure to the structural sections that would differ from the environment they would have been exposed to within the VIB (e.g., no direct sunlight, no contact with rain), and to minimize any changes to the painted surfaces due to exposure to heat from cutting tools.

All testing was done on portions of the structural components at least 3 inches from any cut edge or disturbed surfaces to minimize the risk of testing BTS on non-representative materials. Approximately twenty gallons of BTS was available for application on the VIB structural components. A variety of structures and/or surface features (vertical walls, horizontal) were treated. A grid was established on each of the different structural components for an overall treatment area of up to 100 ft^2 to allow for the collection of ten pre-deployment samples and up to thirty post-BTS deployment samples (Figure 5.1). Each square of the grid was large enough to collect both a pre-BTS -deployment sample and up to three post-BTS-deployment samples. #

BTS formulations were applied to the type of structural material where known values of PCBs in paint above 50 mg/kg had already been verified using a grid sampling technique described above. Once all pre-deployment samples were acquired, the structural components were coated with BTS using either the spray texture applicator or via manual application. The BTS was then sealed with one of two spray sealants to minimize volatilization from the paste.

Of the 100 ft^2 area to be treated, 50 percent of the painted surface area was to be subjected to inactive BTS paste and 50 percent treated with active BTS. The BTS application technique ensured that a minimum BTS thickness of ¼ inch is achieved.

The treated structural components were monitored daily for the first week and then weekly for two to three additional weeks for moisture levels and sealant integrity. After one week, two weeks, and three weeks, and for the VIB four weeks, several samples of the paint, the paste and the sealant from within the grid pattern were analyzed for PCB concentration in order to track



Legend:

- Numbered rectangles are pre-treatment samples.
- Numbered circles are post-treatment samples.
- Black strips across the panels are the I-beams that separate a panel into 2 sections.
- Light gray areas are the steel panels themselves.
- Dark gray areas on the panels are areras treated with active metal.
- White areas on the panels are areas treated with inactive paste.

Sampling Diagrams for Test Panels 1-6
and Test I-Beam 7-8
BTS Paste Application

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Figure
5.1

Sampling Diagrams.ai

extraction efficiency from the paint and degradation capability of the paste. Sample collection and extraction followed the analytical procedures outlined in Section 5.5.

After four weeks at the VIB, all sealant and BTS paste was removed. The inactive paste and its sealant were placed in a drum or pail, shipped back to UCF and the reductant/catalyst and additional ethanol were added to initiate PCB degradation in the recovered inactive paste. The active paste and sealant were subjected to analysis for PCBs in the NASA laboratory.

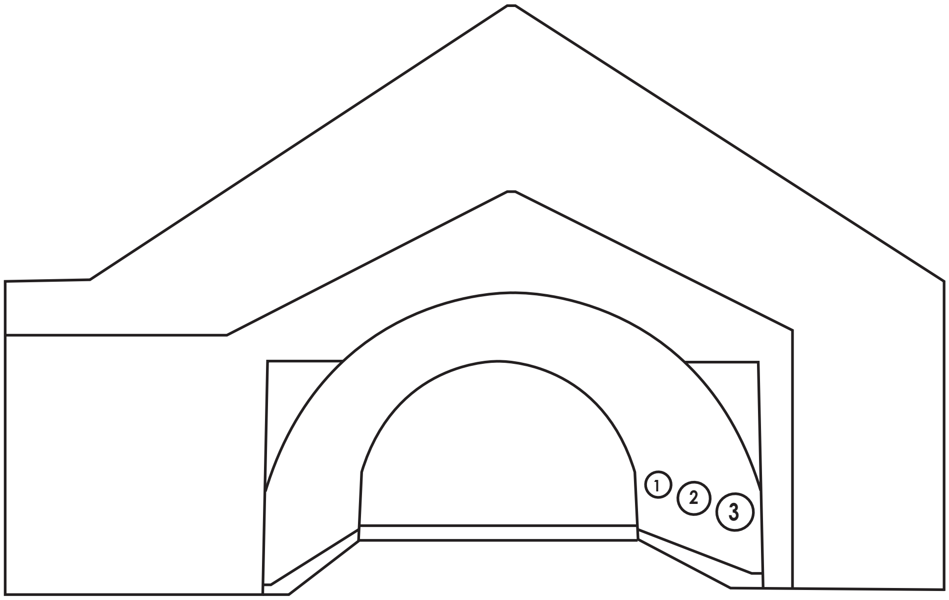
5.3.2 Badger Army Ammunition Plant

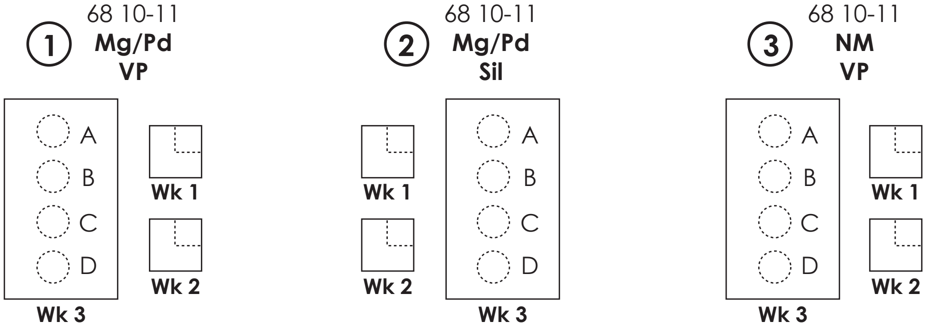
BTS formulations were applied to structural material at the four locations identified in the baseline testing (press houses 6810-11 and 6810-36, the nitrating tanks in 6657-02, and the press staging area) where known values of PCBs above 50 mg/kg have already been verified using the grid sampling technique described below. Once all pre-deployment samples were acquired, the structural components were coated with BTS using either the spray texture applicator or via manual application.

A grid was established on each of the different structural components for an overall treatment area of up to 100 ft^2 to allow for the collection of pre-deployment samples and post-BTS deployment samples (Figure 5.2, Figure 5-3, Figure 5-4 and Figure 5-5). Each square of the grid was large enough to collect both a pre-BTS -deployment sample and up to three post-BTS-deployment samples. Of the area treated, 1/3 of the painted surface area was subjected to inactive BTS paste sealed with the VP, 1/3 was subjected to active BTS sealed with the VP and 1/3 treated with active BTS sealed with the Sil. The BTS application technique ensured that a minimum BTS thickness of ¼ inch was achieved.

The treated structural components were monitored daily for the first week and then weekly for three weeks for moisture levels and sealant integrity. After two weeks, several samples from within the grid pattern were analyzed for PCB concentration in order to track extraction efficiency from the paint. The BTS paste was also analysed for PCB concentration. Sample collection and extraction followed the analytical procedures outlined in Section 5.5.

After three weeks, all sealant and BTS paste were removed and samples of the paint were taken. The inactive paste and its sealant were placed in a drum or pail, shipped back to UCF and the reductant/catalyst and additional ethanol were added to initiate PCB degradation in the recovered inactive paste. The active paste and sealant were subjected to analysis for PCBs in the UCF laboratory.





Legend

⬚	Indicated pre-demonstration sample location
Wk 1	Week 1 sample area
Wk 2	Week 2 sample area
Wk 3	Week 3 sample area
VP	Vinyl Polymer truck bed liner sealant
Sil	Silicon roof sealant
Mg/Pd	Magnesium Palladium, active paste
NM	No metal, inactive paste

Bunker II.ai

Bunker/Press House 6810-11
BTS Paste Application

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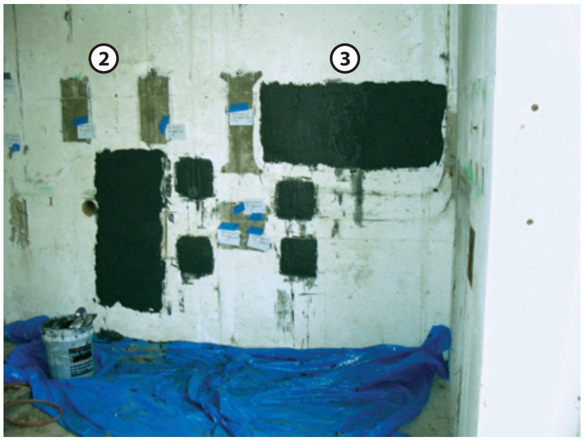
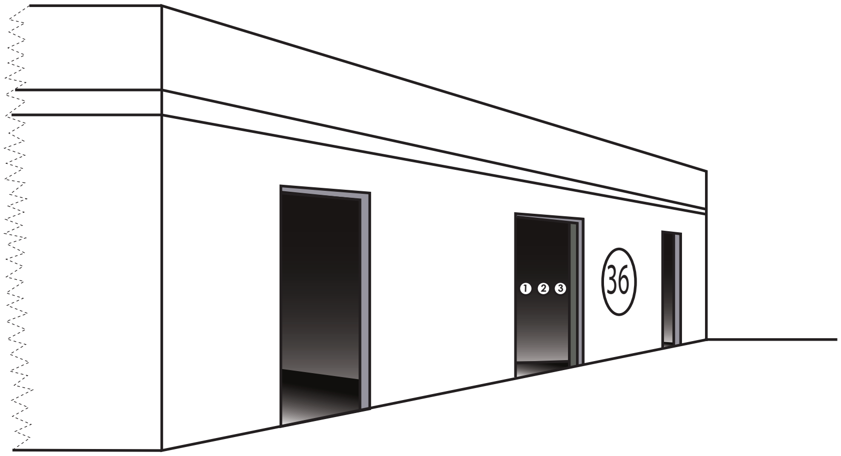
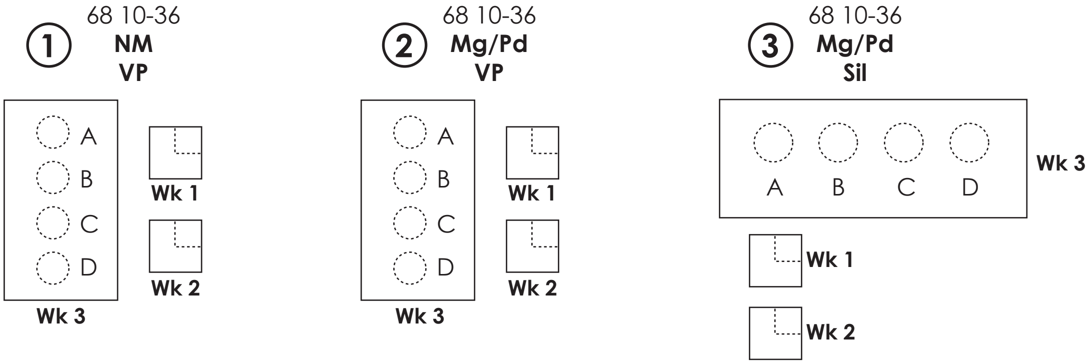
Figure
5.2







Legend

⬚	Indicated pre-demonstration sample location
Wk 1	Week 1 sample area
Wk 2	Week 2 sample area
Wk 3	Week 3 sample area
VP	Vinyl Polymer truck bed liner sealant
Sil	Silicon roof sealant
Mg/Pd	Magnesium Palladium, active paste
NM	No metal, inactive paste

Press Building 6810-36
BTS Paste Application

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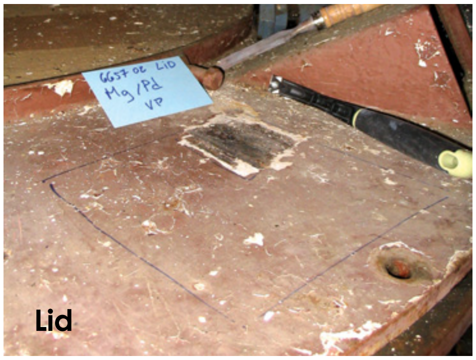
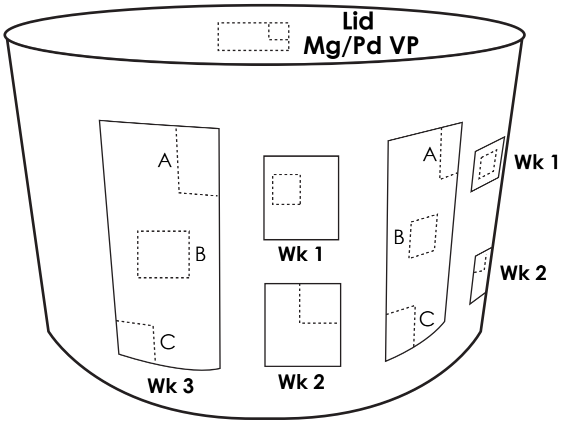
Figure
5.3

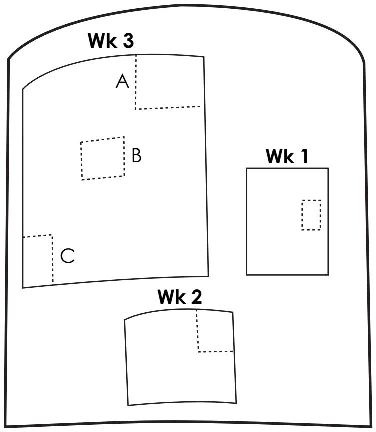
Press Building 36.ai



Large Tank
Mg/PD and NM-VP



Small Tank
Mg/Pd Sil



Legend

⬚	Indicated pre-demonstration sample location
Wk 1	Week 1 sample area
Wk 2	Week 2 sample area
Wk 3	Week 3 sample area
VP	Vinyl Polymer truck bed liner sealant
Sil	Silicon roof sealant
Mg/Pd	Magnesium Palladium, active paste
NM	No metal, inactive paste

Nitrating House 6657-02
BTS Paste Application

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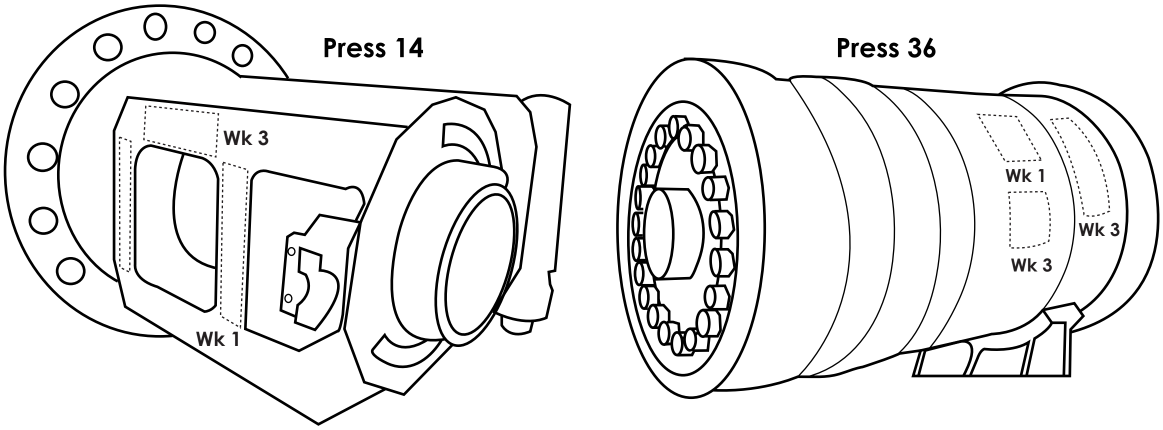
Figure
5.4

Nitrating House.ai



BTS Paste applied to various spots on press #4, 12, 14, 15, 36

Legend

⬚	Indicated pre-demonstration sample location
Wk 1	Week 1 sample area
Wk 2	Week 2 sample area
Wk 3	Week 3 sample area
VP	Vinyl Polymer truck bed liner sealant
Sil	Silicon roof sealant
Mg/Pd	Magnesium Palladium, active paste
NM	No metal, inactive paste

Staging Area.ai

BTS Paste Applied to Presses
BTS Paste Application

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Figure
5.5

5.4 PCBs in Paint Sample Confirmation

A set of paint samples from Badger were sent to a commercial laboratory, Columbia Analytical Services (CAS), of Rochester NY, for confirmatory sample analysis (Table 5-2). Although the concentrations of the paint samples analyzed by UCF and those of CAS were not exactly the same, the concentrations were within expected variability given the differences in concentrations seen in the paint samples taken from each test sections at Badger.

5.5 Operational Testing

There were two main operational phases to this technology demonstration: the *in situ* field demonstration and the follow-up laboratory testing for paste optimization. The field application for both sites involved baseline sampling, BTS paste application and performance monitoring at one, two and three weeks after BTS application (one structural component from the VIB site was monitored at four weeks post-BTS application). All components that were to be tested (paint, paste, sealant and concrete) were taken back to the laboratory at UCF for PCB analysis. All of the paste and sealant that was applied at the field sites was removed; no equipment or materials were left at the field sites.

5.6 Sampling Protocol

5.6.1 Pre-Demonstration Samples

Pre-demonstration samples of the paint from each of the components that were tested (painted metal or concrete) were obtained via the cold-scraping method in order to accurately determine the starting concentration levels of PCBs in the paint. In accordance with ASTM E1729-05, paint samples were collected for analysis using the cold-scraping method with a metal chisel or equivalent scraping tool. Because of the potential for collection of metal substrate during the scraping process when sampling a painted metal surface, a magnet was used to separate steel from the paint samples prior to weighing. This was done to ensure the sample masses were not artificially high, leading to false low PCB concentrations. Sample bags were attached to the surface with tape below (vertical surfaces) or adjacent to (horizontal surfaces) the section of paint to be sampled. Care was taken to collect all of the paint scrapings in the sample bag which was then sealed and labeled with the date, contents and sampling location. Pre-demonstration sample locations are shown on Figures 5-1 5-2, 5-3, 5-4 and 5-5 in Section 5.3. Prior to treatment, pre-demonstration samples of paint were collected from each section that was to be tested. At the VIB, a minimum of three and maximum of 6 pre-demonstration samples (shown as rectangles on Figure 5-1) were collected from each panel or I-beam that was tested. At Badger, a 4 in^2 sample of paint was collected from each of the 1 ft^2 sections and four sets of samples, each 4 in^2 in area, were collected from the 3 ft^2 treatment areas (see Section 5.3 for

TABLE 5-2
SAMPLES FROM THE BADGER ARMY MUNITIONS DEPOT FIELD STUDY ANALYZED BY BOTH UCF AND COMMERCIAL LABORATORY CAS

Vial #	Sample Description	UCF Average Concentration (mg/kg)	CAS Concentration in (mg/kg)	RPD%	Concentration Based On Aroclor 1254 or 1260
1	Staging Area Press #9331 Pre Mg/Pd VP top barrel #14	93	77	19%	1,260
2	Staging Area Press #9331 Pre Mg/Pd VP paint #14	160	89	57%	1,260
3	6657-02 Pre NM VP(1 wk area)	42,660	43,000	1%	1,254
4	6657-02 Pre Mg/Pd sil (1 wk area)	15,319	13,000	16%	1,254
5	Staging Area Mg/Pd VP#15 post 3wk	BDL	2		1,260
6	Staging Area Mg/Pd VP#36 post 3wk	BDL	10		1,260
7	6657-02 Mg/Pd sil post 3wk	8,687	5,100	52%	1,254
8	6657-02 Mg/Pd VP post 3wk B	14,847	17,000	14%	1,254

Notes:
BDL - below detection limit
CAS - Columbia Analytical Services
mg/kg - milligrams per killogram
RPD - relative percent difference

details). The samples from the 3 ft^2 treatment area were labeled as A, B, C, D with A being the top most sample.

Figure 5-6 shows Panel 4 after pre-demonstration samples had been obtained, while Figure 5-7 similarly shows I-Beam 8.

Figure 5-6. Test Panel 4 prior to application of BTS

Figure 5-7. Test I-Beam 8 prior to application of BTS

Figure 5-8 shows where the pre-demonstration samples were taken from Building 6810-11 at Badger for the section that was to be treated with the BTS containing active Mg/Pd particles and sealed with a VP. These samples have the prefix identification of 6810-11 Mg/Pd-VP (see Section 5.4.2 for details). This figure also shows (in the lower right corner) where sections of concrete were cut out from the wall for preliminary treatment tests in November of 2006. The other two treatment sections in 6810-11 were sampled in a similar manner.

Figure 5-8. Location of Pre-Demonstration Samples in 6810-11.

In addition, at Badger, in order to evaluate the potential for the BTS to transfer PCBs from the paint into concrete, samples of concrete were collected prior to BTS application. The paint was scraped off of a roughly 1 in^2 section of concrete where the concrete was to be tested. A sample bag was the taped to the wall below the section to be tested to collect the concrete dust. An electric drill with a concrete drill bit was then used to drill out a section of the concrete to a depth of 3/8-inch. All of the concrete dust from the drill bit was collected in the sample bag which was then sealed and labeled with the date, contents and sampling location. Samples of concrete to a depth of 3/8 inch were taken from each treatment location before and after BTS treatment.

5.6.2 BTS Paste Application

5.6.2.1 <u>Vertical Integration Building</u>

A total of 8 sections were chosen for the testing of BTS, including 6 panel sections and 2 I-Beam sections. Diagrams of each test site and various sampling locations are shown in Figure 5-1, Section 5.3.

In preparation for the demonstration of BTS on the VIB samples, approximately 10 gallons of both active and inactive paste were prepared. The formula used for the BTS (active) is given in Table 5-1. The inactive formula is identical, except for use of slightly less glycerin and the exclusion of Mg/Pd powder.

A modified pressurized sprayer was used in the initial application of BTS (Figure 5-9). Additional solvent was added to the paste (prior to application) to facilitate spraying. A manual application technique (using a trowel) was also used. There was a difference in the thickness of the BTS when applied using the two application methods and it was later shown that the thickness of the BTS affects treatment (see Section 5.6.3 Paste Optimization Studies). Because the thickness of the sprayed BTS was so much smaller than that applied via a trowel (approximately 1/8 to 1/4 of an inch versus 3/8 to 1/2 of an inch, respectively), a majority of the sections were treated with BTS applied manually using a trowel, as shown in Figure 5-10.

Figure 5-9. Application of BTS to VIB samples using pressurized sprayer

Figure 5-10. Manual application of BTS to VIB samples.

Sealants were used to coat the surface of the BTS paste in an attempt to prevent loss of solvent from the BTS following application. Two sealants were tested, 1) a silicone roof sealant (Sil) (mineral spirits-based); and 2) a vinyl polymer truck bed liner (VP) (toluene-based). These were diluted with solvents (ethanol or mineral oil for the Sil, acetone for the VP) in a 3:1 coating: solvent ratio in order to allow for ease of spraying during application. They were both applied using a high volume/low pressure (HVLP) sprayer. Approximately 5 coatings of each material were applied to the BTS surfaces and at least an inch beyond the edge of the BTS paste, to seal the edge of the paste. Care had to be taken when applying the initial coating of sealant and the sealant allowed to set for a short period of time (roughly 5 minutes) before additional coatings could be used. If a heavy coating (high flow rate) of sealant was applied at the start, it tended to displace the BTS from the surface that was being treated. Once the sealant was in place, the BTS paste was allowed to react for the specified times (one, two three or four weeks). Figure 5-11 shows the application of the truck bed liner sealant material.

Figure 5-11. Application of coating material using HVLP sprayer.

5.6.2.2 Badger Army Ammunition Plant

The BTS paste was applied by hand with a trowel for all the Badger applications to a thickness of 3/8 to 1/2 of an inch and sealed with either the VP applied to a thickness of 1/16 of an inch or the Sil applied to a thickness of 1/8 of an inch.

Press House 6810-11

Figure 5-2 is a photo and schematic of the location within the press house 6810-11 where the BTS treatment was applied. There were three main treatment areas arranged in building 6810-11, each consisting of three sections. The treatments consisted of applying the BTS paste to two 1 ft^2 sections, where one of the sections would be sampled after one week of treatment and the second section would be sampled two weeks post application. A third section (3 ft^2) was to be sampled after three weeks. One of the main treatment areas was treated with BTS paste that contained no active Mg/Pd metal. This area was sealed with the VP. A second area was treated with BTS paste that contained Mg/Pd particles and was sealed with the VP sealant. The third area was treated with BTS paste that contained Mg/Pd particles and was sealed with the Sil.

After all of the pretreatment samples were taken, the BTS paste was applied to the wall. The original plan was to spray the BTS onto the test section using a cartridge type sprayer gun as shown in Figure 5-12. However, the temperature dropped below freezing and the paste became too viscous to spray. Therefore, it became necessary to apply the paste to the test section by manual application with a trowel. This technique was used to apply the BTS paste to all other test locations at Badger. After the BTS paste was applied to the test section, the sealant was then sprayed onto the paste to cover it. The VP sealant was sprayed on by using a cartridge spray gun as can be seen in Figure 5-13. The silicon sealant was applied by using a typical commercial spray gun as shown in Figure 5-14.

Figure 5-12. Spraying BTS with cartridge-type spray gun.

Figure 5-13. Vinyl polymer sealant application using cartridge spray gun.

Figure 5-14. Silicon sealant application with spray gun.

Inspection of the 6810-11 treated areas on the day after treatment confirmed that some cracks had formed in both the VP sealant and the silicon sealant. This type of cracking was never observed in laboratory experiments, thus it was speculated that the cold weather, in the 30 to 40 degree Fahrenheit range, was the cause of the cracking. The cracks in the VP sealant were fixed by spraying the VP from an aerosol can (as it is sold commercially) and the cracks in the silicon polymer were fixed by applying the polymer with a brush. The test site was checked each day for a week until week one samples were taken. Any new cracks in the sealant that formed during the week were repaired as described above.

The state of the treated sections at the 6810-11 test site can be seen in the following three figures which show them three days after treatment. Figure 5-15 shows the NM-VP site, Figure 5-16 shows the Mg/Pd-silicon site and Figure 5-17 shows the Mg/Pd-VP site. As the figures show, all of the sealants are intact with no cracks.

Figure 5-15. Three days post BTS application, NM-VP in 6810-11.

Figure 5-16. Three days post BTS application, Mg/Pd-silicon in 6810-11.

Figure 5-17. Three days post BTS application, Mg/Pd-VP, 6810-11.

Press House 6810-36

The same procedure used in press house 6810-11was followed in press house 6810-36. However, there was a major difference in the integrity of the paint on the surface of the concrete walls in 6810-36 as compared to 6810-11. The paint in 6810-36 was much more powdery and friable than the paint in 6810-11. This presented problems in getting the BTS paste and sealant to adhere to the wall. The BTS and sealant would seal to the paint but the paint was not adhering to the wall.

A set of treatment sections similar to those used at 6810-11 were used at 6810-36 as shown in Figure 5-3, Section 5.3.

Figure 5-18 shows the application of the Mg/Pd-BTS using a trowel. Figure 5-19 shows the NM-VP (non metal paste sealed with VP) test location in 6810-36 three days after treatment and Figure 5-20 shows the Mg/Pd-VP (active metal paste sealed with VP) test location after three days of treatment. The 1 ft^2 section three days after BTS application and sealed with silicon polymer (Mg/Pd-Sil) is shown in Figure 5-21. The 3 ft^2 section, three days after treatment is shown in Figure 5-22. As can be seen, the sealant on the 3 ft^2 Mg/Pd-Sil section began to fail immediately and could not be saved for testing after three weeks of treatment. Thus it was decided to switch the planned sections and test it after one week of treatment. The 6810-36 test site was visited each day for one week and when any cracks appeared in the sealant, attempts were made to repair them as described in the 6810-11 site section above.

Figure 5-18. Application of Mg/Pd BTS prior to sealing with VP or silicon sealant, 6810-36.

Figure 5-19. NM-VP test location after three days of treatment, 6810-36.

Figure 5-20. Mg/Pd-VP test location after three days of treatment, 6810-36.

Figure 5-21. Mg/Pd-Sil, three days after treatment, 6810-36.

Figure 5-22. Mg/Pd-Sil, three-foot section, after three days of treatment, 6810-36.

Building 6657-02: Nitrating House

The location of the treatment zones at the nitrating house, Building 6657-02 are shown on Figure 5-4, Section 5.3. There were two nitrating tanks located in this building, one large tank and one smaller tank. The large tank was treated with both the Mg/Pd paste and the NM paste and both of these test sections were coated with VP sealant. The small tank was treated with Mg/Pd paste and then sealed with the silicon polymer. Figures 5-23, 5-24, and 5-25 show the large tank after it was treated and sealed with the silicon polymer. Figures 5-26 and 5-27 show the large tank two days after treatment. As can be seen from the figures, the sealant and BTS is still intact and has not failed.

Figure 5-23. Large tank treated with Mg/Pd-VP and NM-VP immediately after application – view 1.

Figure 5-24. Large tank treated with Mg/Pd-VP and NM-VP immediately after application - view 2.

Figure 5-25. Large tank lid, treated with Mg/Pd-VP, immediately after application.

Figure 5-26. Large tank treated with Mg/Pd-VP and NM-VP, two days after application.

Figure 5-27. Large tank lid treated with Mg/Pd-VP, two days after application.

Presses in the Staging Area

The last test location that was treated was the staging area where five presses were treated (Figure 5-5, Section 5.3). All of the presses were treated with the Mg/Pd paste and sealed with the VP. Only two treatment zones were done for the presses, one to be sampled after one week and the other to be sampled after three weeks. Figure 5-28 shows BTS paste being applied to press 14 and Figure 5-29 shows the VP sealant being applied to press 14. Figure 5-30 shows the two treated areas on press 4, Figure 5-31 shows the two treated areas on press 15 and Figure 5-32 shows the treatment locations on press 36. All of the treated areas were in good shape two days after treatment as can be seen from Figure 5-33, showing the treatment zones on press 4, two days after treatment.

Figure 5-28. BTS paste being applied to press 14.

Figure 5-29. Vinyl polymer (VP) sealant being applied to press 15.

Figure 5-30. Two treated areas on press 4.

Figure 5-31. Two treated areas on press 15.

Figure 5-32. Treated areas on press 36.

Figure 5-33. Treatment areas on press 4 two days after BTS application.

5.6.3 Performance Sampling

5.6.3.1 Vertical Integration Building

Samples were taken periodically over the period of a month for analysis of the both the treated paint and exposed BTS material and sealant. Analysis of paint samples were performed by Kennedy Space Center, while analysis of the BTS itself was performed at the University of Central Florida. Prior to sampling, the coating layer (VP or Sil) was removed. It proved possible to remove this in a single piece, helping to keep the sample below intact for analysis. Figure 5-34 shows the coating material being removed.

Figure 5-34. Removal of coating material after exposure to BTS

The BTS material itself showed variability in appearance/moisture level upon removal of the coating, most likely related to amount of time exposed and some possible cracks in the sealant which allowed solvent evaporation. However, several samples still appeared moist after 3 weeks of exposure, indicating that the solvent adsorption material added to the BTS formulation was helping to retain a portion of the ethanol: limonene solvent. This is crucial in order for degradation of the PCBs to occur *in situ*. Examples of the paste after exposure are shown in Figure 5-35.

Figure 5-35. Varying appearance of BTS (non-metal paste) after exposure to paint and removal of sealant.

5.6.3.2 Badger Army Ammunition Plant

Press House 6810-11

Post-treatment samples were taken after weeks one, two and three. The samples that were taken included all of the sealant coating, all of the paste and a five square-inch section of paint. In addition, concrete samples were taken from some of the treated sections to a depth of 3/8-inch after three weeks of treatment.

Figure 5-36 shows the one week VP, non-metal test location prior to sampling. Figure 5-37 shows the sample site after the VP has been removed. Figure 5-38 shows the Mg/Pd-VP site prior to removing the sealant and Figure 5-39 shows the sample area after the VP has been removed. Figure 5-40 shows the Mg/Pd-silicon sample area prior to removing the sealant and Figure 5-41 shows the sample location after the sealant has been cut away.

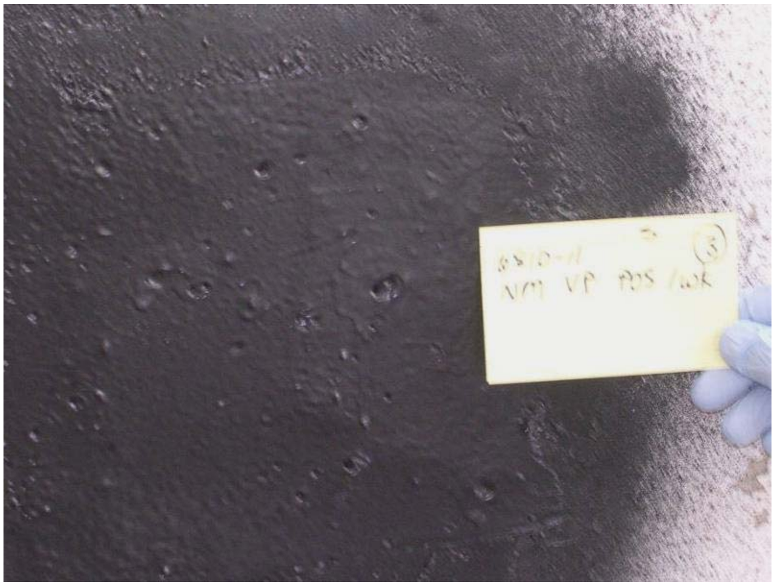


Figure 5-36. One week, post application, before samples were taken, NM-VP 6810-11.

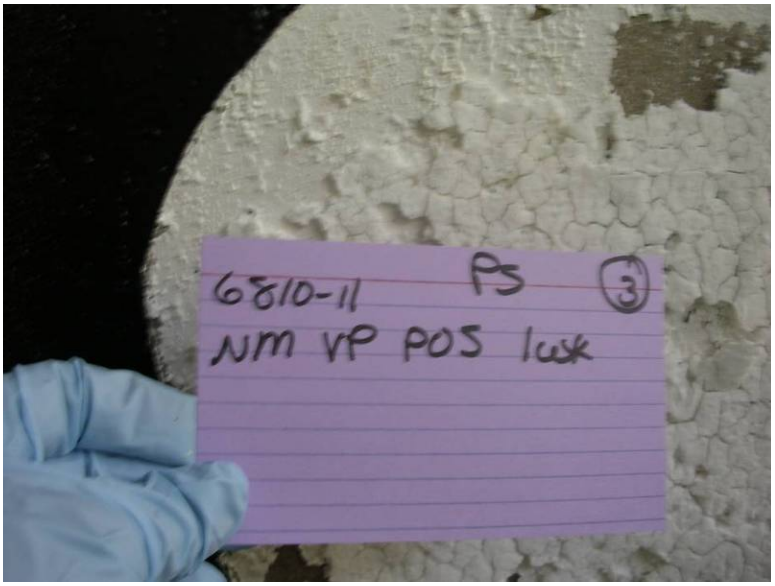


Figure 5-37. One week, post application, after samples were taken, NM-VP 6810-11.

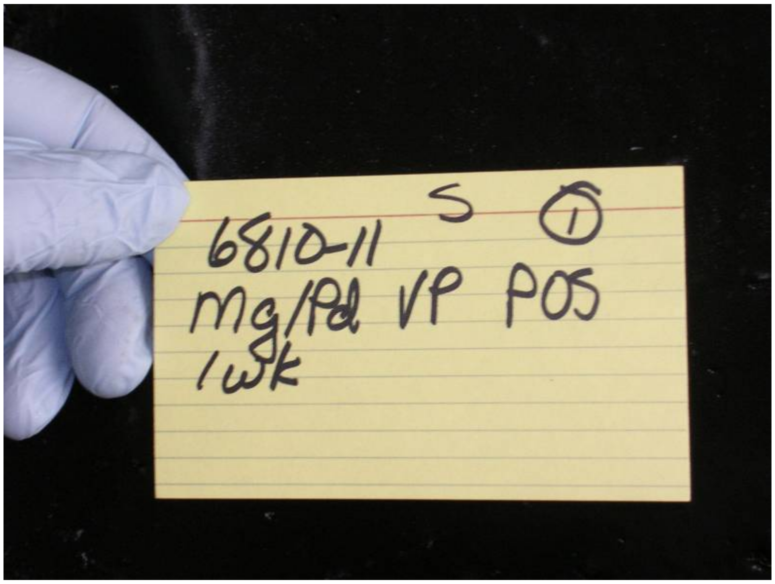


Figure 5-38. One week post application, pre-sampling, Mg/Pd-VP, 6810-11.

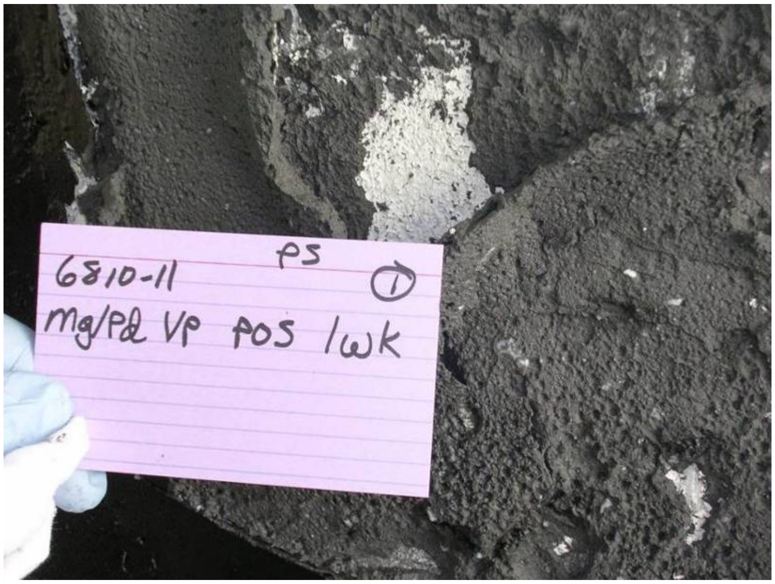


Figure 5-39. One week post application, during sampling, Mg/Pd-VP, 6810-11.

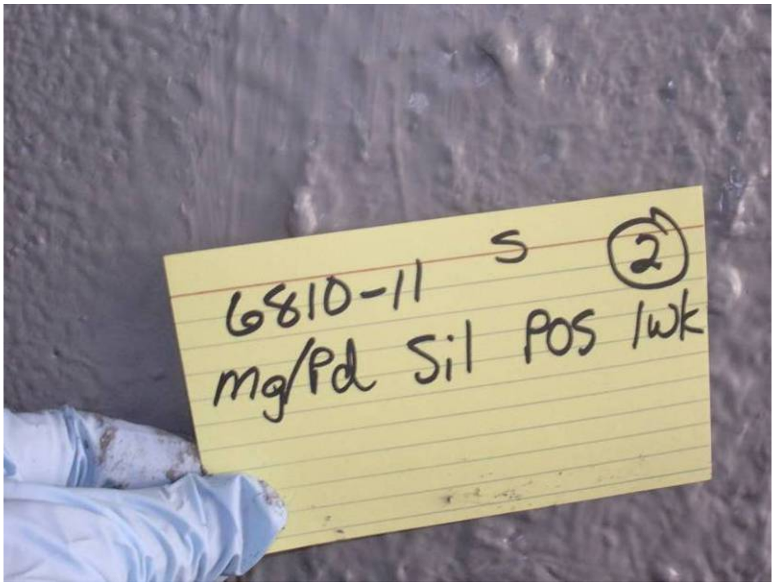


Figure 5-40. One week post application, pre-sampling, Mg/Pd-silicon, 6810-11.

Figure 5-41. One week post application, during sampling, Mg/Pd-silicon, 6810-11.

Figure 5-42 shows the 6810-11 Mg/Pd-VP treatment two weeks after treatment. It can be seen that part of the VP sealant had failed. Thus it was decided to take the two week sample from the 3 ft^2 section and the three-week sample from a 1 ft^2 section (originally identified for sampling at two weeks).

Figure 5-42. Mg/Pd-VP, two weeks after application, 6810-11.

Figure 5-43 shows the Mg/Pd silicon site prior to taking week two samples and that the sealant had not failed at this location. Figure 5-44 shows the Mg/Pd-silicon site during the two week sample was taken.

Figure 5-43. Mg/Pd-silicon prior to two week sampling event, 6810-11.

Figure 5-44. Mg/Pd-silicon after two week sampling event, 6810-11.

Figure 5-45 shows the status of the 6810-11 Mg/Pd-VP treatment site prior to sampling after three weeks of treatment. It can be seen that the condition of the sealant did not change very much between weeks two and three. The three week sample for this treatment location was taken from the 1 ft^2 area that was still intact. Figure 5-46 shows the NM-VP treatment location, prior to taking the three week sample. It can be seen that a portion of the VP sealant broke loose between week two and week three sampling. Figure 5-47 shows the Mg/Pd-silicon treatment area and it can be seen that the 3 ft^2 section is still intact. Figure 5-48 shows the Mg/Pd-silicon site after sampling. Figure 5-49 shows a sample of concrete being taken from the Mg/Pd-silicon site after three weeks of treatment.

Figure 5-45. Mg/Pd-VP prior to three week sampling event, 6810-11.

Figure 5-46. NM-VP prior to three week sampling at 6819-11.

Figure 5-47. Mg/Pd-silicon prior to three week sampling at 6810-11.

Figure 5-48. Mg/Pd-silicon after three week sampling event, 6810-11.

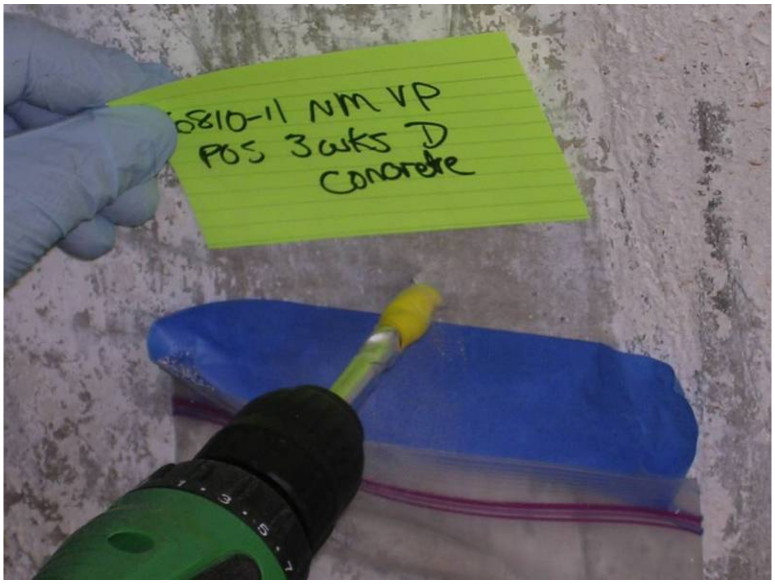


Figure 5-49. Sampling concrete at 6810-11 at NM-VP location.

Press House 6810-36

Samples were collected of the paint, paste and sealant on each of the treatment areas after one week, two weeks and three weeks. In addition, a concrete sample was collected after three weeks exposure to the paste. The following Figures show the status of the treatment sites in building 6810-36 after the two week samples were taken. Figure 5-50 is for the Mg/Pd-silicon, Figure 5-51 is for the Mg/Pd-VP and Figure 5-52 is for the NM-VP sites.

Figure 5-50. Mg/Pd-silicon after two weeks sampling event at 6810-36.

Figure 5-51. Mg/Pd-VP after two week sampling event at 6810-36.

Figure 5-52. NM-VP after two week sampling event at 6810-36.

Figure 5-53 shows the 1 ft^2 test section with Mg/Pd-sil in building 6810-36 that was sampled after three weeks. Figure 5-54 shows the Mg/Pd-VP section that was sampled after three weeks and Figure 5-55 shows the NM-VP section prior to taking the three week sample.

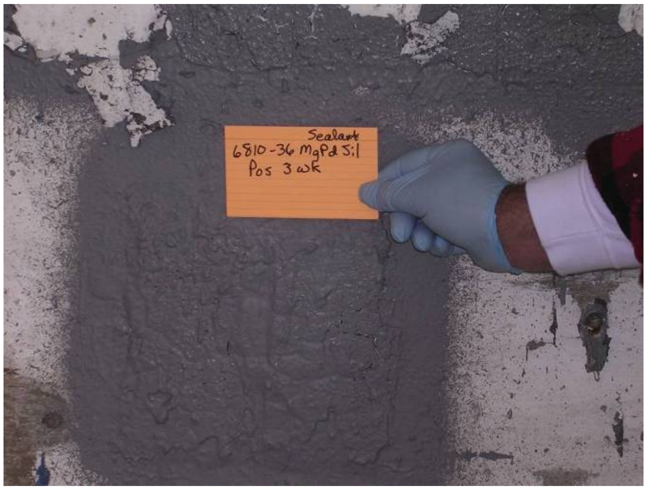


Figure 5-53. Mg/Pd-silicon after three weeks, 6810-36.

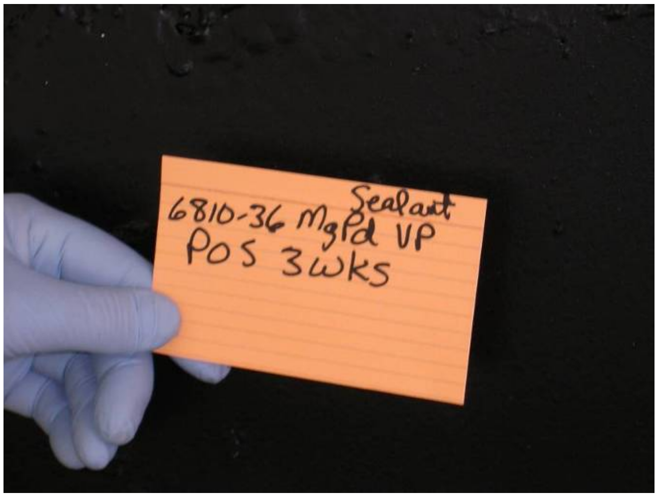


Figure 5-54. Mg/Pd-VP after three weeks, 6810-36.

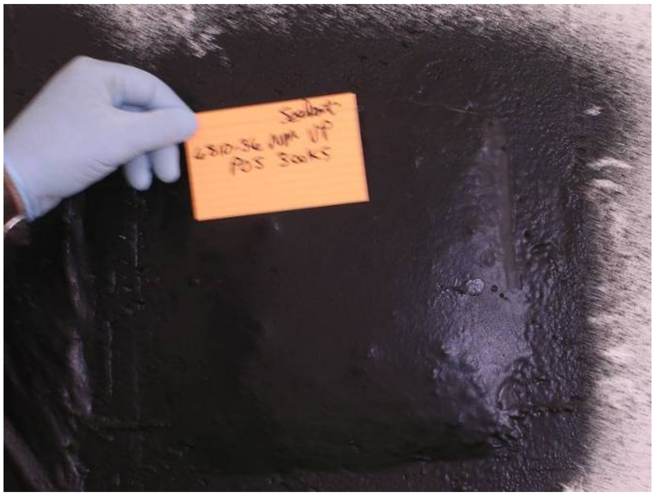


Figure 5-55. NM-VP after three weeks, 6810-36.

Building 6657-02: Nitrating House

Samples were collected of the paint, paste and sealant on each of the tanks that were treated after one week, two weeks and three weeks. Figure 5-56 shows the large tank after one week and as can be seen, the treated sections are still intact. Figure 5-57 shows the one week NM-VP sample being taken and Figure 5-58 shows the one week Mg/Pd-VP sample being taken. Figure 5-59 shows that the 3 ft^2 section on the small tank that was treated with the silicon polymer failed after one week. This section was to be sampled after three weeks of treatment, however, it was decided to take the one week sample from this section and then take the two and three week samples from the 1 ft^2 sections that were still intact.

Figure 5-56. Large tank treated with Mg/Pd-VP and NM-VP one week after application.

Figure 5-57. Sample being taken from large tank treated with NM-VP one week after application.

Figure 5-58. Sample being taken from large tank treated with Mg/Pd-VP one week after application.

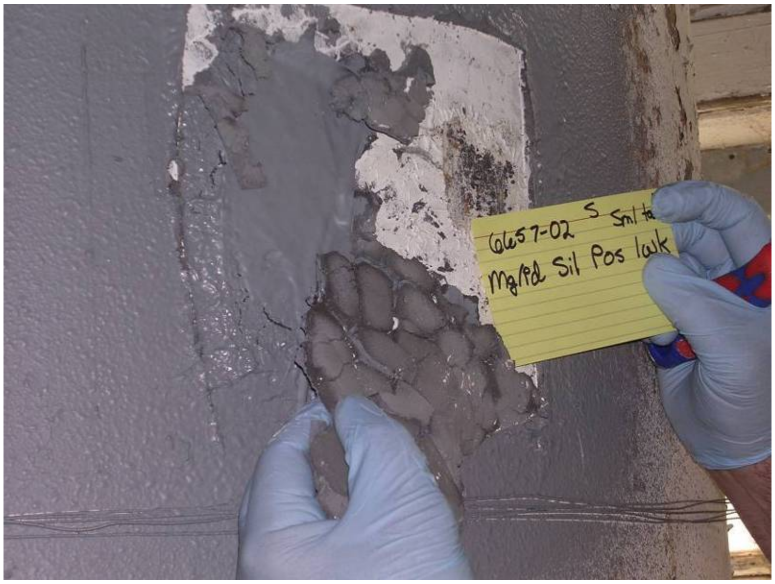


Figure 5-59. Small tank, three-square foot section treated with Mg/Pd-silicon

Figure 5-60 shows the status of the large tank after two weeks. Figure 5-61 shows the status of the large tank after week two samples were taken and Figure 5-62 shows the status of the small tank after week two samples were taken.

Figure 5-60. Large tank after two weeks of BTS treatment.

Figure 5-61. Large tank after sample was taken for two weeks of treatment.

Figure 5-62. Small tank after two week samples were taken.

Figure 5-63 shows that the 6657-02-NM-VP treated section is still intact after three weeks. Figure 5-64 shows the three week sample being taken and Figure 5-65 shows that the Mg/Pd-VP three week treatment section was also intact.

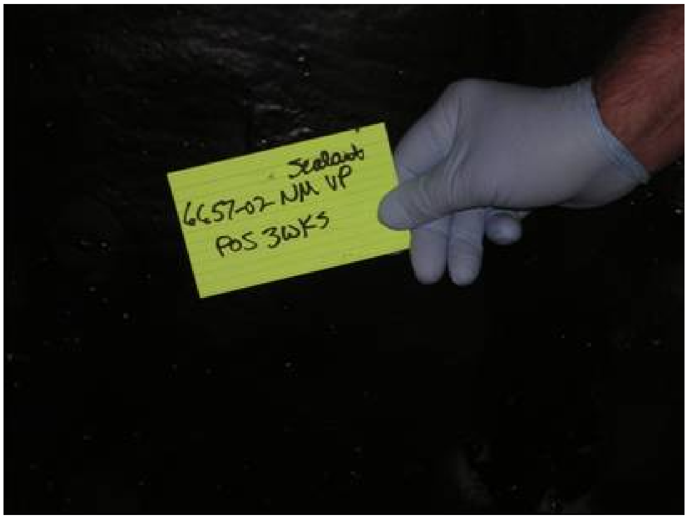


Figure 5-63. Large tank, NM-VP, still intact three weeks after application.

Figure 5-64. Sampling of large tank NM-VP, three weeks after application.

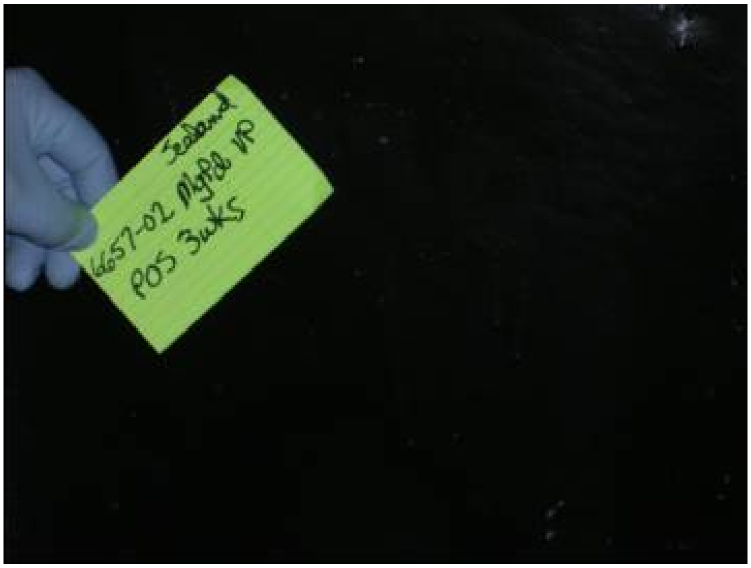


Figure 5-65. Mg/Pd-VP, large tank, three weeks after application.

Presses in Staging Area

Samples of paint, paste and sealant were taken from the presses one week and three weeks after treatment. All of the treatment locations were in good shape with no cracking of sealant or loss of BTS paste from the treatment areas. Figure 5-66 shows the treatment location on press 4 after one week of treatment. Figure 5-67 shows the treatment area after the VP had been removed.

Figure 5-68 shows the treatment area on press 15 after one week of treatment. Figure 5-69 shows the treatment area on press 15 three weeks after treatment.

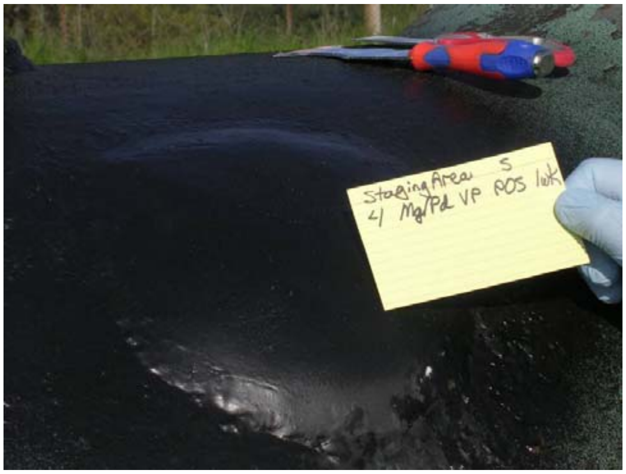


Figure 5-66. Treatment areas on press 4 one week after BTS application, prior to sampling.

Figure 5-67. Press 4, one week after BTS application, VP removed for sampling.

Figure 5-68. Press 15, one week after BTS application, prior to sampling.

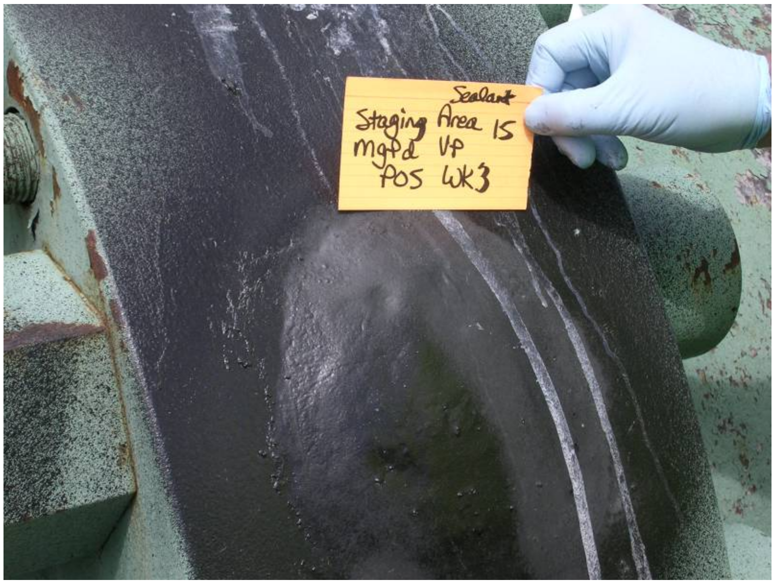


Figure 5-69. Press 15, three weeks after BTS application, prior to sampling.

5.6.4 Analytical Methods

Table 5-3 presents a summary of the analytical methods used in the demonstrations. Information pertaining to calibration of analytical equipment, quality assurance, decontamination and sample documentation can be found in Appendix B and C.

All samples were analyzed using EPA Method 8082 (PCBs by Gas Chromatography). During the development of BTS, a number of analytical methodologies have been developed in order to more clearly interpret PCB chromatograms. During the extraction of PCBs from aged paint or adhesive materials, the nominal extraction procedure using hexane pulls out not only PCBs but also other binding materials. These binding materials can cause irreversible contamination to the GC-MS column and make data interpretation more difficult. Through experimentation, it has been determined that if the material is first lightly dampened with toluene, then extracted in ethanol, made slightly polar with the addition of a small amount of water, and finally extracted again into non-polar hexane, the end chromatograph is cleaner thereby decreasing contamination to the GC column. Internal laboratory studies using controls have shown comparable recoveries using this toluene: ethanol:water into hexane extraction to the hexane: acetone mixture noted in EPA Methods 3550 and 8082.

Prior to analysis, samples were extracted using EPA Method 3550 (Ultrasonic Extraction). An ultrasonic dismembrator on pulse mode was used to sonicate solid samples into the solvent of choice. The only modifications to the extraction Method 3550 was that samples were centrifuged after sonication rather than using a glass wool filter to collect extracts for analysis, thereby reducing handling and waste. For samples which produced extremely dirty extracts (colored or cloudy), a sulfuric acid cleanup outlined in EPA Method 3665 (Sulfuric Acid/Permanganate Cleanup) requiring mixing of 50% sulfuric acid with the solvent extract was used to clean the extract prior to analysis.

This method produces a clean PCB peak envelope that allows a greater signal-to-noise ratio and therefore simpler, more accurate quantification. When these procedures are performed, the limit of detection of PCBs becomes approximately 50 ppb on the gas chromatograph electron capture detector (GC-ECD) and 20 ppb on the gas chromatograph mass spectrometer (GC-MS), both of which are well below TSCA level of 50 mg/kg for hazardous waste.

BTS paste is treated in a similar manner as the paint but with longer ultrasound times and then centrifuged and acid washed with sulfuric acid as described above. Some samples require multiple sulfuric acid washes to produce a clear sample for analysis.

TABLE 5-3
SUMMARY OF SAMPLE HANDLING AND LABORATORY ANALYTICAL DETAILS

	Parameter	Analytical Method	Method Number	Analytical Laboratory [(1)]	Quantitation Limit	Sample Container	Preservative	Holding Time
PASTE	PCB and degradation products in paste	Gas Chromatography	EPA 8082	UCF/KSC	0.10 mg/kg	plastic bottle for collection/glass for extraction	none	N/A
	Moisture Content of BTS	Visual Inspection Manual Inpection	N/A	N/A	N/A	N/A	N/A	N/A
PAINT	PCB and degradation products in paint	Gas Chromatography	EPA 8082	UCF/KSC	0.10 mg/kg	plastic bottle for collection/glass for extraction	none	n/a
	Adhesion of paint	Field Test of Pull-Off Strength of Coatings	ASTM - D4541-02	N/A	N/A	N/A	N/A	N/A
SEALANT	Sealant integrity	Visual Inspection	NA	NA	NA	NA	NA	NA
	PCB and degradation products in sealant	Gas Chromatography	EPA 8082	UCF/KSC	0.10 mg/kg	plastic bottle for collection/glass for extraction	none	N/A

Notes:

N/A - Not Applicable

UCF - University of Central Florida

KSC - Kennedy Space Center

(1) - 10% of the samples will be sent to a conmerical laboratory (to be determined) for confirmatory analysis.

TBD - To be determined

The procedure for extraction/analysis of the BTS material is given below:

1. weight ~0.7 g of paste material,
2. add 10 mL toluene,
3. sonicate mixture for 90 minutes (maximum power, Aquasonic Model 750),
4. centrifuge mixture for ~6 minutes,
5. acid wash mixture using conc. H_2SO_4 (1:1 ratio by volume),
6. remove top layer of resulting mixture and centrifuge for ~6 minutes,
7. permanganate wash mixture using 5% KMnO4 solution (1:1 ratio by volume), and
8. remove top layer, dilute as necessary for analysis by GC-ECD.

5.7 Sampling Results

This section provides a detailed summary of all sampling results; Section 5.7.1 provides the results of the Vertical Integration Building; Section 5.7.2 provides the results of the Badger demonstration; and Section 5.7.3 presents the results of the paste optimization studies. Tables summarizing sampling results can be found in Appendix D.

5.7.1 Vertical Integration Building

PCB Analyses

Pre Demonstration PCB concentrations are shown for all samples in Table D-1, and all samples tested were identified as PCB congener mix 1260. Table D-1 presents the pre-treatment concentrations (mg PCB/kg paint) from various VIB test panels. Figure 5-4 shows the locations of the samples from each panel. As can be seen from the data in Table D-1, concentrations of PCBs in the VIB samples ranged from ~10 to ~100 mg PCB/kg paint. The higher concentration samples (~50,000 mg/kg), (I-Beam 21) previously discussed in the demonstration plan (Demonstration Plan 1, February 2009) were removed during the expedited demolition process of the VIB, and similarly high level samples could not be located in the remaining samples available at the time of the BTS demonstration. However, these lower concentrations allow for the testing to prove the capability of BTS to remediate contaminated building materials to below current regulatory limits (50 mg/kg), as well as show the utility of the technology for the removal of PCBs at levels below 50 mg/kg. This may prove to be important if the technology was ever employed in a country with more stringent limits (for example, in Japan where current regulatory limits for PCBs are 0.5 mg/kg) or if the regulatory limits in the United States are ever reduced to lower levels.

Analysis of exposed paint was performed at Kennedy Space Center in the same manner as the pre-demonstration samples and controls. Table D-1 shows the post-treatment concentrations, length of exposure, and % removal (compared to pre-demonstration concentrations). The data clearly shows that high levels of removal of PCBs from the paint were achieved with both the active and inactive paste formulations. The average removal of PCBs from the paint from the entire test site was nearly 80% with a single application. Several samples were remediated to PCB below detection limits. BTS is capable of removing PCBs from contaminated paints to below the 50 mg/kg concentration level.

As stated above, analysis of BTS material itself was performed at the University of Central Florida. The data produced from the analysis of the BTS is given in Table D-1.

The loss of PCBs in the paint and the presence of PCBs within the BTS confirm the technology's ability to remove chlorinated contaminants from building materials such as paint. The low levels found within the paste are not surprising, considering the relative volumes of treated paint and BTS used (thin layer of paint, thick layer of paste resulting in dilution of the PCB concentrations). However, even though we can say BTS efficiently removes PCBs, it is difficult to make a quantitative analysis of this removal capacity from an examination of the paste, due to the low levels of PCBs originally found in the VIB paint (and consequently in the BTS material). Dechlorination of the original PCB envelope (mixture of PCB congeners) was demonstrated by the formation of the lower chlorinated byproducts and a corresponding PCB envelope shift. Results from the Badger demonstration, where starting concentrations of PCBs in the paint and subsequently in the paste were higher, are presented below in Section 5.6.2.

Adhesion and Structural Integrity Assessments Post-BTS Exposure

Qualitative and quantitative analysis of the painted surfaces were performed following the application and removal of BTS from the different test panels. Initially, a visual inspection indicated that the paint appeared to remain intact following exposure to BTS, however a closer look showed that the upper coat of paint was softened to the point that care had to be taken in the removal of the paste to ensure paint wasn't included in the paste sample (skewing results). This indicated that adhesion/integrity of at least the surface layer of paint was negatively impacted from exposure to the BTS solvent system.

There are several published methodologies for measuring adhesion of coatings. A qualitative method for such a determination is ASTM D3359-02, in which pressure-sensitive tape is applied and removed from cuts made into the coating surface. After removal and assessment of the amount of coating removed by the tape is made (visually), the test is either scored on the 0 to 5 scale or by comparison to descriptions/illustrations. Two test scoring methods are part of ASTM D3359-02, and the selection of which to use depends on the thickness of the coating involved. Testing showed that the paint on the VIB test panels was generally thicker than 5 mils (125µm), indicating Test Method A, the scoring from 0 to 5, was the method to be employed the VIB. This consists of making an X-cut through the film to the substrate, followed by applying pressure-sensitive tape over the cut and removing it. A qualitative assessment of the adhesion is then made and a score of 0 (complete removal) to 5 (no peeling/removal) is given.

Untreated areas of panel 3 and 4 were tested as a control, to determine the adhesion qualities of the paint prior to BTS exposure. Figure 5-70 shows untreated panel 3 before and after the pressure-sensitive tape has been applied and removed, while Figure 5-71 similarly shows untreated panel 4. Both panels (untreated) were scored a 5, indicating no peeling or removal of the paint upon removal of the pressure-sensitive tape. This was the expected result, since neither sample had been exposed to the paint softening effects of BTS.

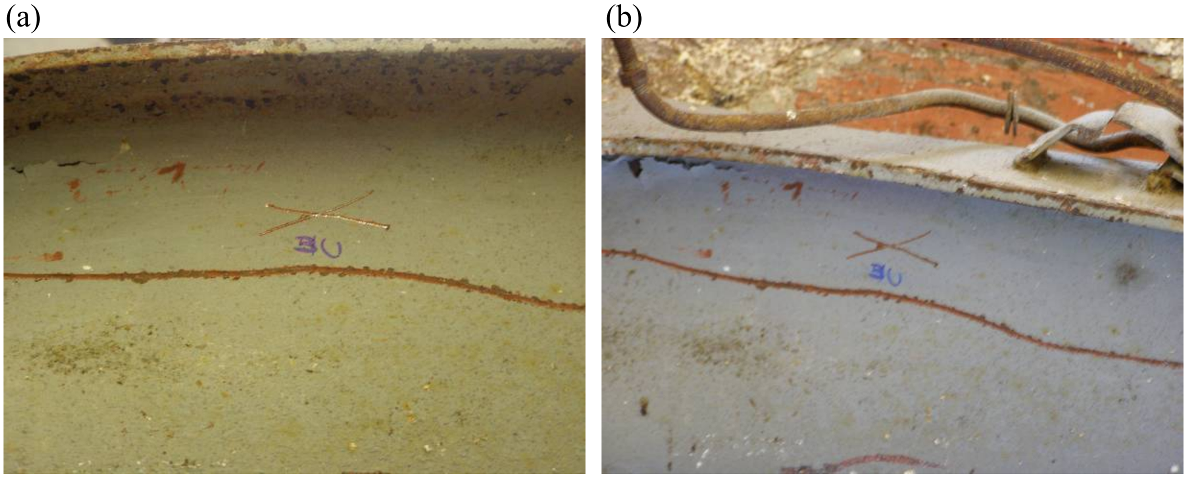


Figure 5-70. Qualitative Adhesion test on Panel 3 (untreated) pre (a)/post (b)

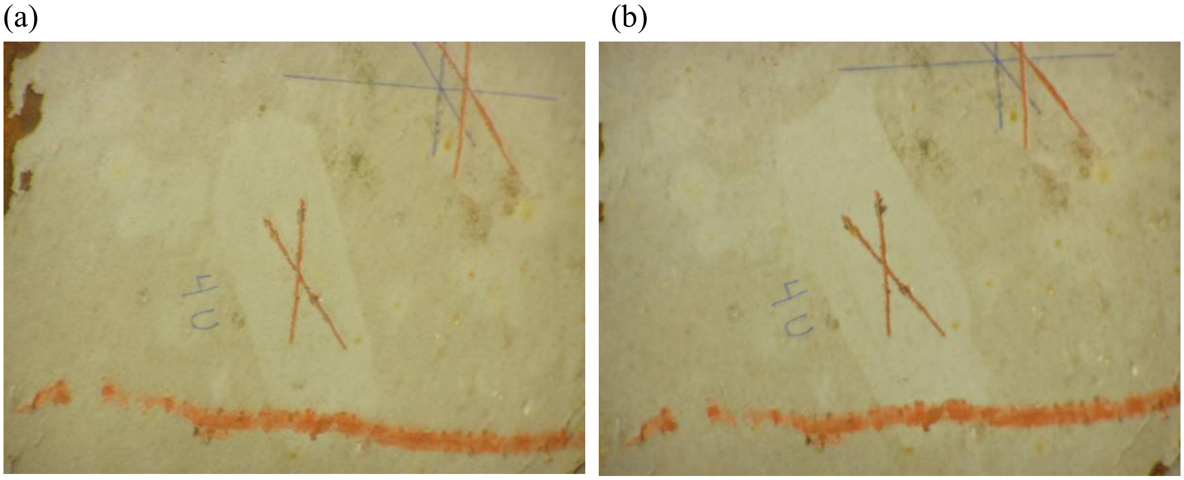


Figure 5-71. Qualitative Adhesion test on Panel 4 (untreated) pre (a)/post (b)

Treated areas of panels 3, 4, and 5 were tested using this qualitative method. Pre/post images of panels 3, 4, and 5 are shown in Figures 5-72, 5-73, 5-74, respectively. These three samples showed varying amounts of impairment of the adhesion qualities of upper layer of paint. Panel 3 showed removal of the paint in small patches between the X-cut of the incision, as well as beyond. According to the scoring criteria, if paint is removed from beyond the area between the acute angles of the X-cut, a score of 0 is assessed for the sample. Panel 4 showed no removal of the paint following the removal of the pressure-sensitive tape, and was given a qualitative score of 5. However this was most likely due to the fact that poor contact was established between the painted surface and tape. Although an effort was made to clean/dry the surface prior to adhesion testing, care had to be taken to ensure the coating was not damaged/removed during the cleaning process. As a result, an oily sheen was visible on the surface of the paint which most likely interfered with an accurate evaluation of the adhesion properties for this sample. Panel 5 showed the most damage to the adhesive properties of the painted surface from exposure to BTS. All of the paint between the X-cut (and beyond) was removed when the tape was removed, which led to a qualitative score of 0 for this test sample. Initial testing seems to indicate a loss of adhesion to samples exposed to BTS. The data for both treated and untreated sample areas is given in Table 5-4.

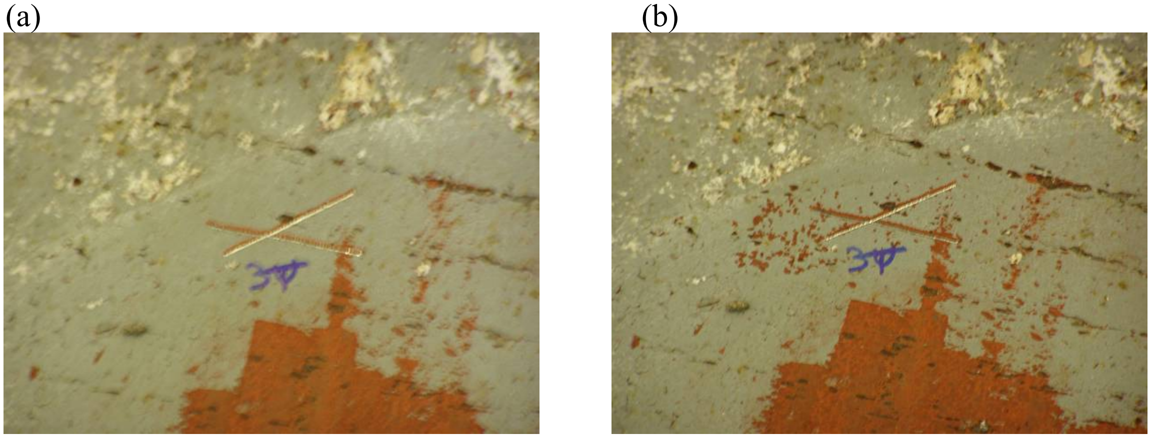


Figure 5-72. Qualitative Adhesion test on Panel 3 (treated) pre (a)/post (b)

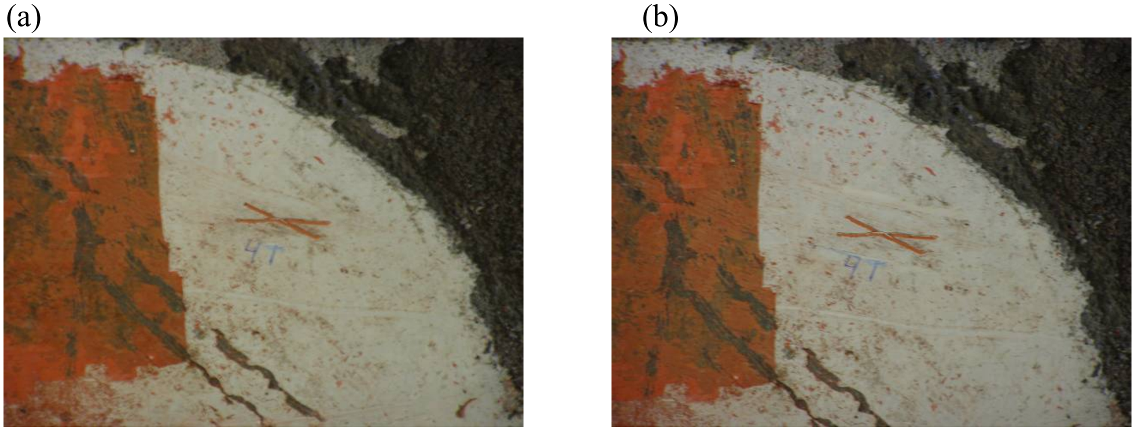


Figure 5-73. Qualitative Adhesion test on Panel 4 (treated) pre/post

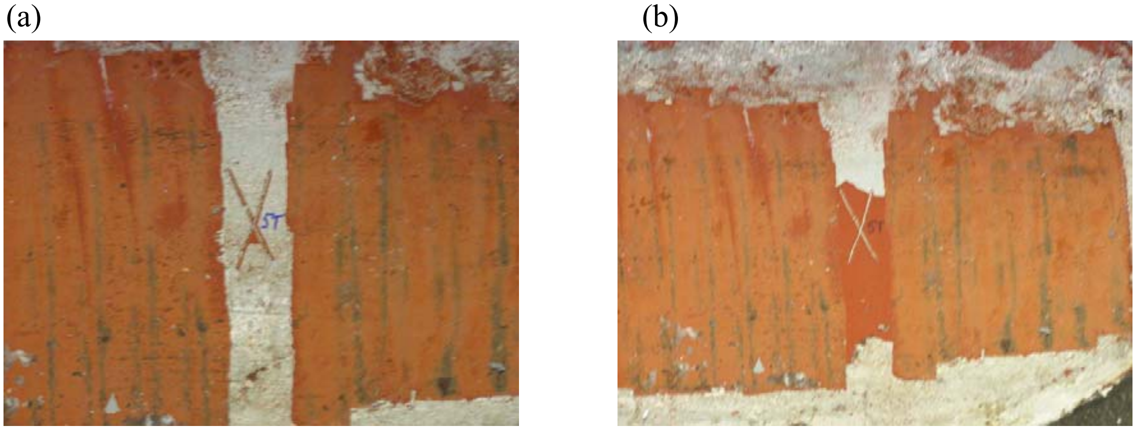


Figure 5-74. Qualitative Adhesion test on Panel 5 (treated) pre (a)/post (b)

Table 5-4. Qualitative analysis of adhesion properties using ASTM D3359-02 for both treated/untreated sample areas.

Panel	Treated	Score
3	Yes	0
4	Yes	5
	No	5
5	Yes	0
	No	5

In several cases, tests were not deemed accurate due to an inability to form sufficient contact between the pressure-sensitive tape and coated surface, leading to a skewing in the results. It was decided that a second test method should be employed o further evaluate the effects of BTS on the adhesion of the paint. A quantitative analysis method of adhesion properties is given in ASTM D4541-02. The end result of this analysis technique is to determine the greatest perpendicular force (in tension) that can be handled by a coated surface before the coating itself is detached. Suitable sample areas were chosen for both untreated (control) and treated areas of VIB test panels and carefully cleaned. A loading fixture is affixed to the sample site using an epoxy adhesive. The samples were allowed to cure for 24hr, at which point they were removed with a Type IV self-alignment adhesion tester (see Appendix A for details on analytical method) using a self-contained pressure source. The final values are presented as force (psi) required to remove the loading fixture from the coated material. Qualitative assessments are made to the amount of material affixed to the loading fixture. This data is presented in Table 5-5.

Table 5-5. Quantitative analysis of adhesion properties using ASTM D4541-02 for both treated/untreated sample areas.

Sample ID	Treated (Y/N)	Force (psi)	Comments
3-1a	Y	459.77	
	N	329.15	Overspray from coating may have affected adhesion
3-1b	Y	845.48	Poor coating caused BTS to dry out quickly
4-5a	Y	208.74	
	N	1353.64	
4-5b	Y	157.72	
	N	382.21	Didn't removal all paint, possibly poor adhesion
5-1	N	804.66	
5-7	N	1282.21	
7-1a	Y	196.50	
	N	761.81	
7-1b	N	623.03	

Less force was necessary to remove the loading fixtures after BTS application had occurred compared to the force necessary to remove the fixtures from untreated samples. A few samples didn’t follow this trend, but this was likely due to the sample surface not being properly prepared resulting in an incomplete bonding of the epoxy material used. Even without removing these possible outliers, the average force necessary to remove treated surfaces was approximately 374 psi compared to approximately 791 psi for untreated surfaces. This is indicative of loss of the adhesive forces between the surface coat and material below the surface. It is interesting to note that in the majority of the tests (both qualitative and quantitative); the loss of adhesion was found to occur primarily between the surface coat and the primer coat. The primer coat itself remained intact after prolonged exposure to BTS, indicating that the majority of structural integrity loss is occurring within the surface coat. It may be that while the surface coat may need reapplication post-exposure to BTS, the primer coat would be suitable for use without removal/replacement.

5.7.2 Badger Army Ammunition Plant

PCB Analyses

Press House 6810-11

Table D-2 gives the PCB concentrations in the paint located at the different treatment sites in the 6810-11 building at times of pre-demonstration, and after one, two and three weeks of treatment. The PCB concentrations in the paint for each of the three test areas as a function of time are shown in Figure 5-75. As can be seen from the data, the major drop in PCB concentration occurs in the first week. The concentrations of PCBs in the paint do not change significantly between week 1 and week 3. The PCB concentrations in the paint at each sample site was so heterogeneous (as evidenced by the different levels of paint painted surface) that small differences in PCB concentrations at each test site between weeks 1, 2, and 3 samples are not meaningful.

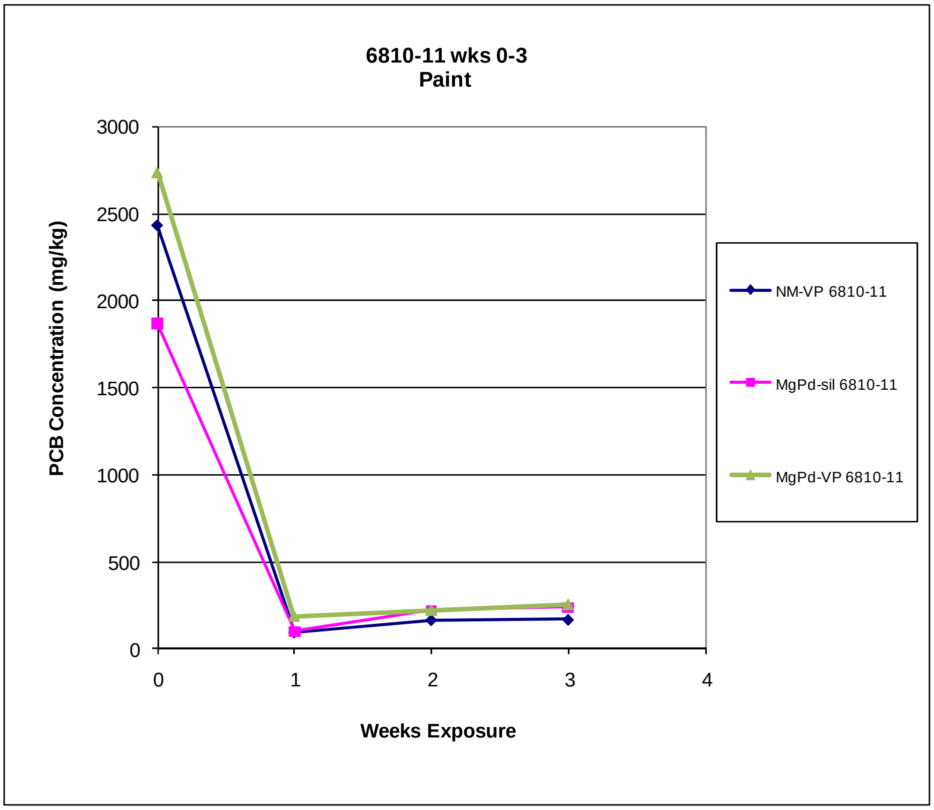


Figure 5-75. Average Concentrations of PCBs in paint at 6810-11 during treatment times of 0-3 weeks.

PCB concentrations in paint in building 6810-11 were between 1,800 mg/kg and 3,880 mg/kg in the pre-demonstration samples. Post-treatment concentrations (1 to 3 weeks of treatment) in paint were between 96 mg/kg and 200 mg/kg. The target of 50 mg/kg in the paint was not achieved with high starting concentrations in the paint because the PCBs extracted into the paste exceeded the degradation capabilities of the Mg/Pd and then the paste became saturated with PCBs. The paste was no longer able to further extract PCBs from the paint. It may require second application to get all concentrations below 50 mg/kg with starting concentrations above 1,500 mg/kg. In addition, there were issues with some of the sealant performance at the low temperatures that added to the inability to meet the 50 mg/kg target in the pain. Observations during this demonstration suggest that the low winter temperatures during the demonstration at Badger caused cracking in the sealant and made it difficult to control evaporation of the solvent. It is recommended that future applications not be conducted during cold winter months.

Samples of BTS paste were collected from each test location at the same time after treatment that paint samples were collected. These samples were analyzed for PCB concentrations by the procedure that is described in Section 5.5. The data from these analyses is given in Table D-2 and the data is plotted in Figure 5-76.

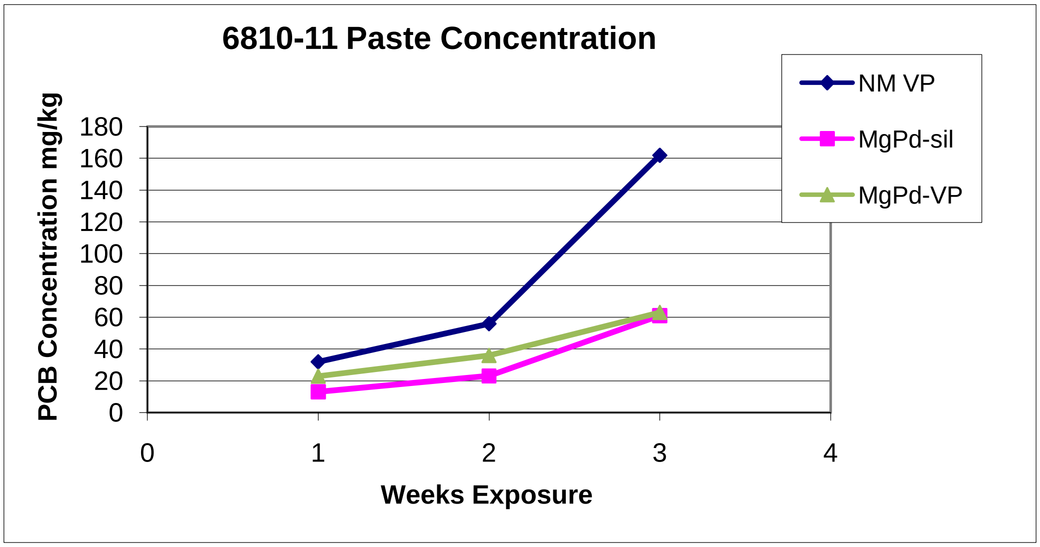


Figure 5-76. Average PCB concentration in paste from building 6810-11 for weeks one through three.

The PCB concentrations in the Mg/Pd BTS were lower than the NM paste. This is as expected because the active Mg/Pd particles are continuously degrading PCBs as they enter the paste. The concentrations of the PCBs in the paste are those that were present in the paste soon after the samples were collected and returned to UCF for analysis. Section 5.6.3 addresses the issue of how PCB degradation continued with time in the Mg/Pd BTS paste and how the PCBs were degraded in the NM BTS after Mg/Pd was added to the paste in the laboratory. The PCB concentration in the paste continued to increase in weeks two and three even though little to no additional PCBs were being removed from the paint. It is believed that there was still some solvent evaporation from the paste over time and after sampling which led to a concentrating effect. Because the PCB concentration in paste is reported on a weight basis, as the solvent in the paste evaporates, the PCB concentration increases.

Samples of concrete were taken prior to treatment and after three weeks of treatment to determine if the BTS paste would transport any PCBs into the concrete. The samples were taken from the surface (after paint removal) to a depth of 3/8 inches into the concrete at several locations in the 6810-11 test area. Table D-3 presents the data for the PCB levels in the concrete prior to treatment and then after three weeks of exposure to BTS paste. The data suggest that BTS paste does not transport PCBs from the paint into the concrete but rather transports PCBs from the concrete into the paste. This property of BTS paste has also been observed and confirmed in laboratory studies.

Both the VP and the Sil sealants from the 6810-11 test site were removed and returned to UCF for analysis. Analysis of the sealant samples showed that there were no detectable PCBs found in any of the sealant.

Press House 6810-36

The data for all of the paint and paste samples for the 6810-36 site are given in Table D-4 and concrete sample data are shown in Table D-5. The data for paint samples are plotted in Figures 5-77 and the data for paste samples are plotted in Figure 5-78. The data for the 6810-36 test site show significant reduction in the PCB levels from the paint as was also observed for the 6810-11 site. In addition, the concrete samples document that the BTS paste does not transport PCBs into the concrete but transports PCBs out of the concrete. As with the sealant from the 6810-11 site, sealant samples from the 6810-36 site had no detectable PCBs.

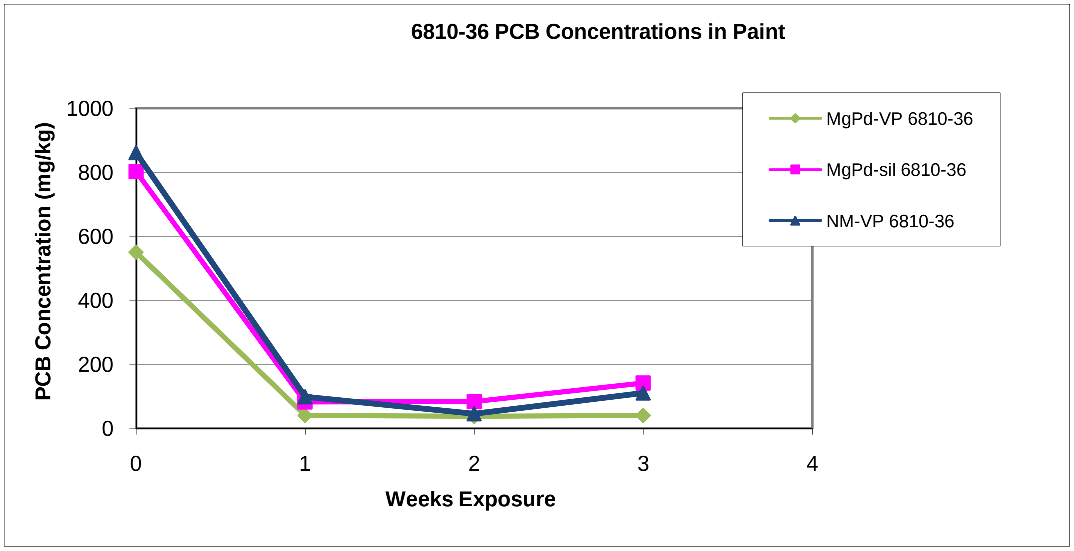


Figure 5-77. Average PCB concentrations in paint samples from 6810-36.

PCB concentrations in paint in building 6810-36 were between ~ 400 mg/kg to as high as ~900 mg/kg prior to treatment. Post treatment concentrations in paint were between 96 mg/kg and 200 mg/kg. PCBs concentrations decreased after one week of BTS application but would appear to require second application to get all concentrations below 50 mg/kg. It is believed that target of 50 mg/kg of PCBs in the paint was not achieved in part because the PCBs extracted into the paste may have exceeded the degradation capabilities of the Mg/Pd in the paste for the Mg/Pd paste and the concentration of PCBs in the paste increased. For the NM BTS the paste had an elevated concentration of PCBs that was no longer able to extract additional PCBs from the paint. The performance of the BTS paste was also negatively impacted by the conditions of the paint at this building which prevented achieving a good seal of the BTS and resulted in the loss of ethanol from the paste. In addition, the paste was applied to a porous surface that reduced the amount of ethanol available in the paste (there was some loss of ethanol to the concrete). The starting concentrations in the paint were similar to those in the Staging Area (see below) and all the treated paint in the staging area was treated to below 50 mg/kg. In the Staging Area, the BTS was applied to painted metal and the paint was in better conditions that that on the painted concrete in building 6810-36

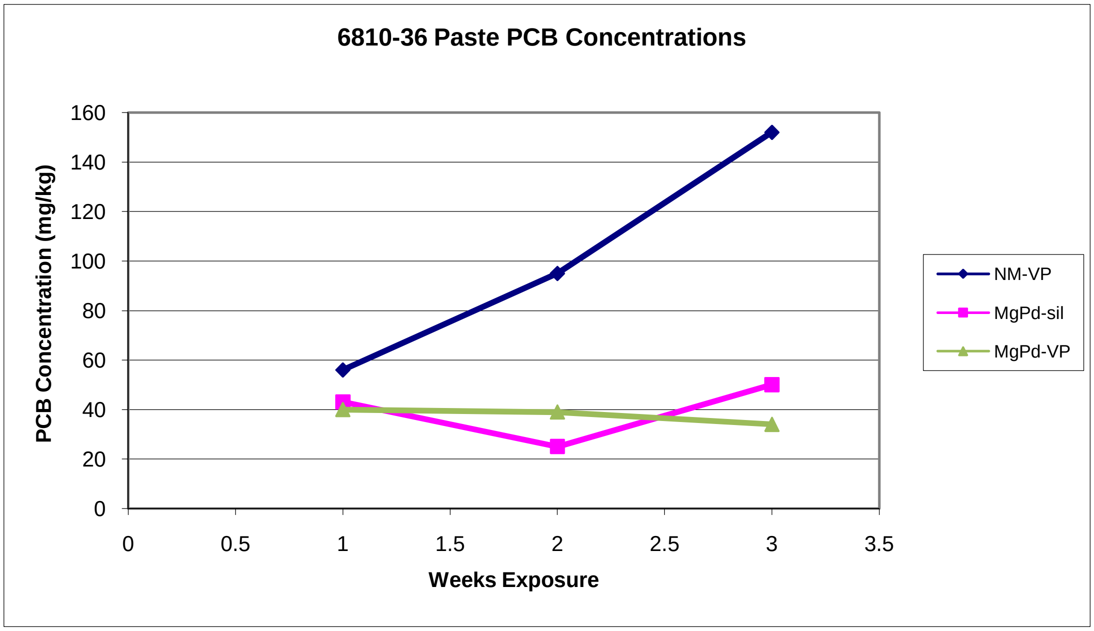


Figure 5-78. Average PCB concentrations in paste sampled at 1-3 weeks after BTS application, building 6810-36.

Figure 5-78 shows that the concentration of PCBs in the NM-VP paste was again higher than that of the other two sample locations (Mg/Pd-VP and Mg/Pd-Sil) as was seen in building 6810-11 samples. Table D-5 shows again (as was seen in 6810-11) that the PCB concentrations in the concrete underneath the BTS treatment actually have lower PCB concentrations after treatment. As mentioned above, this is as expected because the active Mg/Pd particles are continuously degrading PCBs as they enter the paste. The concentrations of the PCBs in the paste are those that were present in the paste soon after the samples were collected and returned to UCF for analysis. Section 5.6.3 addresses the issue of how PCB degradation continued with time in the Mg/Pd BTS paste and how the PCBs were degraded in the NM BTS after Mg/Pd and solvent were added to the paste in the laboratory. The PCB concentration in the paste continued to increase in weeks two and three even though little to no additional PCBs were being removed from the paint. It is believed that there was still some solvent evaporation from the paste over time and after sampling that led to a decrease in the mass of paste. Because the PCB concentration in paste is reported on a weight basis, as the solvent in the paste evaporates, the PCB concentration increases.

All of the VP and Sil sealant from the 6810-36 test site was removed and returned to UCF for analysis. Analysis of the sealant samples confirmed that there were no PCBs found at detectable levels.

Building 6657-02: Nitrating House

Table D-6 gives the PCB concentration prior to treatment and after weeks one, two and three of treatment for Building 6657-02. The data are also plotted in Figure 5-79. In fact, these are the highest PCB concentrations that the BTS paste has ever been tested on. These high levels may be due to the fact that the tanks were coated with several layers of paint. Even though the BTS treatment was able to remove more than 50% of the PCBs from the paint after only one week of exposure, high concentration were still left in the paint. Laboratory studies have shown that where PCBs are located in multiple layers of paint, the transport of the PCBs into the paste is much slower from the deeper paint layers. In addition, as mentioned above, the paste becomes more concentrated with PCBs and is no longer able to extract the PCBs from the paint. Such coatings require multiple applications of BTS in order to reach levels lower than 50 mg/kg. Paste sample data from 6657-02 is shown in Table D-6 with a graphical representation shown in Figure 5-80.

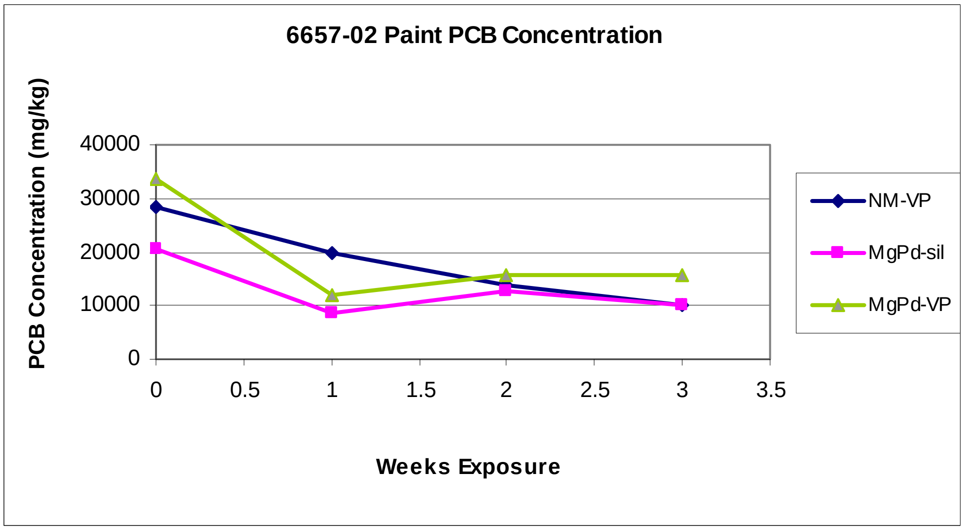


Figure 5-79. Average PCB concentrations in paint samples taken from 0-3 weeks after BTS application.

The PCB concentration data from the paste from 6657-02 are more scattered than what was observed for 681-11 and 6810-36. It is believed that this to be due to some paint chip contamination in the paste. Before the paste samples were analyzed, attempts were made to remove all visual paint chips from the paste. However, some of the smaller chips could have been missed resulting in higher than expected PCB concentrations in the paste.

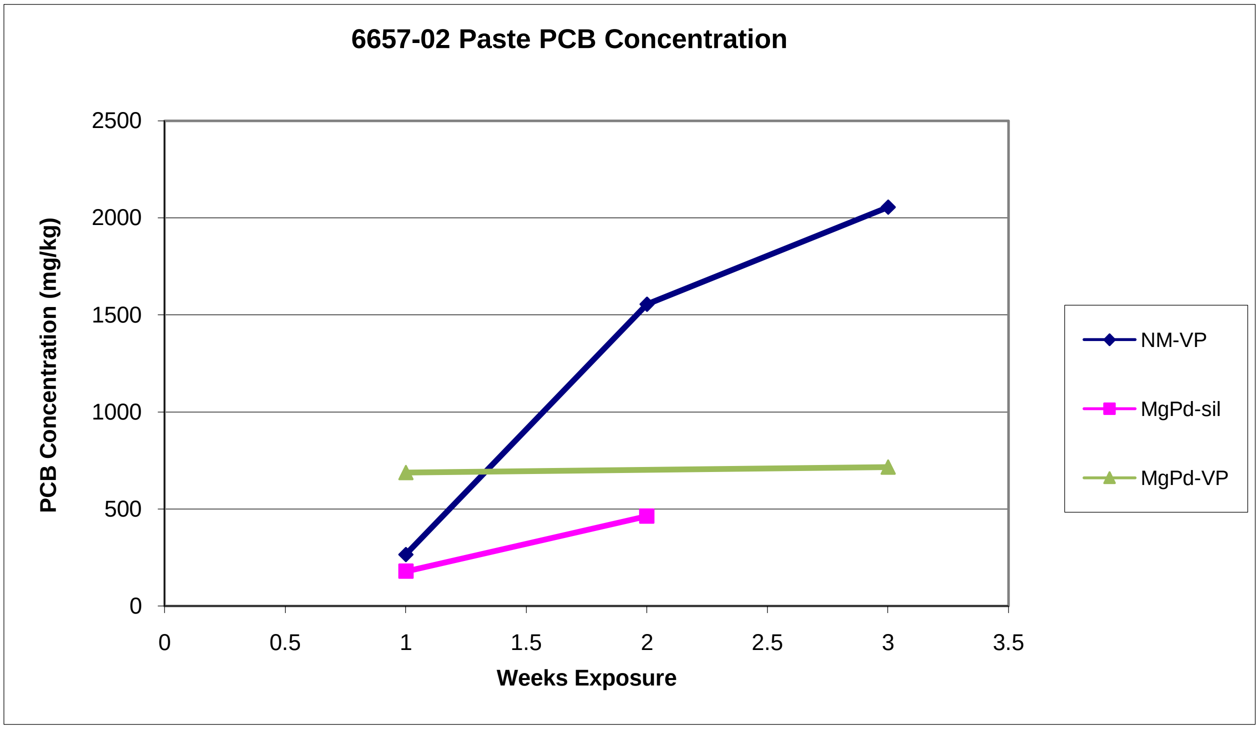


Figure 5-80. Average PCB concentration in pastes from building 6657-02 for weeks 1-3.

The extraordinarily high concentration of PCBs in the paint at this location presented a scenario where it was not possible to achieve reduction to 50 mg/kg (from as high as 5% PCB by mass) in one application. The very high concentrations yield a situation where more PCBs are present than can be handled by the bimetal available in the paste or degraded sufficiently in the time period that solvent remains in the paste to facilitate treatment. As mentioned above, the paste becomes saturated with PCBs and is no longer able to extract the PCBs from the paint. Figure 5-80 shows that the concentration of PCBs in the NM-VP paste was again higher than that of the other two sample locations (Mg/Pd-VP and Mg/Pd-Sil) as was seen in building 6810-11 samples.

All of the sealant, both the VP and the Sil, from the building 6657-02 test site was removed and returned to UCF for analysis. Analysis of the sealant samples confirmed that there were no PCBs found at detectable levels.

Presses in the Staging Area

The PCB concentrations in the paint samples collected from the presses in the staging area are shown in Table D-7. The data for these samples is plotted in Figure 5-81. As can be seen from the data, the PCB concentrations in the paint on the presses are much lower than those that were observed at other test locations on the walls and on the tanks. At all of the sample sites that were treated, the PCB levels were reduced to very low levels, below the regulatory limit of 50 mg/kg after only one week of exposure. Table D-7 lists the concentrations of PCB found in the paste samples taken from all five presses treated in the staging area and the data is plotted in Figure 5-82.

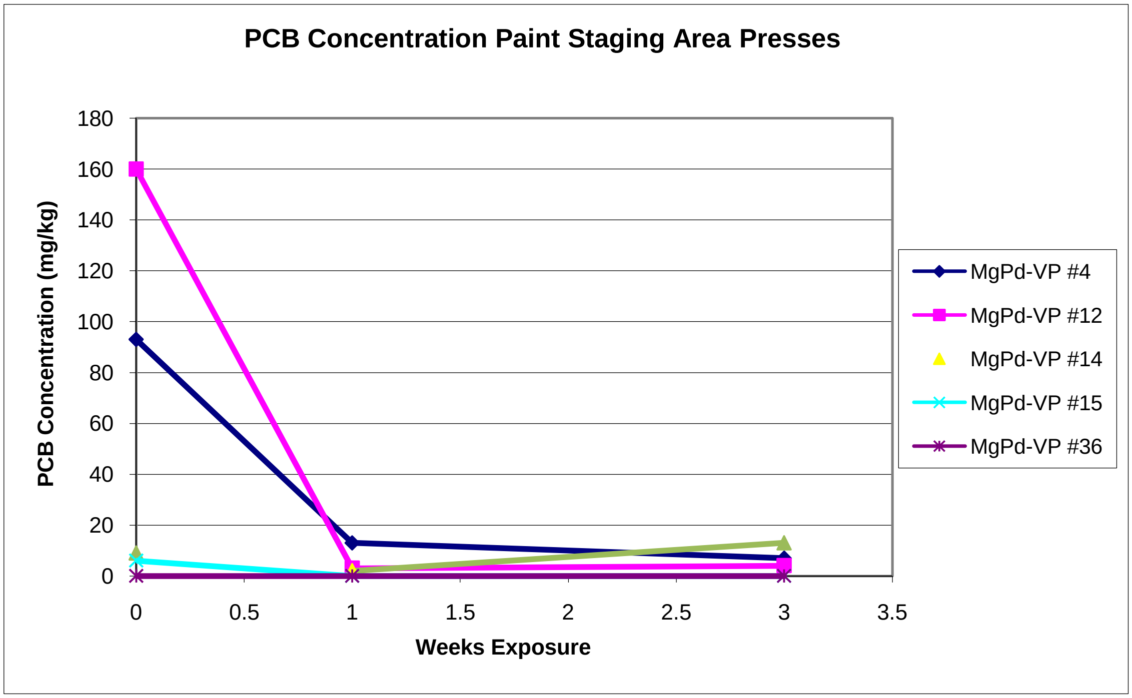


Figure 5-81. Average PCB concentrations in paint samples from various staging area equipment at 0, 1, and three weeks after BTS exposure.

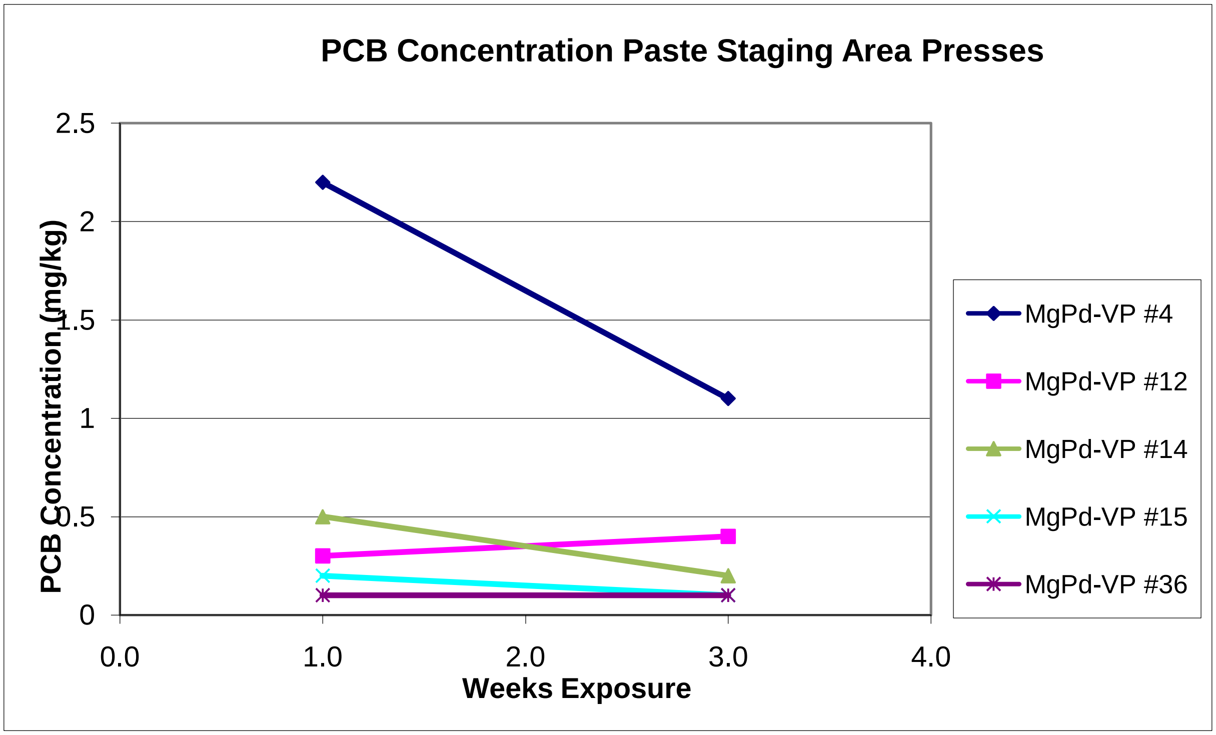


Figure 5-82. PCB concentration in paste from staging area (all presses) for weeks 1 and 3.

All of the sealant, both the vinyl polymer and the silicon polymer from the staging area test site was removed and returned to UCF for analysis. Analysis of the sealant samples confirmed that there were no PCBs found at detectable levels.

Adhesion and Structural Integrity Assessments Post-BTS Exposure

As stated in the Badger demonstration plan (Demonstration Plan 2, February 2009), qualitative and quantitative analysis of painted surfaces at the various test sites were to be performed following the application and removal of BTS from the test surfaces. Initial visual inspection of the painted surfaces at the four different test sites, two concrete press bunkers, the nitrating house and the staging area, indicated that the paint was in poor condition prior to treatment with BTS. Significant peeling and flaking of the paint was evident at each of the test sites. Figure 5-83 gives a representative view of the quality of the paint in the concrete bunkers.

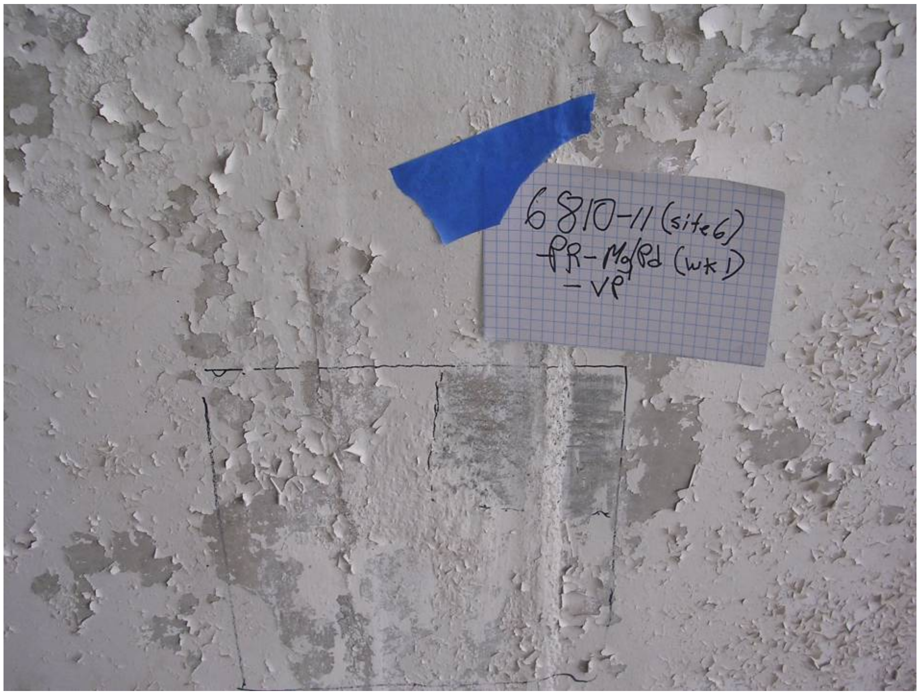


Figure 5-83. Representative quality of the paint in concrete bunker 6810-11.

Figure 5-84 gives a representative view of the quality of the paint on the tanks in the nitrating house and Figure 5-85 gives a representative view of the quality of the paint on the presses in the staging area.

Figure 5-84. Quality of the paint on the tanks in the nitrating house.

Figure 5-85. Quality of the paint on the presses in the staging area.

As with the VIB, ASTM D3359-02 was used to evaluate the adhesion of the paint pre and post-treatment. However, when the paint at the Badger test sites was subjected to this procedure prior to any exposure to BTS, they all failed (all scored 0). Thus, the effect of the BTS on the integrity of the painted surfaces could not be assessed.

5.7.3 Paste Optimization Studies

As discussed in previous sections of this report, all of the BTS paste that was used in the Badger field test was returned to UCF for further study. Two types of paste were used in the field test, one contained Mg/Pd particles (Mg/Pd BTS) and the other contained no metal particles (NM BTS). Immediately after the paste samples were returned to UCF, they were analyzed for PCB concentrations. Samples of the paste that contained more than 10 mg/kg of PCBs were chosen for further degradation studies.

The additional degradation studies involved adding ethanol containing 1% acetic acid to the paste to re-wet the Mg/Pd containing paste. Sufficient acidified ethanol was added to make the paste take on a bread dough type of consistency. Ethanol had to be added to the paste because some of the paste had dried out *in situ* before removal from the test site. Some of the paste dried out during the field test because some of the sealant developed cracks allowing the ethanol to evaporate. This is believed to have been caused by the cold weather conditions during the demonstration at Badger. At the test sites where the sealant remained intact, the paste did not dry out during three weeks of exposure. In order for PCB degradation to take place, a proton donor solvent such as ethanol must be present. The reason for adding the acetic acid is that studies conducted at UCF after the Demonstration Plans were submitted showed that the addition of a small amount of acetic acid to the ethanol significantly increased the rate of PCB degradation. These studies also showed that acidified ethanol with Mg particles was as effective or in some cases more effective than the Mg/Pd particles in non-acidified ethanol at degrading PCBs. This discovery is extremely important because it means that Pd is not required in the BTS formulation, which reduces the cost of the BTS paste significantly. Plain acidified Mg particles were not used during the Badger test because the Demonstration Plan called for using BTS with Mg/Pd particles and BTS without any Mg/Pd particles. However, acidified Mg particles were tested on the BTS paste that was returned to UCF from the Badger test site. The data obtained for the PCB degradation performance of the acidified Mg particles are reported in this section along with the data obtained for the Mg/Pd particles.

For the purpose of illustrating how the PCB degradation process in the BTS paste is confirmed and documented, paste samples were spiked with Aroclor 1254 and then degradation was monitored over a period of one month. Figure 5-86 shows the chromatogram for a paste that was spiked to a concentration of 14.8 mg/kg (shown in black) at 0 hour. The sample was treated with Mg/Pd at a level of 10% and ethanol containing 1% (by volume) of acetic acid. The PCB concentration after one week is indicated by the chromatogram shown in red. Both the shift in the peak envelope as well as the lower peak areas indicate that PCB degradation has occurred and the concentration dropped to 6.8 mg/kg after one week.

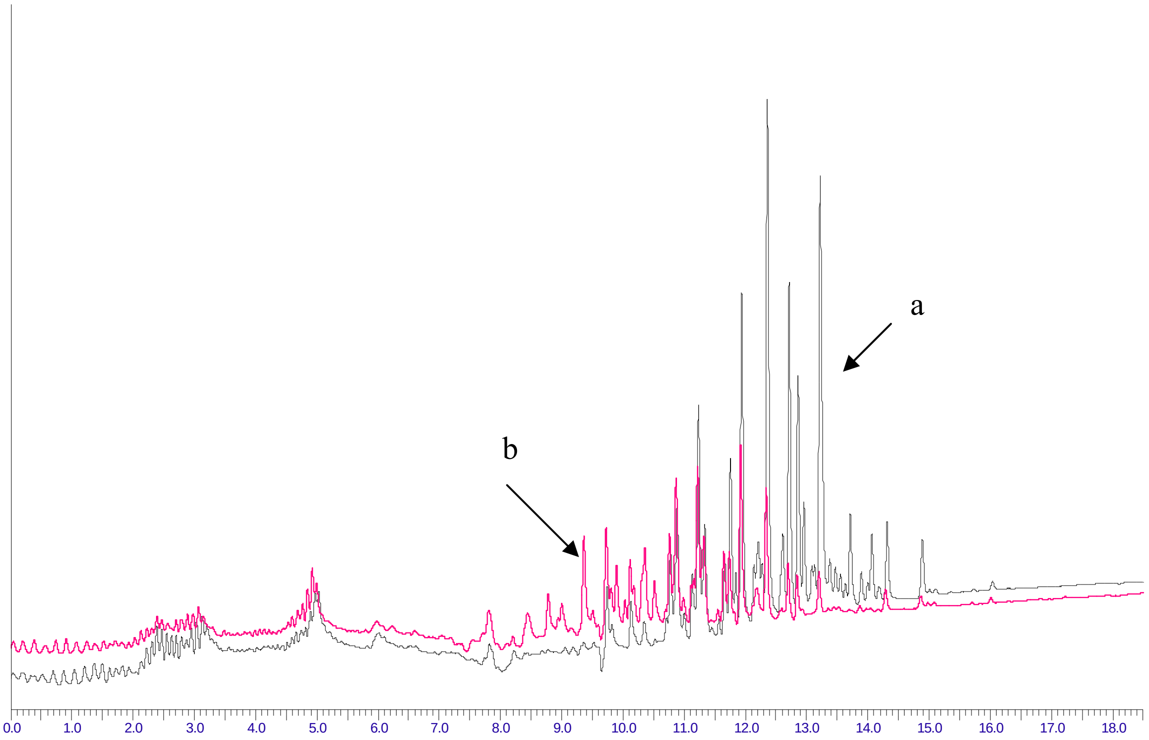


Figure 5-86. Chromatograms of BTS paste spiked with Aroclor 1254 and then treated with acidified Mg/Pd (a) time 0 hour and (b) after one week of treatment.

A second paste sample was spiked with Aroclor 1254 and then treated with Mg particles (10% by weight) and ethanol containing 1% acetic acid. The chromatograms at time equals 0 hour and after one week are shown in Figure 5-87. Again, as can be seen from the shift in the peak envelope and reduction in peak area, significant degradation occurred in one week from 14.8 mg/kg to 3.8 mg/kg.

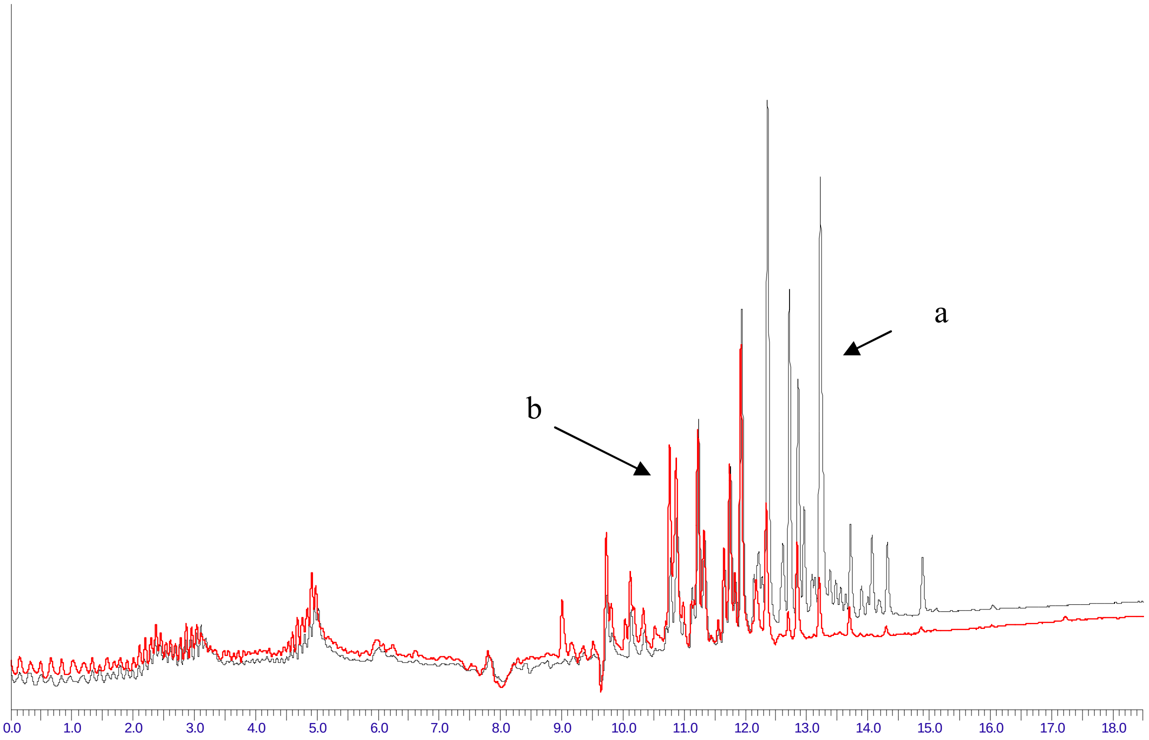


Figure 5-87. Chromatograms of BTS paste spiked with Aroclor 1254 and then treated with acidified Mg (a) time 0 hr, and (b) after one week of treatment.

After the spiked paste tests were completed, treatment of the Badger BTS paste was performed using both Mg/Pd and Mg in conjunction with the use of ethanol containing 1% acetic acid. Table 5-6 shows the results of the PCB degradation that occurred in the non-metal paste that had been recovered from various Badger test sites after Mg or Mg/Pd at 10% by weight had been added to the paste. Only samples from sites 6810-36, 6810-11 and 6657-02 were tested because

TABLE 5-6
PCB DEGRADATION OF NON-METAL BADGER PASTE SAMPLES AFTER THREE AND SEVEN DAYS OF TREATMENT WITH Mg OR Mg/Pd AND ETHANOL CONTAINING 1% BY VOLUME ACETIC ACID

		PCB Concentrations in BTS Paste							
		Mg				**Mg/Pd**			
Sample Name	**Original Conc(mg/kg)**	3 day	average	7 day	average	3 day	average	7 day	average
NM 6810-36 1wk	226	16 22	19	11 23	17	15 13	14	47 25	36
NM 6810-36 2wk	207	8 2	5	4 3	3	4 4	4	3 4	4
NM 6810-11 3wk	622	16 81	48	58 48	53	43 36	40	22 14	18
NM 6657-02 1wk	1544	123 22	72	36 23	29	107 87	97	97 115	106
NM 6657-02. 2wk	2797	32	46	22	29	154	171	129	128

Notes:

mg/kg - milligram per kilogram
Mg - magnesium
Mg/Pd - magnesium/palladium

as shown in the previous section, the PCB concentration in the paste samples taken from the staging area were so low (less than 2 mg/kg) that degradation studies on them would have been of no value.

As can be seen from the data presented in Table 5-6, significant PCB degradation was observed in the paste even after only three days of treatment. Evidence of this can be seen in the NM-6657-02 2 week sample, where the PCB levels dropped from an initial value of 2,797 mg/kg to 46 mg/kg after only three days of treatment with Mg. The other piece of information that is of significant importance is that the Mg particles performed as well as or better than the Mg/Pd particles. This is extremely important because the removal of the Pd from the BTS system reduces the cost of the treatment metal particles by more than 50%. In addition it eliminates potential concerns related to the release of Pd into the environment.

As discussed in the preceding paragraphs, evidence of PCB degradation is documented by the reduction in the area of the chromatographic peaks, and by the shift in the Aroclor peak envelope to lower molecular weight PCB congeners. Figure 5-88 shows the overlap of chromatograms of paste sample NM 6657-02 1 week sample, before treatment (1544 mg/kg PCB) and after three days of treatment with Mg (123 mg/kg). As can be seen from the chromatograms, there was significant reduction in the Aroclor envelope of peaks after treatment as well as a shift in the envelope. The samples were run at the same dilution and chromatograph response so that the chromatograms can be compared directly.

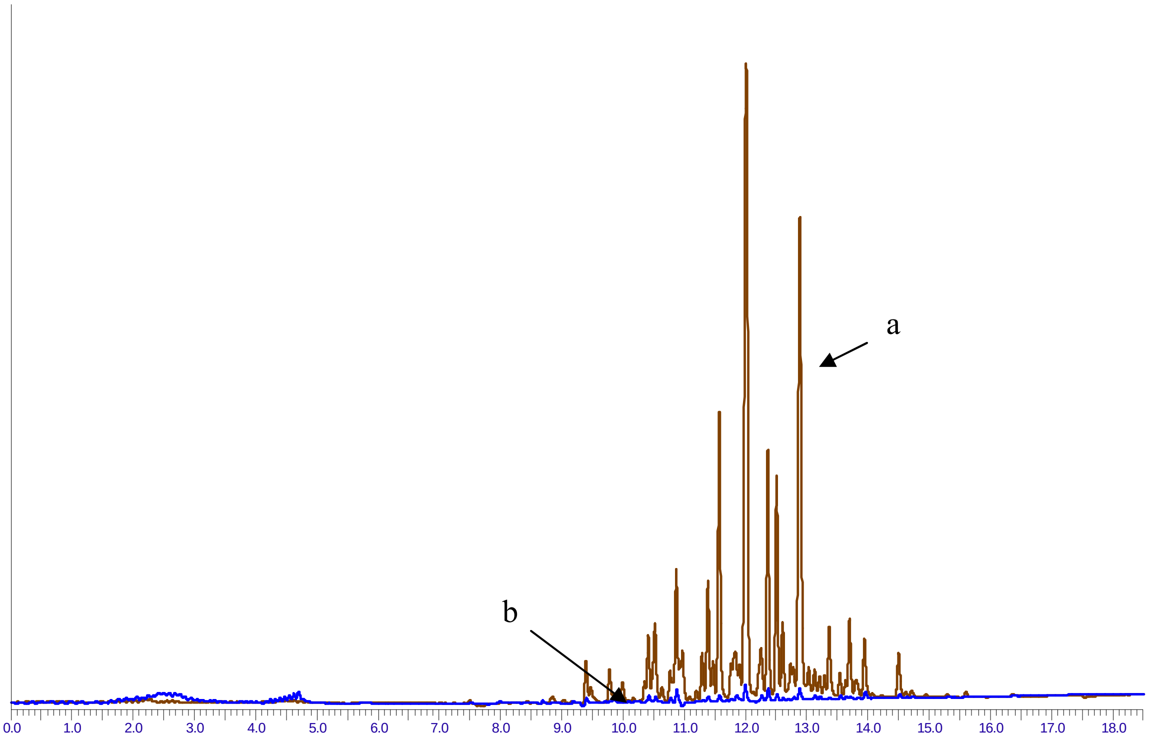


Figure 5-88. Overlap in chromatograms of paste sample NM 6657-02 1 week sample, before treatment (1544 mg/kg PCB) and after three days of treatment with Mg (123 mg/kg PCB). Data for (a) NM 6657-02 1week (original) 1544 mg/kg= **brown** and (b) after 3 days reacting with Mg metal 123 mg/kg= **blue.**

A shift in the Aroclor peak envelope can be seen clearly in Figure 5-89. What is presented in this figure is a chromatogram of an Aroclor standard sample and a paste sample from the NM6657-02 1week sample, after seven days of treatment with Mg metal. The peak areas cannot be compared because the samples are at different dilutions. However, the purpose of the Figure is to simply show the shift in the peak envelope as proof of the degradation process.

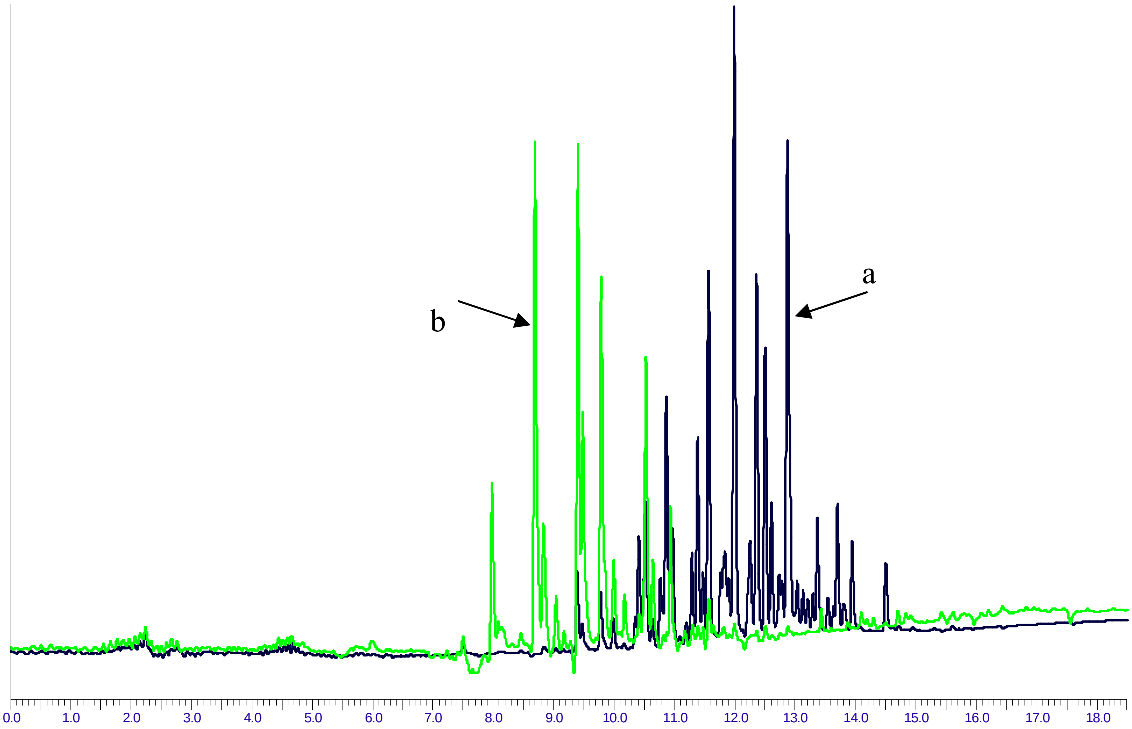


Figure 5-89. A shift in the Aroclor peak envelope with data for (a) Aroclor standard 1254= **black** and (b) Badger sample NM 6657-02-1 wk after reacting with Mg metal for 7 days= **green.** This is to show the change in peak shifting as the dechlorination reaction occurs. The concentrations are not meant to be compared.

Additional evidence of PCB degradation in the other Badger paste samples can be seen in Figures 5-90 and 5-91. Figure 5-90 shows the chromatograms for the paste sample obtained from the Badge NM6810-36-1wk sample site, at the original PCB concentration (226 mg/kg) and after 3 days of treatment with Mg particles (10 wt %) and acidified ethanol. Figure 5-91 shows chromatograms of the original paste and after 7 days of treatment. As can be seen from the chromatograms, proof of significant PCB degradation is evidenced by both the shift in the peak envelope and the large drop in peak area.

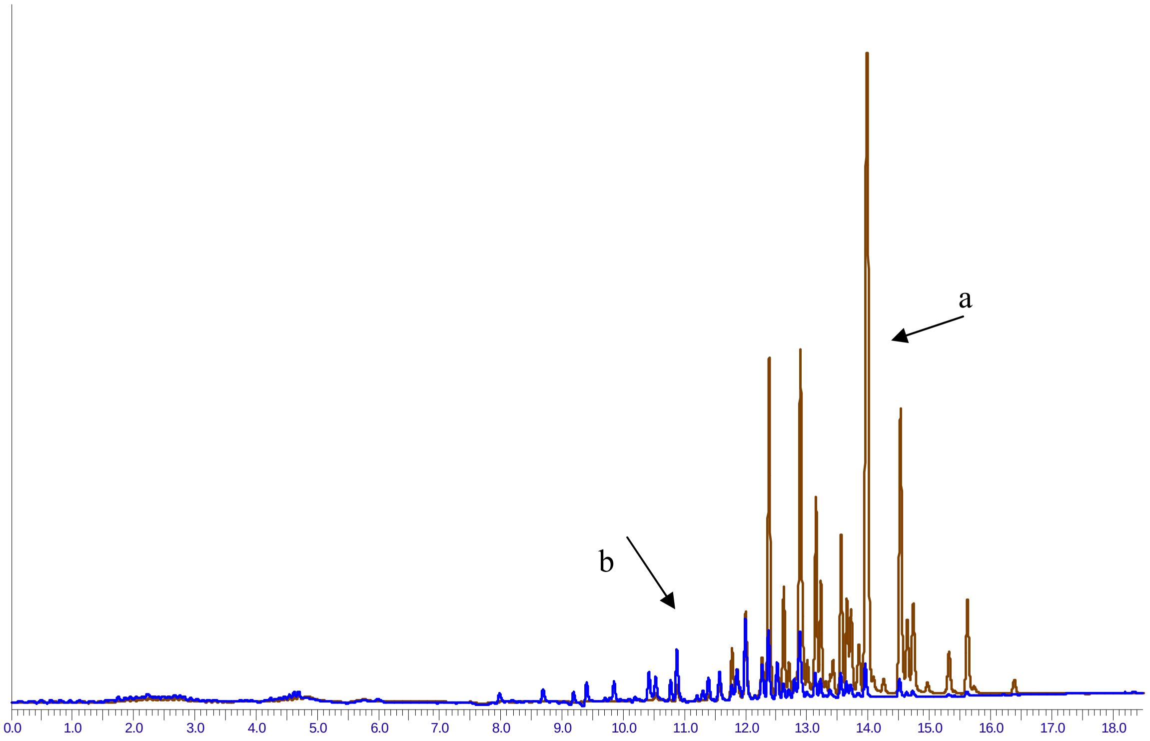


Figure 5-90. Chromatograms for the paste sample obtained from the Badge NM6810-36-1wk sample site, at the original PCB concentration (226 mg/kg) and after 3 days of treatment with Mg particles (10 wt %) and acidified ethanol. Data for (a) NM 6810-36 1wk (original) 226 mg/kg = **brown** and (b) after 3 days reacting with Mg metal 22 mg/kg = **blue.**

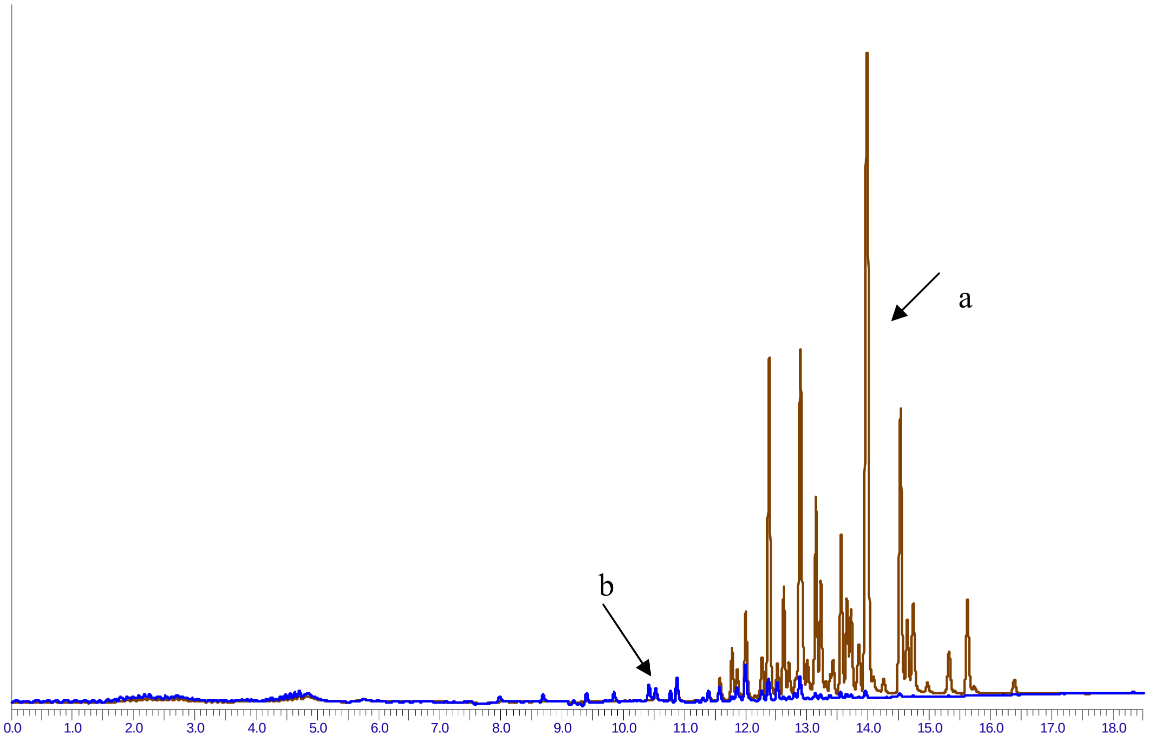


Figure 5-91. Chromatograms of the original paste and after 7 days of treatment. Data showing (a) NM 6810-36 1wk (original) 226 mg/kg = **brown** and (b) after 7 days reacting with Mg metal 11 mg/kg = **blue.**

A bar graph showing the change in PCB concentration upon treatment for the samples that were listed in Table 5-6 is shown in Figure 5-92.

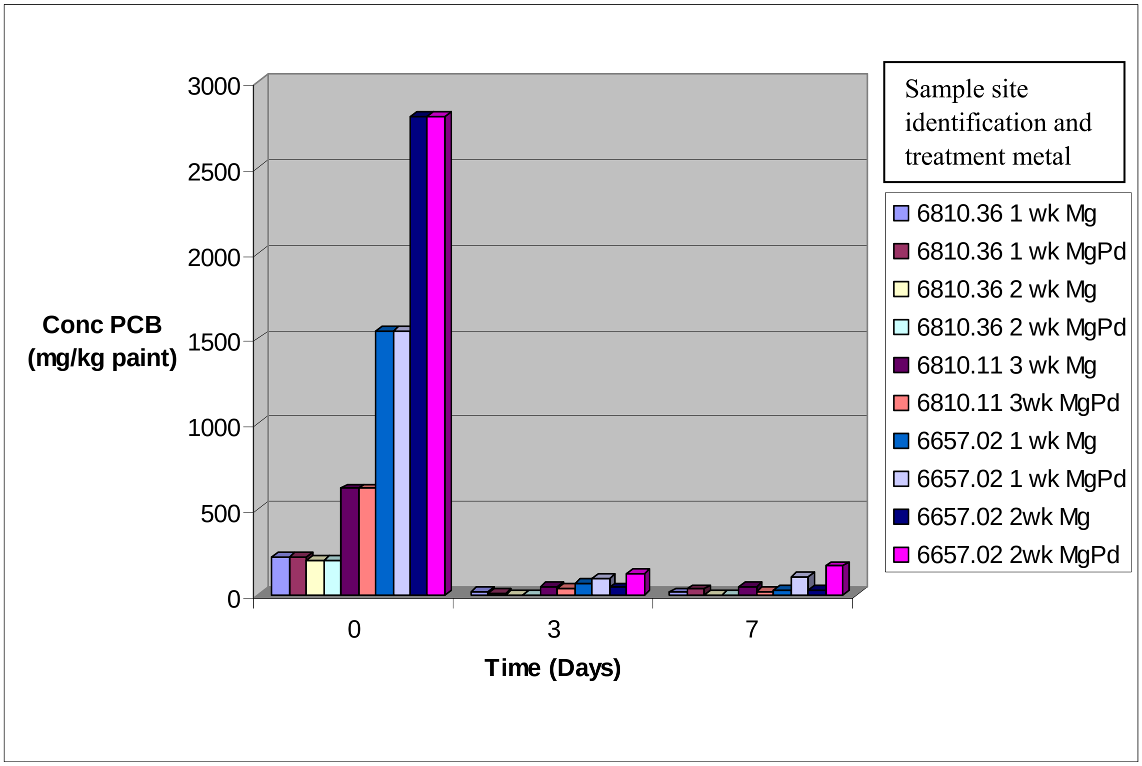


Figure 5-92. Results of PCB degradation by Mg and Mg/Pd particles added to the non-metal Badger paste samples.

In order to determine if the addition of a higher concentration of Mg to the paste would enhance the rate of PCB degradation, studies were conducted where 15% (wt.) of Mg particles were added to the nonmetal containing paste samples. In addition, 5% (wt%) was added to Badger paste samples that contained Mg/Pd particles but that had not dechlorinated the PDBs to below 50 mg/kg because of the high starting concentrations. The reason for adding Mg to the paste that already contained Mg/Pd is that the rate of the PCB degradation reaction had decreased significantly in these samples after they were removed from the Badger test sites. The rates were enhanced somewhat by adding acidified ethanol, but they only reached a level of one-fourth that of fresh Mg/Pd paste. The reason for this inability to reactivate the aged Mg/Pd particles to original rate levels is still being studied but present evidence suggests that a hard layer of impervious MgO is formed on the outside of the particle. The use of acidified ethanol after the MgO layer is formed is only effective at penetrating a small fraction of the layer. However, when acidified ethanol is used during the initial preparation of the BTS paste, the formation of the impervious layer of MgO appears to be minimized to a level where reaction rates remain high.

The data for this phase of the study is presented in Table 5-7. As can be seen from the data, this study was conducted over a 21 day period. The data show that the addition of greater than 10% Mg (i.e. 15%) to the nonmetal paste increases the rate of PCB degradation. In addition, the addition of 5% Mg (wt %) to the paste that originally contained 10% (wt %) of Mg/Pd caused the rate of the PCB degradation to increase. A bar graph representation of the data presented in Table 5-7 is given in Figure 5-93.

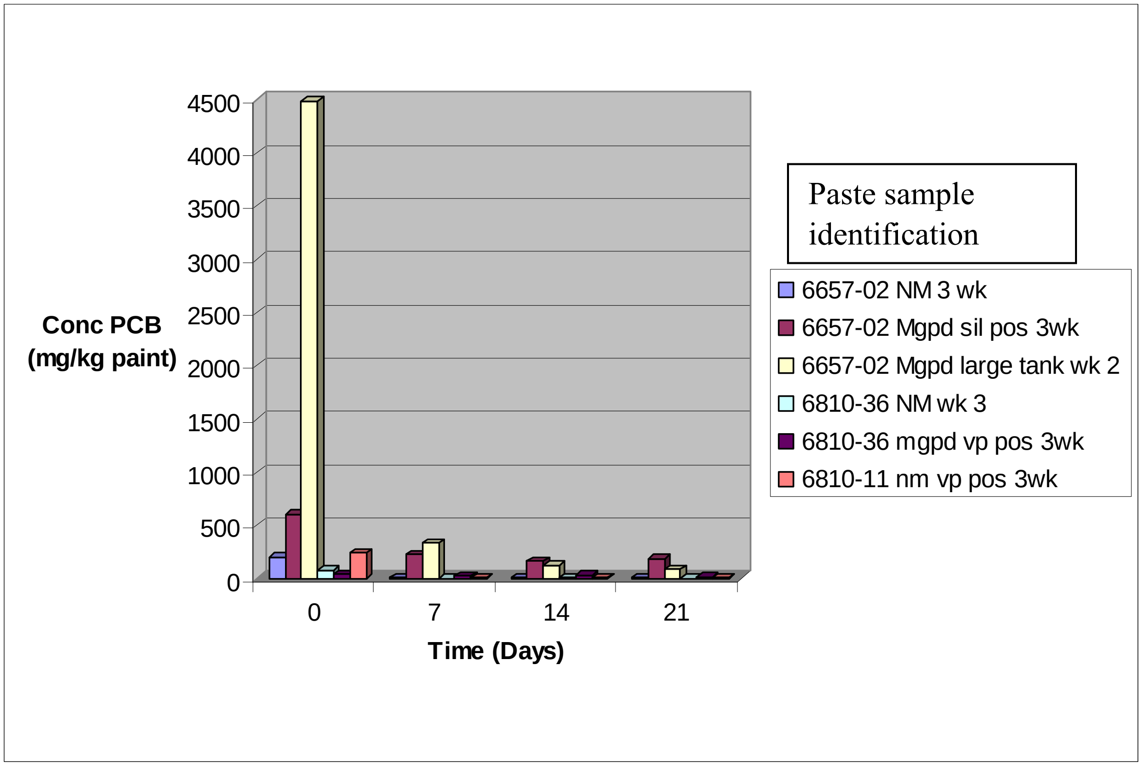


Figure 5-93 PCB degradation in BTS paste after the addition of Mg (10%wt) to the nonmetal samples (NM) and 5% Mg to the samples that already contained 10% Mg/Pd.

Solvent Evaporation rate

A sample of paste was placed on a metal surface having a surface area of 54.19 cm^2 and was observed over a period of 24 hours with no cover over the surface of the paste. Its change in mass over time correlates to the solvent evaporation rate. Figure 5-94 displays this rate. The data show that more than 60% of the solvent was lost from the paste within 24 hours. This experiment clearly demonstrates the need to seal the paste after it is applied to painted surfaces.

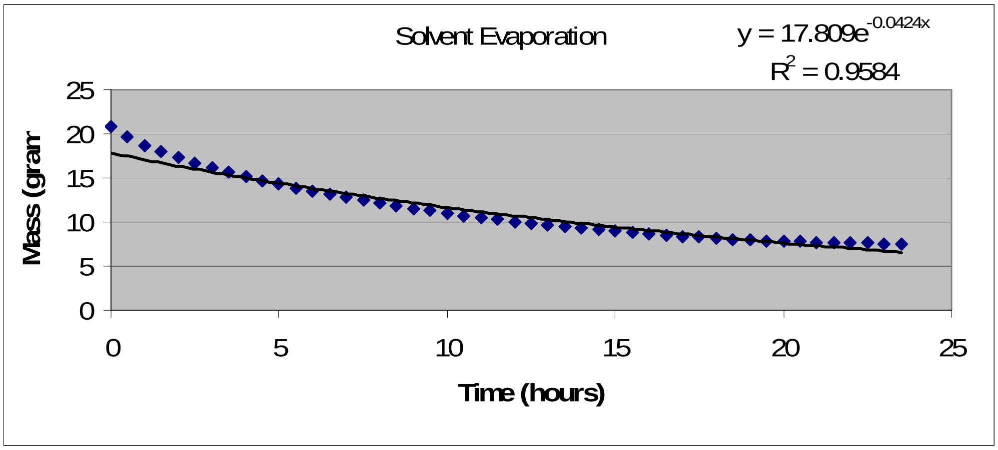
TABLE 5-7
PCB DEGRADATION IN BTS PASTE AFTER THE ADDITION OF Mg TO THE NON METAL SAMPLES AND 5% Mg TO THE SAMPLES THAT ALREADY CONTAINED 10% Mg/Pd

Sample Name	Orig. Conc. (mg/kg)	PCB Concentration (mg/kg)					
		7 day	average	14 day	average	21 day	average
6657-02 NM 3 wk	199	NS	2	3	4	2	2
		2		4		2	
6657-02 MgPd sil pos 3wk	605	245	219	195	194	126	150
		193		193		175	
6657-02 MgPd lrg tank wk 2	4490	541	331	138	121	92	83
		122		104		74	
6810-36 NM wk 3	74	1	1	2	2	1	1
		NS		1		1	
6810-36 MgPd VP pos 3wk	34	14	20	36	27	15	15
		26		19		16	
6810-11 NM VP pos 3wk	239	3	4	3	2	1	2
		6		1		3	

Notes:

mg/kg- milligram per kilogram
Mg - magnesium
Mg/Pd - magnesium/palladium
NS - no sample
NM - no metal (inactive paste)
VP - vinyl polymer sealant

Figure 5-94 Loss of solvent (g) over time (hr)



Optimal Thickness Test

The following study tested the thickness of paste needed to remediate a painted surface over a period of 14 days. PCB congener 151 was added to commercial paint which was painted on a metal surface and treated with paste of varying thickness. A glass cover was placed over the paste (but not touching it) to minimize loss of solvent. The original PCB concentration of the contaminated paint was 57mg/kg. Table 5-8 displays the percentages of PCBs removed for each thickness over 14 days.

The sample with a 0.1268" layer of BTS paste applied on the contaminated paint surface removed 82.2% of the PCBs in the paint by day 3. In comparison, the thicker layer of paste (0.6340 inch) removed 84.1% of the PCBs from the paint by day 3. There is not a very significant improvement in extraction amount or rate between the thinner and thicker layers of paste despite the five-fold increase in thickness. However, the thicker layers performed better over longer periods of time. This is probably due to a greater loss of solvent from the thinner layers or less build up in the concentration of PCBs in the paste for the thicker layers.

TABLE 5-8
PERCENT REMOVAL OF PCBs DEPENDENT ON BOTH THE AMOUNT OF TIME EXPOSED TO CONTAMINATED PAINT (57 Mg/Kg), AND THICKNESS OF PASTE USED

Day	**Thickness of Paste (inches)**				
	0.1268	0.2536	0.3804	0.5072	0.634
	Percent Removal				
3	82%	82%	87%	89%	84%
7	78%	71%	91%	86%	85%
14	70%	81%	88%	85%	90%

6 PERFORMANCE ASSESSMENT

The section provides a summary of all data analysis conducted in support of the assessment of performance objectives.

6.1 Distribution and Adherence of the BTS

One of the qualitative performance objectives is that the BTS applicator is able to evenly distribute the paste on the surface to be treated. The metric was evaluated by assessing the adherence of a 0.25 to 0.5 inch layer of the BTS to an object over the time period of exposure to treated surfaces. BTS paste was applied to surfaces to be treated using two types of application methods and its thickness estimated. The paste was visually inspected periodically (every two to 5 days for the first week and then weekly for up to a month) to determine if it was able to adhere to the surfaces (both vertical and horizontal treated surfaces) by inspecting for leaks, sections of paste pulling away from the surface or bubbling up off the surface.

This objective was met. BTS was applied using a spray applicator and hand trowel application method. In some cases the paste did not adhere well to the vertical painted surfaces but this was due to adhesion issues with the paint (old and friable paint on concrete surface) and not due to the pastes ability to adhere to the surface. Applications were done using the hand trowel method at Badger due to the paste thickening in the cold. At the VIB, where both the spray applicator method and hand trowel application were used, the spray applicator resulted in a much thinner layer of BTS being applied compared with the trowel application which did affect the treatment ability of the BTS so preference was given to the hand application method. At the VIB, the paste adhered to vertical and horizontal surfaces without any problems in a consistency that could be spray applied or spread with a trowel by hand. It was determined that it was faster to apply the paste with the trowel in a layer of the desired thickness rather than using a spray applicator which required multiple passes to get the desired thickness.

6.2 Adherence of Sealants

The metric was evaluated by assessing the adherence of the sealant to the BTS, the ability to apply the sealant evenly over the surface of the paste, and its ability to dry to a non-tacky, non-porous layer that reduced volatilization of BTS solvent. Two sealants were tested, a vinyl polymer (VP) truck bed liner and a silicone-based roof sealant (Sil). The sealant and underlying BTS paste were inspected every one to two days, depending on location, for the first five days and samples were collected of the paste and sealant weekly for up to a month after application. The sealants ability to adhere to the BTS and to reduce volatilization of the ethanol in the BTS was evaluated by visual inspection (looking for leaks around the edges of the sealant, looking for bubbles or drooping of sealant away from BTS surface) and manual inspection by pressing lightly on the sealed surface to test the fluidity of the paste.

This objective was met using both sealants but some difficulties were encountered with old friable paint and cold temperatures. Both sealants were able to provide an effective seal to minimize ethanol evaporation from the BTS paste for the three weeks of application. In the cases where there were issues with the condition of the paint (old and friable paint on concrete) there were issues with both the sealant and paste sticking to the surface and the seal failed. However, this had to do with the paint itself being unable to adhere to the surface and not the sealant. In addition, the cold temperatures during the Badger deployment did affect the ability of the sealant to remain flexible and some cracking of the sealant did occur. If possible, it would be preferable to apply BTS in above freezing conditions to avoid the risk of sealant failure. If applications must be done in below freezing temperatures, additional care must be taken to inspect and repair cracks in the sealant as they appear.

6.3 Ease of implementation

The ease of use of this technology was evaluated based on our experience in the field. The implementability was evaluated by the qualitative assessment of the ease with which the operator was trained to handle and apply both the paste and the sealant in the various field situations.

This objective was met with respect to both the ease of handling and applying both the paste and sealant on the various surfaces and locations that were treated. The equipment required for the manufacturing of BTS on Site using the acidified ethanol and Mg would be readily available through the paint industry. The application equipment used to apply BTS and the sealants was all readily available through local hardware suppliers or the paint industry. The procedures used to manufacture the BTS were well established procedures and were simple enough to be conducted by field technicians with training in the manufacturing techniques. The procedures used to apply the BTS were standard and well established procedures and the procedures were simple enough to be conducted by field technicians with training in basic application techniques and handling techniques of the BTS and sealants.

6.4 Reduction in PCB Concentrations in Treated Paint to Less Than 50 mg/kg

A key performance objective is the reduction of PCB concentrations in the treated material to less than 50 mg/kg. The metric was evaluated by collecting samples of the paint pre-BTS application and then weekly for up to a month for analysis of PCB concentrations. This was a destructive analysis and was done on one section of the test area on a weekly basis until all sections had been analyzed.

This objective was partially met. One application of paste was effective in achieving this target after only one week of treatment in all cases where the starting concentration in the paint was less than approximately 500 mg/kg, especially if the surface being treated was metal and not concrete. In cases where the starting concentrations in the paint were greater than 500 mg/kg, significant reductions (93%) in PCB concentrations were achieved but more than one application of paste is necessary to reduce concentrations below 50 mg/kg.

The starting concentrations in the VIB were low and the BTS was able to achieve the target of 50 mg/kg for the treated surfaces. PCB concentrations in paint in building 6810-11 at badger were between 1,800 mg/kg and 3,880 mg/kg in the pre-demonstration samples. Post-treatment concentrations (1 to 3 weeks of treatment) in paint were between 96 mg/kg and 200 mg/kg (Table D-2 and Figure 5-75). PCB concentrations in paint in building 6810-36 were between ~ 400 mg/kg and ~900 mg/kg prior to treatment and post treatment concentrations in paint were between 96 mg/kg and 200 mg/kg (Table D-4 and Figure 5-77). The target of 50 mg/kg in the paint was not achieved with starting concentrations of PCBs in the paint of greater than about 400 to 500 mg/kg. It is believed that in part this was because the PCBs extracted into the paste may have exceeded the degradation capabilities of the Mg/Pd in the paste for the Mg/Pd BTS and then the paste became concentrated with PCBs. For the NM BTS the paste became concentrated with PCBs before complete extraction of PCBs from the paint could occur. The PCB-saturated paste was no longer able to further extract PCBs from the paint. The performance of the BTS paste was also negatively impacted by the conditions of the paint at building 6810-36 and 6810-11. The paint was friable and peeling which prevented the BTS from achieving a good adherence to the wall and prevented the sealant from providing and effective seal of the paste which resulted in the loss of ethanol from the paste. In addition, the paste was applied to a porous surface that reduced the amount of ethanol available in the paste (loss of ethanol to the concrete, magnified by the amount of concrete that was treated that was exposed without any paint to seal it).

The starting concentrations in the paint for building 6810-36 were similar to those in the Staging Area and all the treated paint in the Staging Area was treated to below 50 mg/kg (Table D-7). In the Staging Area, the BTS was applied to painted metal and the paint was in better conditions that that on the painted concrete in building 6810-36. For building 6657-02 the starting concentrations in the paint were very high (>20,000 mg/kg) and it wasn't possible to get the concentrations below 50 mg/kg due to the paste PCB concentration as discussed above. However, like the Staging Area, the BTS was applied to painted metal and the reductions in concentrations were significant after one week of BTS exposure (reductions of more than 13,000 mg/kg; Table D-6).

6.5 Reduction in PCB Concentrations in BTS Paste to Less Than 50 mg/kg

The reduction of PCB concentrations in the paste to less than 50 mg/kg is another key performance objective. The metric was evaluated by collecting samples of the paste weekly for up to a month for analysis of PCB concentrations. This was a destructive analysis that was done on one section of the test area on a weekly basis until all sections had been analyzed. For BTS that did not contain the activated metals for degradation (non-active paste) a subset of the paste sample was analyzed for PCBs and then the activated metal and, if necessary, additional ethanol, were added to the non-active paste to promote degradation (post-application activated paste). Samples of the post-application activated paste were then analyzed for PCB concentrations.

This objective was partially met. For the active paste (metal in the paste) if the starting paint concentrations were below roughly 2,500 mg/kg then the concentrations in the paste were less than 50 mg/kg with the exception of the 3 weeks samples from Badger Building 6810-11 where concentrations were still slightly above the 50 mg/kg target (60 mg/kg; Table D-2). If the pre-treatment paint concentrations were very high (> 20,000 mg/kg) then the active metal paste was not able to degrade all of the PCBs in the paste to below 50 mg/kg although some dechlorination did occur in the paste. If very high concentrations are present in the paint this yields a situation where more PCBs are present than can be handled by the Mg/Pd available in the paste or that can degraded sufficiently in the time period that solvent remains in the paste to facilitate treatment. As mentioned above, the paste becomes concentrated with PCBs. Even when Mg/Pd and additional ethanol was added in the laboratory to the active paste that had been exposed to the very high starting concentrations it was not possible to get the concentrations in the paste to below 50 mg/kg after 21 days (Table 5-7). The rates of dechlorination were enhanced somewhat by the addition of acidified ethanol, but they only reached a level of one-fourth that of fresh Mg/Pd paste. The reason for this inability to reactivate the aged Mg/Pd particles to original rate levels is still being studied but present evidence suggests that a hard layer of impervious magnesium oxide (MgO) is formed on the outside of the particle. The use of acidified ethanol after the MgO layer is formed is only effective at penetrating a small fraction of the layer. However, when acidified ethanol is used during the initial preparation of the BTS paste, the formation of the impervious layer of MgO appears to be minimized to a level where reaction rates remain high.

For the non metal paste, which was activated in the lab after removal from the field by the addition of ethanol and the active metal (Mg and acid or Mg/Pd), the concentrations were reduced to below 50 mg/kg for all samples using either the acidified ethanol and Mg and/or ethanol and the Mg/Pd.

6.6 Impact to Paint Adherence

One of the secondary objectives was to evaluate the impact to the paint adherence after exposure to BTS. The metric was evaluated by visually assessing the condition of the paint after BTS application as well as by using the field test for pull-off strength of coatings (ASTM D3359-02).

When the paint at the Badger test sites was subjected to the ASTM D3359-02 procedure prior to application of BTS, the paint at all the test sites failed. Thus, the effect of the BTS on the integrity of the painted surfaces at Badger could not be assessed.

There were difficulties in using ASTM D3359-02 at the VIB on post-treatment samples due to preparation of the surface prior to testing and ASTM D4541-02 was also used to evaluate the paint adherence for the VIB panels.

This objective was not met in that the BTS negatively affected the adherence and strength of at least the surface layer of paint.

7 COST ASSESSMENT

This section presents the results of a cost assessment to implement remediation of PCB containing painted structures using BTS. Cost data collected during the technology demonstration were evaluated as described in the following sections. Section 7.1 describes a cost model that was developed for the application of BTS with a comparison to disposal of structures without treatment; Section 7.2 presents an assessment of the cost drivers for the application of the technology; and Section 7.3 presents the results of an analysis of the cost model.

7.1 Cost Model

A cost model was developed to assist remediation professionals in understanding costs associated with the BTS technology. The cost model identified the major cost elements required to implement the BTS technology at typical structures. A summary of the actual costs for pilot-scale implementation of the BTS technology at Badger and the VIB is presented in Table 7-1.

The major cost categories tracked during the field demonstrations at Badger and the VIB were:

Capital Costs –

Design and Planning – This cost element includes activities required to scope the project, such as site visits required to evaluate size and configuration of the surface coating which may impact the application process of the solvent paste, how many applications of solvent paste may be required, and whether a single-phase treatment (active solvent paste containing catalyst) or two-phase (inactive solvent paste with catalyst added later) treatment may be more cost effective.

Treatability Testing - This cost element includes the labor required to collect and analyze samples of PCB-containing material samples before and after bench-scale treatment. This laboratory evaluation will inform the final design of the application of the solvent paste, number of probable applications required, and whether to use a single-phase or two-phase treatment. This cost element provides scale-up information required for the manufacture of the catalyst and the solvent paste.

Manufacturing of the Catalyst System and BTS Reagent for Field Testing - Based on scale-up information developed during treatability testing, the volume and consistency of solvent paste required and whether it will be applied as an inactive or active paste have been determined. The amount of catalyst required to treat the expected PCB concentrations has also been determined. Costs include raw materials for the solvent paste and catalyst and manufacturing labor.

TABLE 7-1
ACTUAL COSTS FOR BTS TECHNOLOGY DEM/VAL

Cost Element	Data Tracked During the Demonstration/Per Site	Cost	
Capital Costs			
Design & Planning	- Personnel required and associated labor	Labor	$1,433
		Other Costs	$270
Treatability Testing	- Personnel required and associated labor for treatability testing activities	Labor	$27,300
		Other Costs	$12,000
Manufacturing of the Catalyst System and BTS Reagent for Field Testing	- Personnel required and associated labor for BTS application activities - Mobilization costs - Costs for BTS and applicaiton equipment	Labor	$8,568
		Other Costs	$7,000
Application of BTS to DoD Test Structures/Materials	- Personnel required and associated labor for BTS application activities - Mobilization costs - Costs for BTS and application equipment	Labor	$7,600
		Other Costs	$9,300
Removal of BTS to DoD Test Structures/Materials	- Personnel required and associated labor for BTS removal activities - Demobilization costs	Labor	$2,400
		Other Costs	$1,200
Performance Monitoring			
Baseline Characterization	- Personnel required and associated labor - Mobilization costs - Supplies and equipment for sampling - Sample shipment and laboratory analytical costs - Labor associated with data reporting	Labor	$7,400
		Other Costs	$3,900
Performance Monitoring	- Personnel required and associated labor - Mobilization costs - Supplies and equipment for sampling - Sample shipment and laboratory analytical costs - Labor associated with data reporting	Labor	$18,800
		Other Costs	$17,800

Application/Removal of BTS to DoD Test Structures /Materials - In this cost element, the catalyst, solvent paste, and sealant are mobilized to the site, along with the appropriate application/removal and waste handling equipment and demobilization costs. Costs include labor hours to apply/remove the solvent paste and sealant.

Performance Monitoring Costs

Baseline Characterization - This cost element addresses activities and equipment required to collect and analyze samples of coating material to assess and report the presence and concentration of PCBs prior to any treatment. Information developed during this process informs initial design for the project.

Performance Monitoring - This cost element addresses activities and equipment required to collect and analyze samples of coating material to assess and report the presence and concentration of PCBs during in situ treatment. Results inform the number of applications of solvent paste which may be required.

The cost model that is shown in Table 7-1 were based on the actual costs that were tracked in the Demonstrations. More weight was given to the costs from the Badger Demonstration since this demonstration occurred at a more remote facility (VIB Demonstration was done at a facility located very near to the NASA office and laboratory).

The cost model was then applied for two template sites requiring disposal or demolition of buildings or structures covered in paint containing 250 mg/kg PCBs. Two model structures were used; the first a painted concrete (porous surface) building that was to be demolished; and the second a painted metal tank (non-porous surface) that required disposal. For ease of comparison, both structures were assumed to have a painted surface area of 8000 square feet.

The starting PCB concentration of 250 mg/kg was chosen because during this demonstration, this concentration was been shown to be able to be treated in place with one application of an active paste. It was assumed that it will require one week of exposure of the BTS to the painted surface to completely extract the PCBs from the paint. The BTS would then be transferred to drums or roll-off bins and allowed to sit for another week to allow for complete degradation of the PCBs in the BTS paste prior to disposal as non-hazardous waste.

A set of eight pre-treatment paint and concrete samples are assumed to be required to characterize the site (8,000 ft^2 to be treated) prior to treatment and eight post treatment samples of the paint, concrete and paste to evaluate the performance of the treatment.

Cost estimates for the BTS technology were prepared for both painted concrete and painted metal because of the potential differences in how these materials may need to be handled.

Based on the EPA's Polychlorinated Biphenyl (PCB) Site Revitalization Guidance Under the Toxic Substances Control Act (TSCA) (November 2005), porous and non-porous surfaces are treated differently. If disposal or reuse is considered for a building where PCB-containing paint is used on a porous surface, the paint must be removed and treated as PCB bulk product waste. The porous material must then be tested (i.e., concrete core samples) and, if PCBs have migrated into the porous surface, then the porous materials must be handled as PCB remediation waste and disposed of in a TSCA-approved landfill. For non-porous surfaces, the paint can be removed and treated as a PCB bulk product waste and if the non-porous surface is shown to be clean, the item can be reused, recycled or disposed of as non-hazardous waste.

Costs have been organized into three major categories: Start-up/Shut Down, Operations and Maintenance, and Waste Handling. Start-up/Shut Down includes materials and labor costs associated with mobilization and demobilization, permitting, BTS operator training, site preparation, sample collection, and treatability testing. Operations and Maintenance includes materials and labor costs associated with BTS preparation, application, and analysis, demolition or paint removal, equipment decontamination and the testing/analysis of waste streams and metal surfaces, as appropriate. Waste Handling includes materials and labor costs associated with transportation and disposal of hazardous and non-hazardous demolition debris and waste, decontamination/wash fluids, BTS waste and metal recycling.

7.2 Cost Drivers

The costs to implement the BTS technology for treatment of PCBs in paint will vary significantly from site to site. The key costs drivers are listed below, along with a brief discussion of their impact on cost.

Nature and Extent of Contamination

- **Concentrations of PCBs in paint** – The higher the PCB concentrations are in the painted surfaces, the more likely it will be that multiple applications of BTS may be required to achieved removal and/or degradation of the PCBs to below 50 mg/kg.

- **Conditions of the surface to be treated** – If the paint to be treated is old, weathered, damaged and/or friable, proper adherence of the BTS and sealant to the surface may be difficult to achieve. This may make it difficult to achieved removal of PCBs to below 50 mg/kg with a single application of the BTS.

- **Geometry and simplicity of the surface to be treated –** If the surface of the structure to be treated is very convoluted, with many grooves or hard-to-access surfaces (pipes strapped to walls that are painted with PCB paint) this will increase the effort required to properly apply the BTS to the surfaces to be treated.

- **Additional constituents which require treatment** – For example, at Badger, materials with the potential of containing residue of explosive or energetic compounds must be incinerated prior to disposal. However, the presence of PCBs in concentrations > 50 mg/kg require incineration in an oven operating in conformance with 40 CFR 761.72(a) and if the material must be cut into manageable sizes, a special permit is required to perform the cutting operations. These conditions may make BTS application more favorable.

Environmental Conditions

- **Temperature and Weather Conditions –** Although the BTS has been shown to degrade PCBs at cold temperatures (4ºC) the rate is slower and there may be difficulties with the performance of the sealants in cold temperatures that could affect the number of times sealant or BTS may need to be applied.

Available Infrastructure & Site Access

- **Available infrastructure** - The availability of existing infrastructure (e.g., on site or nearby landfills, storage buildings, and utilities) can reduce the cost of technology implementation.
- **Site Access** - Sites having limited access for equipment and personnel (e.g., difficult terrain, obstructions, or other complicating factors such as explosives storage) may incur higher costs when implementing the technology.
- **Site Location –** If the site is located in a remote area, there will be additional mobilization costs. However, there will also be a significant increase in costs for transport to and disposal of hazardous waste in a properly permitted landfill, which may make the cost benefit of treating a structure with BTS more favorable. The costs for transportation and disposal of PCB impacted waste could be much more significant if the distances are greater or the site is very remote or difficult to access such as on an island, or in the arctic.

7.3 Comparison of Cost Analysis

BTS, when deployed at the perceived end of a facility's lifecycle, may represent an additional, rather than an alternative, cost in that facility's final disposition. As such, an extremely simplified cost model has been applied to the costs associated with alternative facility disposal methods and a BTS treatment scenario. For the purposes of this costs analysis, only those costs and environmental liability due to PCB concentrations in excess of the regulatory limit of 50mg/kg are being considered. However, given that BTS can extend structure reuse by removing PCBs without damaging the structure may also be considered an alternative to final disposition.

The most probable scenarios associated with the application of BTS are as follows:

1. Baseline conditions (untreated facility). Environmental liability due to PCB concentrations is retained.
    - Demolition of facility or structure.
    - Steel and concrete is not recycled due to PCB concentrations.
    - Demolition debris is landfilled as hazardous waste.
2. BTS is applied to the facility's coatings and other materials as appropriate. Environmental liability due to PCB concentrations is eliminated. Two scenarios may be available: Structure reuse or demolition.
    - Structure reuse: Life cycle of building is extended, reducing unit capital cost associated with the structure.
    - Demolition of facility or structure.
        - Steel and concrete may be recycled, providing an opportunity to defray costs associated with PCB treatment and/or demolition.
        - Demolition debris is landfilled as non-hazardous waste.

The success of BTS was evaluated by comparing the added cost of BTS treatment to the reduced costs associated with the landfilling of demolition debris, the potential revenue generation from steel and concrete recycling, and perhaps the extended life/re-use of the facility which would not otherwise be cost-effective.

Using the template site conditions described above, estimates of costs were developed for each of the following alternatives:

- Concrete structure demolition, untreated and disposed of in a TSCA landfill;
- Concrete structure demolition, treated prior to demolition with BTS, disposed of non-hazardous landfill and concrete recycled;
- Concrete structure treated with BTS and reused. This alternative addresses those structures where PCB concentration in paints or other materials drive the requirement for demolition;
- Metal tank, untreated and disposed of in a TSCA landfill;

- Metal tank, conventional paint removal using sandblasting, sandblast waste sent to TSCA landfill and metal tank recycled; and

- Metal tank, treated with BTS and then recycled.

Based on the cost models shown in Table 7-2 and 7-3, it appears that it would be cost effective to apply BTS to a concrete building prior to demolition compared with the costs of demolishing the building and disposing of the waste in a TSCA landfill. Demolition and disposal costs of the untreated concrete building are estimated to be approximately $200,000, while the cost of treating the painted concrete with BTS, followed by demolishing the building and recycling the concrete is estimated to be approximately $180.000. In the third scenario, BTS treatment and building reuse rather than demolition, estimated costs are even lower at approximately $150,000.

In contrast, if the structure to be treated is a metal tank, then it does not seem to be cost effective to address the PCB-containing paint by either conventional sandblasting or BTS treatment in order to recycle the tank. Straight disposal of the untreated tank is estimated to cost approximately $25,000 versus removal of the paint with sandblasting and recycling the tank which is estimated to cost $105,000 and treatment with BTS and recycling the tank which is estimated to cost $140,000. It must be noted that both untreated tank debris and sandblast waste will retain PCBs and associated environmental liability issues, while there are no long term liability issues if the tank is treated with BTS before disposal.

As mentioned above, the costs for transportation and disposal of PCB impacted waste could be much more significant if the distances are greater or the site is very remote or difficult to access such as on an island, or in the arctic.

All the scenarios evaluated in the costs model have assumed the treatment of PCB-containing materials down to the Federal regulatory limit of 50 mg/kg. However, some states or countries may have stricter regulations with respect to the disposal of PCB impacted wastes. According to http://www.ehso.com/EHSO_PCB.htm (Environmental Health and Safety online) the Federal regulation for non-PCB waste is 50 mg/kg, but some state regulations are 5 mg/kg. For the situation with the Navy properties in Japan, the regulations are very strict and any waste with PCB concentrations >0.5 mg/kg cannot be disposed of in Japan. This requires that the US Navy transport all materials impacted by PCBs to another location for disposal greatly increasing the costs. Thus, a more restrictive regulatory limit will tend to increase the cost effectiveness of BTS treatment of facilities over straight demolition and disposal. BTS can reduce PCB concentrations down to non-detect (detection limit of 1 mg/kg) but the lower the target treatment concentration is the greater the potential exists for the need for additional applications of BTS, and therefore greater costs, to achieve these targets.

TABLE 7-2
COMPARISON OF COST SCENARIOS - POROUS

		Painted Porous Surface (Concrete Building)								
		Concrete Building Untreated Demolished, Sent for Disposal Directly to TSCA Landfill			Concrete Building Treated with BTS - Demolished, Sent for Disposal in Non-Haz Landfill and Concrete Recycled			Concrete Building Treated with BTS and Reused		
	Unit	Quantity	Unit Price	Cost	Quantity	Unit Price	Cost	Quantity	Unit Price	Cost
Start-Up/Shut Down										
Mobilization	LS	1	$ 4,000	$ 4,000	1	$ 5,500	$ 5,500	1	$ 1,500	$ 1,500
Sample Collection and Treatability Tests	LS	0		$ -	1	$ 12,500	$ 12,500	1	$ 12,500	$ 12,500
Permitting, Approvals	LS	1	$ 3,200	$ 3,200	1	$ 4,000	$ 4,000	1	$ 4,000	$ 4,000
Site Preparation (Disconnect/Abandon Utilities, Dust and Erosion Control)	LS	1	$ 7,400	$ 7,400	1	$ 5,500	$ 5,500	0		$ -
Train BTS Operators	HR	0	$ 255	$ -	8	$ 255	$ 2,040	8	$ 255	$ 2,040
Demobilization	LS	1	$ 4,000	$ 4,000	1	$ 5,500	$ 5,500	1	$ 1,500	$ 1,500
Sub-Total				***$ 18,600***			***$ 35,040***			***$ 21,540***
Operations and Maintenance										
Labor – BTS Application	HR	0	$ 55	$ -	20	$ 55	$ 1,100	20	$ 55	$ 1,100
Labor - Demolition and Segregation of Debris	LS	1	$ 6,200	$ 6,200	1	$ 6,200	$ 6,200	1	$ 6,200	$ 6,200
Consumables and Supplies (PCB-impacted dust mitigation etc)	LS	1	$ 1,400	$ 1,400	0		$ -	0		$ -
Lab Analysis of Progress of PCB Degradation	Sample	0	$ 65	$ -	8	$ 65	$ 520	8	$ 65	$ 520
BTS Materials Including Sealant	SF	0	$ 15	$ -	8000	$ 15	$ 118,960	8000	$ 15	$ 118,960
Consumables and Supplies (Application of BTS)	LS	0	$ 1,500	$ -	1	$ 1,500	$ 1,500	1	$ 1,500	$ 1,500
Equipment Decontamination	LS	1	$ 1,000	$ 1,000	0		$ -	0		$ -
Test/Analysis of Waste Streams	Sample	0		$ -	8	$ 100	$ 800	8	$ 100	$ 800
Sub-Total				***$ 8,600***			***$ 129,080***			***$ 129,080***
Waste Handling										
Transportation of Demolition Debris to Non-Haz Landfill	TON	118	$ 22	$ 2,596	118	$ 22	$ 2,596	0	$ 22	$ -
Disposal of Demolition Waste in Non-Haz Landfill	TON	118	$ 80	$ 9,440	118	$ 80	$ 9,440	0	$ 80	$ -
Delivery of Bins and Transportation of Demolition Debris to TSCA Landfill	TON	182	$ 530	$ 96,460	0		$ -	0		$ -
Disposal of Demolition Waste in TSCA Landfill	TON	182	$ 350	$ 63,700	0		$ -	0		$ -
Transportation of Decontamination/Wash Fluid (non-haz)	GAL	100	$ 5	$ 475	100	$ 5	$ 500	50	$ 5	$ 250
Disposal of Decontamination/Wash Fluid (non-haz)	GAL	100	$ 1	$ 100	100	$ 1	$ 100	50	$ 1	$ 50
Transportation of Concrete for Recycling (~60% of Material)	TON	0		$ -	182	$ 22	$ 4,004	0	$ 22	$ -
Transportation of BTS Waste (non-Haz)	TON	0		$ -	0.5	$ 22	$ 11	0.5	$ 22	$ 11
Disposal of BTS Waste in Non-Haz Landfill	TON	0		$ -	0.5	$ 80	$ 40	0.5	$ 80	$ 40
Sub-Total				***$ 172,771***			***$ 16,691***			***$ 351***
TOTAL				**$ 199,971**			**$ 180,811**			**$ 150,971**

Notes

Assume starting concentration in paint is 250 mg/kg

Assumes treatment of 8000 square feet surface area for both building and tank

Large variability in transportation and disposal costs depending on distance to landfill and the concentrations in the waste but costs for transportation and disposal to TSCA landfill is based on

Assumes that State/EPA regulations would require the porous substrate to be disposed as hazardous waste in TSCA-regulated landfill if painted surface has >50 mg/kg PCBs

Assume for painted concrete, 45 lbs of substrate per square foot of painted area or 44 sq ft per ton of substrate

LS - lump sum

SF - square foot

TABLE 7-3
COMPARISON OF COST SCENARIOS - METAL

		Painted Non-Porous Surface (Metal Tank)								
		Metal Tank Untreated (Paint Not Removed) and Disposed of in TSCA Landfill			Metal Tank, Paint Removed by Sandblasting and Tank Recycled			Metal Tank Treated with BTS and Recycled		
	Unit	Quantity	Unit Price	Cost	Quantity	Unit Price	Cost	Quantity	Unit Price	Cost
Start-Up/Shut Down										
Mobilization	LS	1	$ 1,500	$ 1,500	1	$ 1,500	$ 1,500	1	$ 1,500	$ 1,500
Sample Collection and Treatability Tests	LS	0		$ -	0		$ -	1	$ 9,500	$ 9,500
Permitting, Approvals	LS	1	$ 3,000	$ 3,000	1	$ 3,000	$ 3,000	1	$ 1,500	$ 1,500
Site Preparation (Disconnect/Abandon Utilities, Dust and Erosion Control)	LS	0	$ 500	$ -	1	$ 500	$ 500	1	$ 2,500	$ 2,500
Train BTS Operators	HR	0		$ -	0		$ -	8	$ 255	$ 2,040
Demobilization	LS	1	$ 1,500	$ 1,500	1	$ 1,500	$ 1,500	1	$ 5,500	$ 5,500
Sub-Total				***$ 6,000***			***$ 6,500***			***$ 22,540***
Operations and Maintenance										
Labor – BTS Application	HR	0	$ 55	$ -	0	$ 55	$ -	16	$ 55	$ 880
Labor - Demolition and Segregation of Debris	LS	10	$ 55	$ 550	-	-	-	-	-	-
Consumables and Supplies (PCB-impacted dust mitigation etc)	LS	0	$ 1,400	$ -	1	$ 2,000	$ 2,000	-	-	-
Lab Analysis of Progress of PCB Degradation	Sample	0		$ -	0		$ -	8	$ 65	$ 520
Sandblasting Costs (including labor)	SF	0	$ 8,000	$ -	8000	$ 10	$ 80,000	0	$ 8,000	$ -
BTS Materials Including Sealant	SF	0	$ 15	$ -	0	$ 15	$ -	8000	$ 15	$ 118,960
Consumables and Supplies (Application of BTS)	LS	0	$ 1,500	$ -	0	$ 1,500	$ -	1	$ 1,500	$ 1,500
Equipment Decontamination	LS	0	$ 500	$ -	1	$ 500	$ 500	0		$ -
Test/Analysis of Waste Streams	Sample	3	$ 100	$ 300	8	$ 100	$ 800	8	$ 100	$ 800
Test/Analysis of Metal Surface	Sample	0	$ 100	$ -	5	$ 100	$ 500	5	$ 100	$ 500
Sub-Total				***$ 850***			***$ 83,800***			***$ 123,160***
Waste Handling										
Transportation of Demolition Debris to Non-Haz Landfill	TON	0	$ 22	$ -	-	-	-	-	-	-
Disposal of Demolition Waste in Non-Haz Landfill	TON	0	$ 80	$ -	-	-	-	-	-	-
Delivery of Bins and Transportation of Demolition Debris to TSCA Landfill	TON	20	$ 530	$ 10,600	-	-	-	-	-	-
Disposal of Demolition Waste in TSCA Landfill	TON	20	$ 350	$ 7,000	-	-	-	-	-	-
Transportation of Decontamination/Wash Fluid (non-haz)	GAL	50	$ 5	$ 238	50	$ 5	$ 238	50	$ 5	$ 250
Disposal of Decontamination/Wash Fluid (non-haz)	GAL	50	$ 1	$ 50	50	$ 1	$ 50	50	$ 1	$ 50
Transportation of Concrete for Recycling (~60% of Material)	TON	-	-	-	-	-	-	-	-	-
Transportation of BTS Waste (non-Haz)	TON	0	$ 22	$ -	0	$ 22	$ -	0.5	$ 22	$ 11
Disposal of BTS Waste in Non-Haz Landfill	TON	0	$ 80	$ -	0	$ 80	$ -	0.5	$ 80	$ 40
Transportation of Sandblasting Waste to TSCA Landfill	TON	0	$ 530	$ -	24	$ 530	$ 12,720	0	$ 530	$ -
Disposal of Sandblasting Waste in TSCA Landfill	TON	0	$ 430	$ -	24	$ 350	$ 8,400	0	$ 350	$ -
Transportation of Metal Tank to Recycling	TON	0	$ 12	$ -	20	$ 12	$ 240	20	$ 12	$ 240
Recycled Steel Scrap	TON	-	-	-	20	$ (350)	$ (7,000)	20	$ (350)	$ (7,000)
Sub-Total				***$ 17,888***			***$ 14,648***			***$ (6,409)***
TOTAL				**$ 24,738**			**$ 104,948**			**$ 139,291**

Notes

Assume starting concentration in paint is 250 mg/kg

Assumes treatment of 8000 square feet surface area for both building and tank

Large variability in transportation and disposal costs depending on distance to landfill and the concentrations in the waste but costs for transportation and disposal to TSCA landfill is based on

Assumes that State/EPA regulations would require the porous substrate to be disposed as hazardous waste in TSCA-regulated landfill if painted surface has >50 mg/kg PCBs

Assume for painted concrete, 45 lbs of substrate per square foot of painted area or 44 sq ft per ton of substrate

LS - lump sum

SF - square foot

8 Implementation issues

This section provides information that will aid in the future implementation of the technology.

8.1 Potential Environmental Issues

8.1.1 Regulatory Issues

Permits would be required to treat and dispose of wastes that have been or are impacted with PCBs. The type of permit and limits of PCB concentrations in the waste for disposal will vary by State and sufficient time should be allowed to get the permits.

8.1.2 Air Discharge

The BTS process described will not normally result in discharge of chemicals to the atmosphere with the exception of some loss of solvent (ethanol) from the paste.

8.1.3 Waste Storage, Treatment, and Disposal

The BTS process will result in the generation of a small amount of waste that can be allowed to dry and then disposed of as non-hazardous waste that must be managed consistent with other non-hazardous waste.

8.2 End-User Issues

Potential end-users of this technology include responsible parties for sites where PCBs are present in structures in paint or other building components. End-users will have an interest in the technology because it can potentially treat PCBs *in situ* and allow for reuse of buildings and other structures without concern regarding the future liabilities related to PCBs or allow for a lower cost less than for conventional demolition and disposal approaches and other PCB handling technologies like sandblasting. End-users and other stakeholders may have concerns regarding: 1) the effectiveness of the technology in reducing concentrations of PCBs below appropriate criteria; and 2) the costs of the treatment versus disposal of untreated materials and the evaluation of the long term liability associated with disposal of untreated materials.

8.3 Procurement Issues

There are no specialized equipment components required to implement BTS and no specialized services required. There are a number of magnesium powder and other ingredient vendors. There are no significant procurement issues with the application of this technology.

8.4 Design Issues

Application in cold weather (i.e., below freezing) may be difficult because of difficulties in maintaining an effective seal with the sealant used to prevent the solvent from evaporating during treatment.

Treatment of paint with high concentrations of PCB (i.e., greater than ~ 500 mg/Kg) that may require multiple applications of BTS paste or the use of thicker layers of BTS paste to achieve treated paint criteria of 50 mg/Kg.

Treatment of painted surfaces where the paint is friable or is not adhering to the surface to be treated. In these situations it may be appropriate to remove loose paint from the surface prior to treatment of the surface with the BTS paste. The paint removed from the surface prior to application of the BTS paste could be treated on site using a non-thickened version of the BTS reagent to extract PCBs from the paint chips and degrade the PCB.

Application of BTS paste to irregular surface areas requires a thinner, sprayable form of the solvent paste to adequately cover the exterior. Sealants tested in this demonstration are all sprayable materials and do not pose any application difficulty and will maintain required moisture levels for PCB extraction from the surface coating. As with any surface, the need for removal of spent solvent and reapplication of fresh solvent paste is dependent upon initial PCB concentrations and required final PCB concentrations. If the irregular surface to be treated is small and accessible, it may be removed and placed in a tank of solvent paste (dip tank), to ensure complete saturation of the coating.

Application of BTS paste to large, flat surfaces, such as a vertical wall may be most easily accomplished with a sprayable form of the paste, then covered with a sprayable sealant to maintain required moisture levels for PCB extraction.

Formulation of the BTS paste to be applied to the painted surface. This work has demonstrated that it is possible to use a treatment formulation that contains Mg in an acidified ethanol solvent. This formulation of Activated Metal Treatment System (AMTS) may be more attractive than the Mg/Pd formulation because of lower cost and avoiding the use of Pd.

Use of a single step or two step process for the treatment of surfaces. This work has suggested that it is possible to use a two step process of extraction of PCBs using a non-metal paste and then activating the non-metal paste in a separate container following extraction (Figures 2-1, 2-1, Section 5.1).

Regulatory limits and therefore target treatment concentrations are also an important design issue. The Federal regulation for the disposal of a waste as a non-PCB waste is 50 mg/kg, but some state regulations are 5 mg/kg. For the situation with the Navy properties in Japan, the regulations are very strict and any waste with PCB concentrations >0.5 mg/kg cannot be disposed of in Japan. This requires that the US Navy transport all materials impacted by PCBs to another location for disposal greatly increasing the costs. Thus, a more restrictive regulatory limit will tend to increase the cost effectiveness of BTS treatment of facilities over straight demolition and disposal. When designing a treatment where regulatory or target treatment concentrations are very low, it is important to design for the potential need of additional applications of BTS to achieve the remediation target. For instance for an application with a starting concentration of 500 mg/kg, one application of BTS may be sufficient to get the concentrations below 50 mg/kg but a second application may be necessary to get it below 5 mg/kg.

8.5 Follow-On work

Studies conducted at UCF after the project was initiated have shown that the Pd catalyst can be removed from the BTS paste and a small amount of acid added to make a paste that is both less expensive and more reactive. The addition of a small amount of acetic acid to the ethanol significantly increased the rate of PCB degradation. These studies also showed that acidified ethanol with Mg particles was as effective or in some cases more effective than the Mg/Pd particles in non-acidified ethanol at degrading PCBs. Present evidence suggests that a hard layer of impervious magnesium oxide (MgO) is formed on the outside of the Mg particles over time. The use of acidified ethanol after the MgO layer is formed is only effective at penetrating a small fraction of the layer. However, when acidified ethanol is used during the initial preparation of the BTS paste, the formation of the impervious layer of MgO appears to be minimized to a level where reaction rates remain high. This discovery is extremely important because it means that Pd is not required in the BTS formulation, which reduces the cost of the BTS paste significantly.

BTS has been modified by removing the Pd and is referred to as Activated Metal Treatment System (AMTS). AMTS consists of activated elemental magnesium in a green-solvent solution capable of hydrogen donation. As mentioned above, the AMTS is less expensive than BTS through further modifications to the formula (i.e., different sorbent material as well as removal of the Pd). If the costs for AMTS are used in place of BTS in the cost tables in Section 7, it would be cost effective to apply AMTS to a concrete building prior to demolition compared with the costs of demolishing the building and disposing of the waste in a TSCA landfill. Demolition and disposal costs of the untreated concrete building are estimated to be approximately $200,000, while the cost of treating the painted concrete with BTS, followed by demolishing the building and recycling the concrete is estimated to be approximately $180.000. If AMTS is used in this scenario instead of BTS the cost is estimated to be approximately $75,000.

In contrast, if the structure to be treated is a metal tank, then it does not seem to be cost effective to address the PCB-containing paint by either conventional sandblasting, BTS treatment or AMTS treatment in order to recycle the tank. Straight disposal of the untreated tank is estimated to cost approximately $25,000 versus removal of the paint with sandblasting and recycling the tank which is estimated to cost $105,000 and treatment with BTS and recycling the tank which is estimated to cost $140,000. Treatment with AMTS and recycling the tank is estimated to cost $33,000.

NASA and Scientific Specialists jointly hold the patents for both BTS and AMTS and there is already commercial sector interest; the technologies are licensed to two companies with other licenses in process

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Appendix A: Points of Contact

Appendix A: Points of Contact

A summary of contact information for key personnel associated with the technology demonstration is presented in **Table A-1**.

Point of Contact	Organization	Phone/Fax/E-mail	Role in Project
Dr. Nancy Ruiz	NFESC	(805) 982-1155 Fax (805) 982-4304 nancy.ruiz@navy.mil	Principal Investigator
Tom Krug	Geosyntec Consultants	(519) 822-2230 Ext. 242 Fax (519) 822-3151 tkrug@geosyntec.com	Technical Advisor
Suzanne O'Hara	Geosyntec Consultants	(519) 822-2230 Ext. 234 Fax: (519) 822-3151 sohara@geosyntec.com	Project Manager
Dr. Jacqueline Quinn	NASA	(321) 867-8410 Fax (321) 867-9161 Jacqueline.W.Quinn@nasa.gov	Technical Advisor and Project Direction at NASA
James Captain	NASA/UCF	(321) 867-8185 lab: (321) 861-3624 fax: (321) 861-2925 James.Captain-l@ksc.nasa.gov	Technical Advisor/Field QA/QC
Dr. Cherie Geiger	UCF	(407)823-2135 Fax(407)823-2252 cgeiger@mail.ucf.edu	Technical Advisor and Project Direction at UCF
Dr. Chris Clausen	UCF	(407)823-2293 Fax(407)823-2252 clausen@mail.ucf.edu	Technical Advisor and Project Direction at UCF

Appendix B: A Final Report on Development of a Field Deployable Methodology for Safely and Effectively Degrading PCB Contaminated Oils and Machine Shop Equipment Located at the U.S. Navy Shipyard in Yokosuka, Japan

APPENDIX B

A Final Report

on

Development of a Field Deployable Methodology for Safely and Effectively Degrading PCB Contaminated Oils and Machine Shop Equipment Located at the U.S. Navy Shipyard in Yokosuka, Japan

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INTRODUCTION

Polychlorinated biphenyls have been associated with a variety of risks to humans and ecosystems and the proper management of PCB contaminated materials has proven to be a widespread, complex, and costly issue (NCR, 2001). PCBs have been widely used in oils ranging from capacitor oils to machine oils to mineral oils due to their myriad of beneficial properties for extending the lifetime of the functional use of the oil. Unfortunately, leakage of these oils or improper disposal has caused serious environmental and health issues. Attempts at proper disposal of these contaminated oils include storage in hazardous waste landfills or incineration, both of which offer less than optimal solutions. The problem of contaminated oils actually represents two issues from a scientific standpoint: 1) separation of the PCBs from the oil and 2) subsequent degradation of the PCBs. Achieving solutions to these issues would leave clean, non-contaminated oil and no PCBs to dispose of in a landfill or by incineration.

Analytical research has led to the discovery of several methods for separation and analysis of PCBs from oil matrices. These include dimethylsulfoxide extraction, normal-phase liquid chromatography, gel permeation chromatography, reverse-phase liquid chromatography (Numata, et al., 2008), molecular distillation in a short-path evaporator (Cvengros and Filstein, 1999), and chromatography using sulfoxide-bonded silica (Numata, et al. 2007). The use of low density polyethylene packing material for separation of PCBs from polar and non-polar solvents has been investigated but with little success in oils and other nonpolar substrates (Sklarsova, et al. 2008). While these methods may be useful in laboratory experiments, they have not been useful for applications at contamination sites. Various forms of alumina have been found to have a range of sorptive capacities for separation of environmental contaminants from their matrices including PCBs from waste oils. Surface modifications on alumina can be used to tailor the mineral to differing uses including solvent purification, PCB removal from different matrices, lead removal from water, biomass clean-up, etc. Therefore, alumina has been shown to have the properties necessary to separate PCBs from oil, but degradation of the PCBs still must be accomplished. In order for that to occur, there must be an option for desorption of the PCBs from the alumina to an appropriate solvent for the chosen degradation method.

There has been a great deal of research focused on the degradation of PCBs in the past two decades. This class of molecules is resistant to simple reductive dehalogenation that has been used for many chlorinated aliphatics. Other recent methods for PCB degradation include gamma radiolysis (Tajima, et al. 2008), catalytic degradation with palladium on carbon (Kume et al. 2008), catalytic degradation with palladium on nanoiron (Zhang, et al., 1997), microbial degradation (Ahn, et al., 2006), dechlorination on calcium oxide, or treated fly ash (Gao, et al., 2008) at temperatures above 350°C. All of these methods have limitations and with the exception of catalytic degradation, yield less than 60% degradation of Aroclor mixtures. Most of these methods are relatively slow (low degradation within 30 days) and require very controlled conditions (ex. argon atmosphere, electric current, either high temperature or controlled temperature) for even that level of effectiveness. With the exception of gamma radiolysis, these methods either have not been tested in oils or require that the PCBs be introduced via a low molecular weight solvent, not an oil substrate.

Research at the University of Central Florida (UCF) and NASA, Kennedy Space Center has shown that palladium mechanically alloyed with magnesium metal produces an active bimetal system that can degrade PCBs in methanol or ethanol in hours to weeks (depending on the PCB congener). Researchers at UCF found that using magnesium mechanically milled with carbon (no palladium) in acidified ethanol (1% acetic acid) produced reaction rates that far exceeded those found with the palladium/magnesium system (with or without acid). Using the magnesium/carbon/acidified ethanol system, Aroclor 1260 can be completely degraded within 24 hours.

UCF has obtained a limited amount of lubricating oil from several milling and cutting machines owned by the U.S. Navy (Yokosuka, Japan). This oil was contaminated at very low levels of Aroclor 1260. In order to mimic the higher concentration oils, the wash oils were spiked with Aroclor 1260 to 1-2 mg/kg. Researchers at UCF have demonstrated that an active alumina can capture PCBs in this oil and the PCBs can then be desorbed at will with an ethanol rinse. Magnesium ball-milled with graphite can then be added to the ethanol and the mixture can then be acidified with acetic acid, followed by PCB degradation.

It is envisioned that this technology can be incorporated through piping to the oil reservoir and pumped through the alumina adsorption bed, even while the machine is in operation. When the sorption bed reaches its PCB mass capacity, it can be replaced with a clean sorption bed to continue the oil recirculation process. As the clean oil circulates through the machine, more of the trapped PCB (PCBs in seals and gaskets) will transfer to the oil and be removed onto the sorption bed. The sorption beds can then be shipped to UCF for desorption of the PCBs from the alumina substrate and subsequent degradation with the magnesium/graphite/acidified ethanol system described above.

FEASIBILITY STUDIES

Problem Statement

A Navy operation located in Yokosuka, Japan has a series of machines for operations in milling and cutting industrial materials. Over the years, oils containing polychlorinated biphenyls (PCBs) have been used for lubrication in these machines and during this time, PCBs have contaminated the interior of the surfaces of the equipment, including seals and gaskets. Attempts have been made to 'clean' the PCBs out of the equipment by replacing the contaminated oil with new (wash oil) oil but a PCB concentration above the regulatory limit (Japanese limit of 0.5 mg/kg) persists even in the new oil. The oil received by UCF was the new oil (wash oil), not the original, more highly contaminated oil.

Initial Approach

The researchers at UCF have been tasked without specific funding for this project with analyzing and attempting to remove and degrade the PCBs from this oil sample. Once the sample was received, the UCF researchers carried out a literature search to identify analysis methods that would achieve the limits of detection necessary to quantitate the PCBs in the sample. After a clean-up and analysis method was determined, work continued with the goal of removing/degrading PCBs in the oil sample using active metals and bimetal systems. We have achieved success with degradation of the PCBs in the sample oil and are proceeding to develop a system that can remove the PCBs from the oil in a technology that is compatible with the equipment being used on site. Our goal was to develop a technology that will allow the continued use of the machines, or if shut-down is necessary, remove the PCBs from the oil in the machinery without dismantling the machines.

Samples and Date of Receipt

On July 30, 2008, Drs. Clausen and Geiger (University of Central Florida) obtained an approximately 125.0 mL sample of machine oil originally shipped to Dr. Quinn (NASA, Kennedy Space Center) from Yokosuka Japan. The sample consisted of a light weight machine oil. Reports supplied by Christine Ricafrente from the Naval facility in Yokosuka specify that oils from various machines are as high as 4.8 mg PCB/kg oil to a low of 0.01 mg PCB/kg oil. However, the oil that was received by UCF was a wash oil used after the most highly contaminated oil was removed from the planning machine. The wash oil concentration was 0.16 mg PCB/kg oil. During conversation with Dr. Nancy Ruiz (Navy) on 9/11/08, we have requested more oil samples from Yokosuka as well as the specifications for the oil and the supplier so we can begin more tests on PCB spiked oil. Dr. Quinn (NASA) provided a portion (approximately 125 mL) of another sample of the wash oil from Yokosuka which was received at UCF on 9/12/08.

Experimental Procedures

Analysis: 2-mL of oil is acid washed with sulfuric acid by adding 0.5 mL of concentrated sulfuric acid to the oil, shaking vigorously for 2-minutes, then adding another 0.5-mL of acid and shaking. This is repeated four times, the oil layer is separated from the acid and 1.0-microliter is injected directly onto a gas chromatograph-electron capture system. Conditions for the GC-ECD are as follows: injector temperature of 275°C, GC oven temperature profile starting at 120°C and holding for one minute, ramping at 20°C /minute to 200°C , ramping at 10°C /min to 270°C and holding at that temperature for 15 minutes. The column is an RTX 5, 30 meters in length and 0.25-mm id., and column flow of 1.33 mL/minute helium. Calibration curves were generated by using the oil spiked with increasing amounts of Aroclor 1260. A sample calibration curve (using the method of standard editions) is included in the data below. Using this method, we have spiked the oil (already containing a small amount of PCBs) with increasing amounts of Aroclor 1260. This yields a graph that shows an X-intercept at approximately -0.19 mg/kg, the offset caused by the concentration of the PCBs in the oil. This common method for determining concentrations yields a concentration of approximately 0.19 mg PCB/kg oil.

Extraction efficiency for this method was determined to be 102%. This slight overage is likely due to a small amount of oil being trapped in the acid layer and thus the PCBs being concentrated in the remaining oil layer. The detection limit using this extraction and analysis method is 0.035 mg PCB /kg of oil.

Degradation experiment, preparation and extraction: For all experiments to determine degradation of PCBs with the metal/bimetal in the machine oil matrix, 2-mL of solution was used with 50-mg of metal or bimetal /mL of solution, varying only as we have run low on the machine oil in the past week. When we have used a lower volume of oil, the metal or bimetal ratio was kept constant with earlier experiments. The solution used contained ethanol (acidified to 1% by volume acetic acid) mixed with the oil at an acidified ethanol concentration ranging from zero to 50% acidified ethanol. The oil/acidified ethanol solutions were spiked with Aroclor 1260 to a concentration of between 1.0 mg PCB/kg oil to 2.0 mg PCB/kg oil in order to have a sufficient quantity to verify trends in degradation and to aid in measuring by-product formation.

Extraction and analysis: At set time periods, vials were opened and extracted and an acid clean-up was performed prior to analysis on GC-ECD. The vial contents were filtered with a Whatman 45 micron pore-size nylon filter syringe, 0.5 mL of water was added (to aid in separating the ethanol solution), the vials were shaken for two minutes then centrifuged to separate the aqueous/ethanol layer from the oil layer. The oil layer was then removed and acid washed as described in the Analysis section (above). The samples were then analyzed on the GC-ECD (also described in the analysis section). Extraction efficiency was again tested and found to be the same as for the calibration methods described above.

RESULTS

The initial challenge in achieving our objectives was to simply be able to accurately analyze for the PCBs in the oil matrix. Figure 1 below shows a calibration curve for PCBs in this matrix. As described in the Analysis section above, the calibration had to be done with the contaminated oil so the Method of Standard Additions was done to determine both the concentration of the oil and to obtain an accurate method for quantitating the concentration in this matrix.

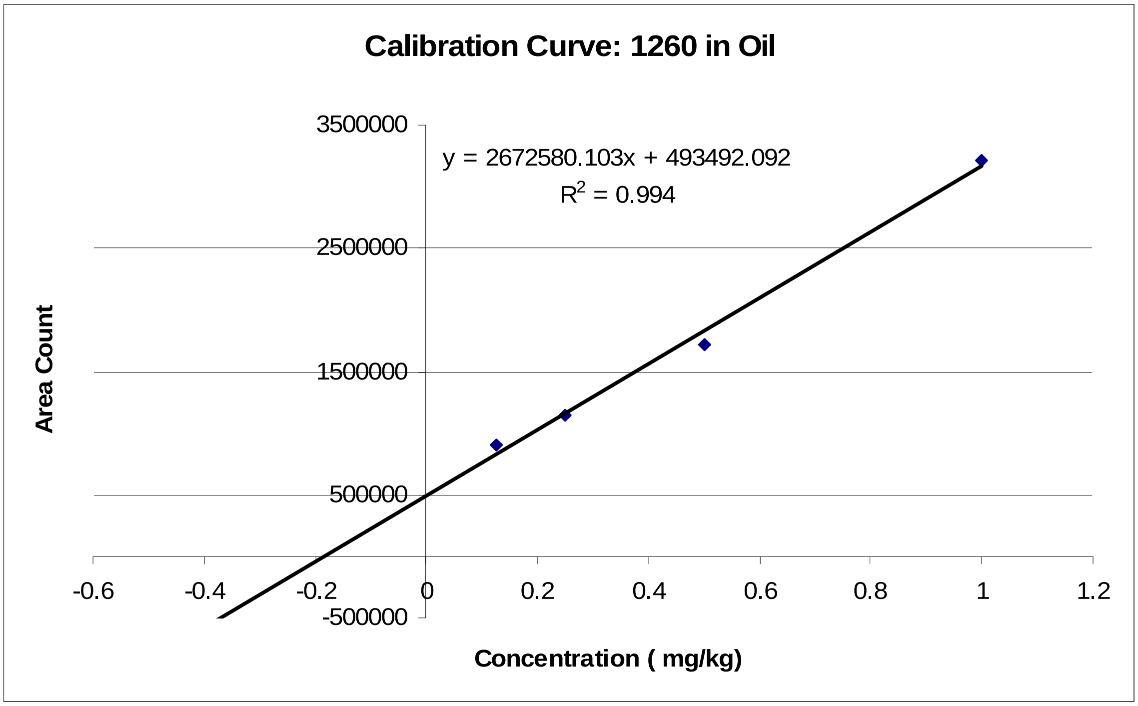

Figure 1. Calibration of Aroclor 1260 and concentration of contaminant PCBs in the oil obtained from the Yokosuka, Japan Naval facility.

Several different experiments to determine PCB degradation options have been carried out over a period of four weeks. The candidate metal/bimetal systems that were tried included: magnesium metal alone, magnesium metal ball-milled with carbon, and magnesium metal ball-milled with palladium on carbon. The magnesium metal used in this work is 3-4 micron particles. The following descriptions and graphs show the preliminary results achieved using these different metal particles with varying concentrations of acidified ethanol.

Magnesium metal alone: Magnesium metal, with no carbon or carbon/Pd, showed no activity toward degradation of the PCBs in these experiments. This was consistent no matter the concentration of acidified ethanol used (1% or 50%) or the metal loading (50 mg/mL or 500 mg/mL). All of these experiments were carried out for only 24 hrs. but since no degradation was obvious in that time period, further experiments over longer time periods were not conducted.

Magnesium metal ball-milled with carbon: Magnesium ball-milled with carbon showed no degradation in 24 hrs. with only 1% acidified ethanol but did show significant degradation with 50% acidified ethanol in 24 hrs. Figure 2 below shows the concentration change from the initial concentration to one day and seven days.

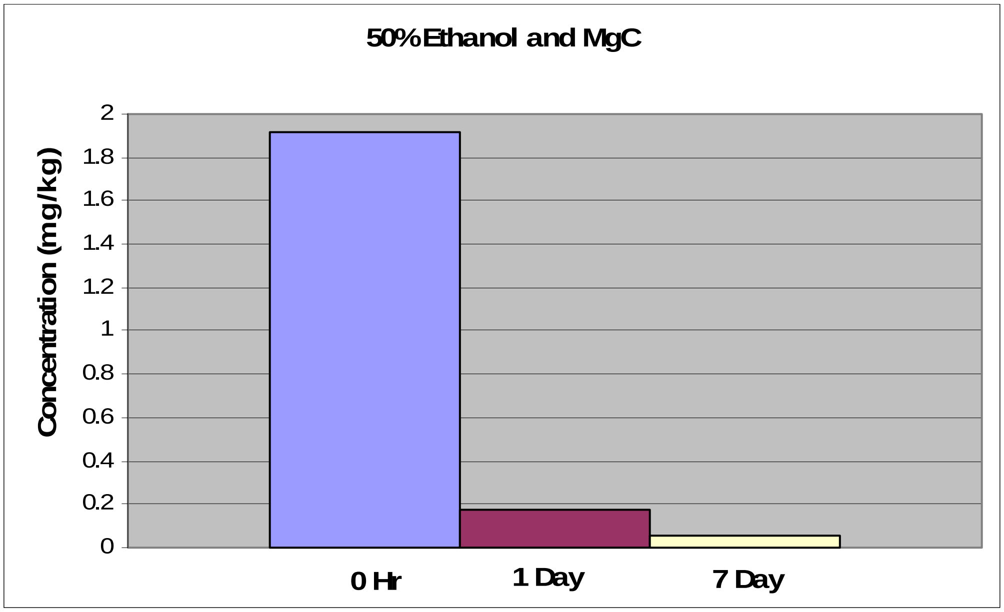

Figure 2. Concentration changes in PCB concentration in oil with 50% acidified ethanol and 50 mg MgC/mL of solution. Starting concentrations of 1.93 mg/kg.

This reactive system produces by-products (lower chlorinated PCBs) that are quickly consumed and degraded. Figure 3 (a-c) illustrates the formation and degradation process of these by-products over a six day period. Due to the short time period since we have had access to the samples, full identification and rate information has not been performed.

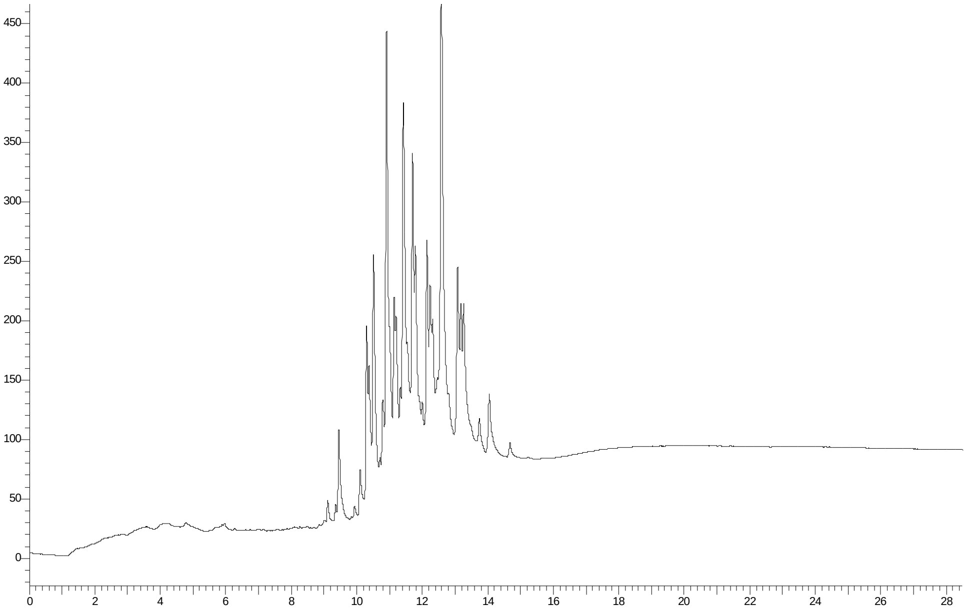

Figure 3 a. A 50% acidified ethanol/oil solution at time zero.

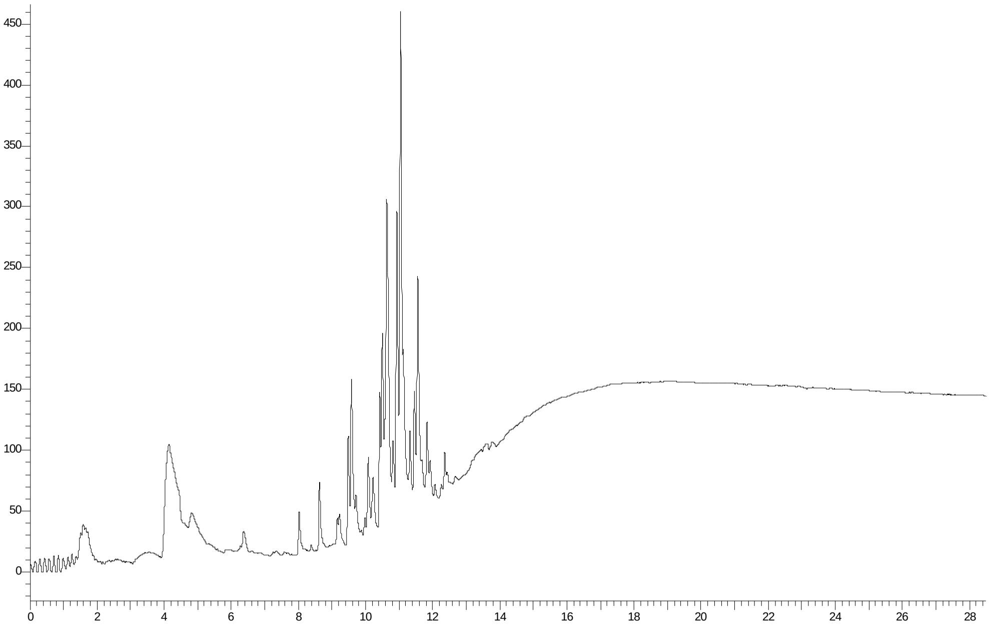

Figure 3 b. A 50% acidified ethanol/oil solution with 50 mg MgC/mL after one day.

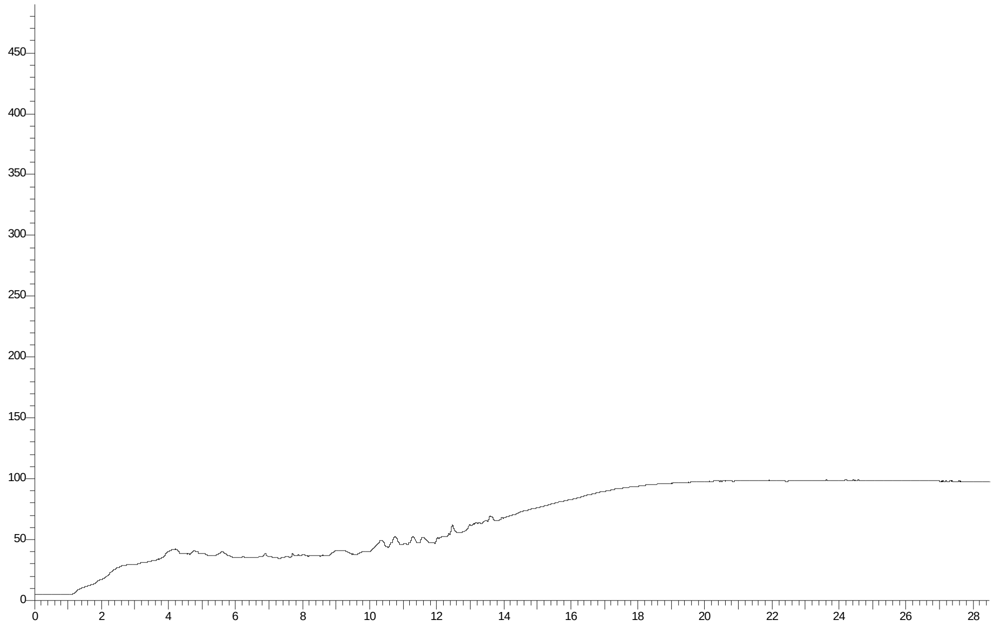

Figure 3 c. A 50% acidified ethanol/oil solution with 50 mg MgC/mL after seven days.

Magnesium metal ball-milled with Pd on carbon: The Mg/Pd bimetallic system allowed for slight degradation when 1% acidified ethanol was used with a metal loading of 500 mg MgPd/mL of solution. However, a very rapid degradation occurred with 50% acidified ethanol with 50 mg MgPd/mL of solution. Figure 4 in the following text shows degradation to detection limits within 24 hrs.

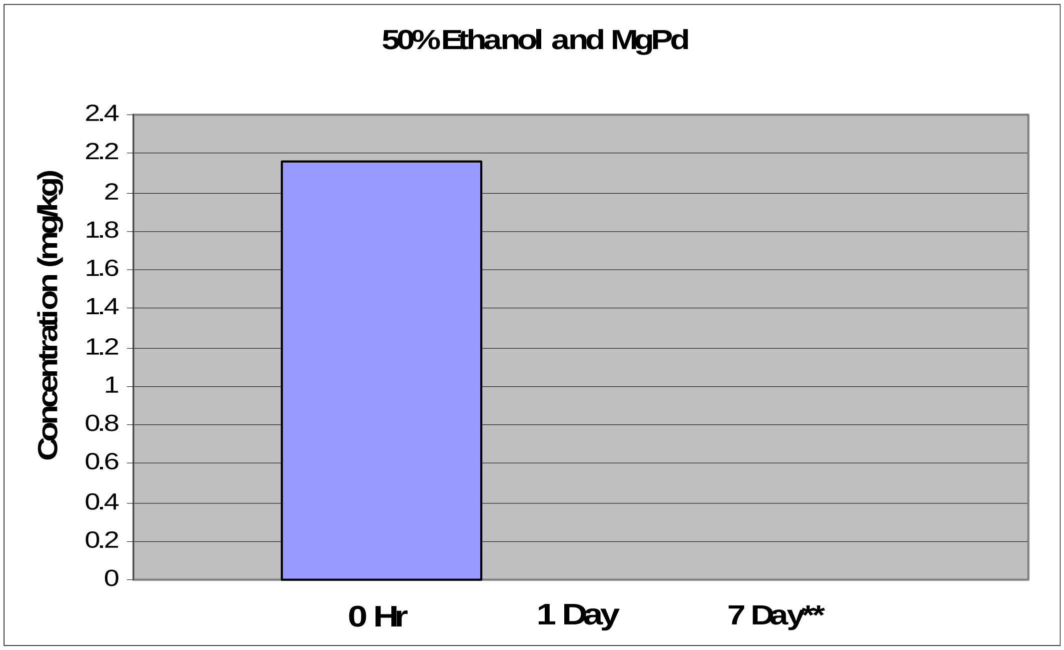

Figure 4. Degradation of PCBs in oil with 50 mg MgPd/mL of solution. (**Sample could not be isolated). Starting concentration of 2.2 mg/kg.

The figures below are included to show that a degradation mechanism is occurring with the parent PCB peaks disappearing and lower chlorinated peaks appearing and then follow-on degradation of these daughter products occurs. Figure 5a is a chromatograph of the time zero concentration with Mg/Pd (50 mg/mL of solution) in 50% acidified ethanol/oil solution. Figures 5 b and c are chromatograms at two days and six days, respectively. Note that the original peak envelope of the Aroclor 1260 mixture disappears and only a small concentration of daughter products (likely less than 2% of the original Aroclor concentration) remains. Further identification and quantitation of these products will be done in the next month if funds are obtained to continue this project.

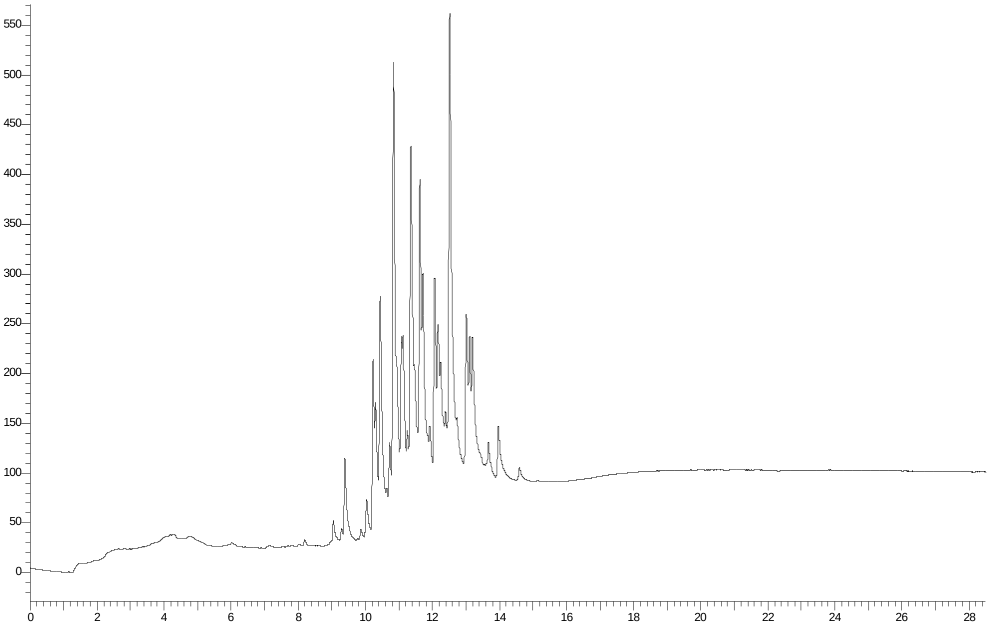

Figure 5a. Acidified ethanol/oil solution spiked with Aroclor 1260 at time zero.

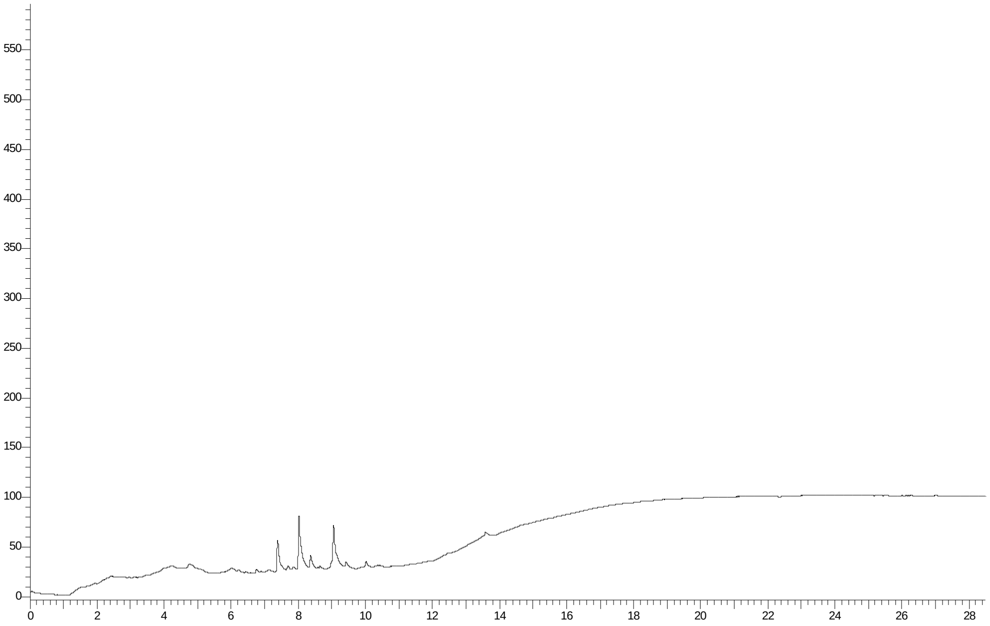

Figure 5b. Acidified ethanol/oil solution spiked with Aroclor 1260 with 50 mg MgPd/mL solution after two days.

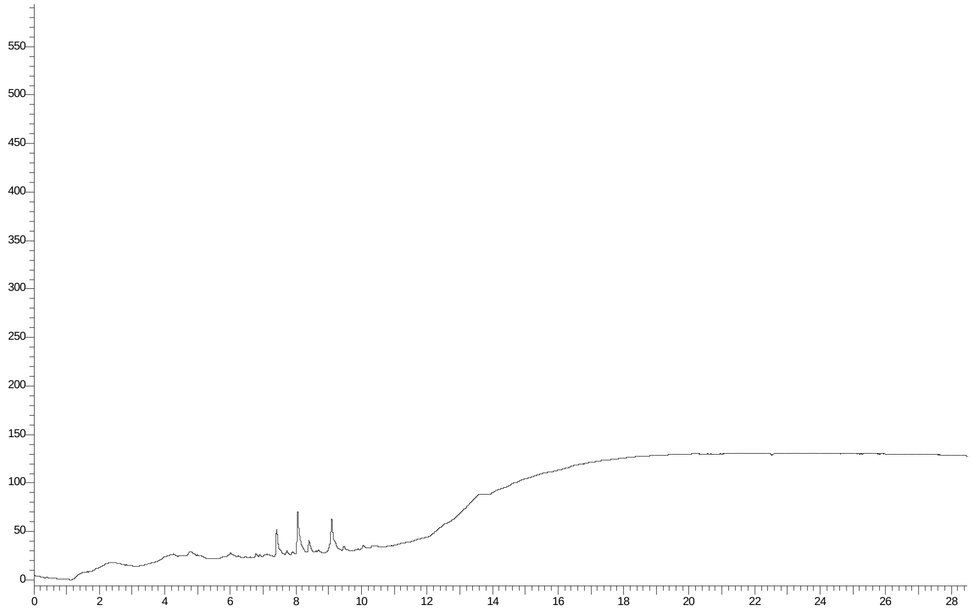

Figure 5c. Acidified ethanol/oil solution spiked with Aroclor 1260 with 50 mg MgPd/mL solution after six days.

A NEW APPROACH-THE USE OF A PCB ADSORPTION/DEGRADATION PROCESS

In the interest of developing a process for PCB degradation in the machine oil that does not involve the introduction of acidified ethanol into the machinery, the UCF team decided to test a PCB adsorbtion/degradation concept. Various adsorbent materials were tested for their ability to adsorb PCBs. A series of syringes was set up, each containing a small plug of glass wool (to prevent the escape of adsorbent material) and 3 mL of column material. The small-scale testing concept was dictated by the small amount of test oil that was made available to us. A picture of the experimental set-up is shown in Figure 6. A 5 mL volume of machine oil, spiked to approximately 1 mg/kg with PCB 1260, was passed through each syringe. This eluent was then acid-washed prior to GC-ECD analysis to determine whether the PCBs had been adsorbed. Materials included silica gel, Fluorosil, crushed carbon black, activated carbon (from a VOC mask filter) both crushed and uncrushed, activated alumina (Fisher), and super activated alumina I (Alltech). Of these adsorbents, only the super activated alumina was successful in removing PCBs from the oil.

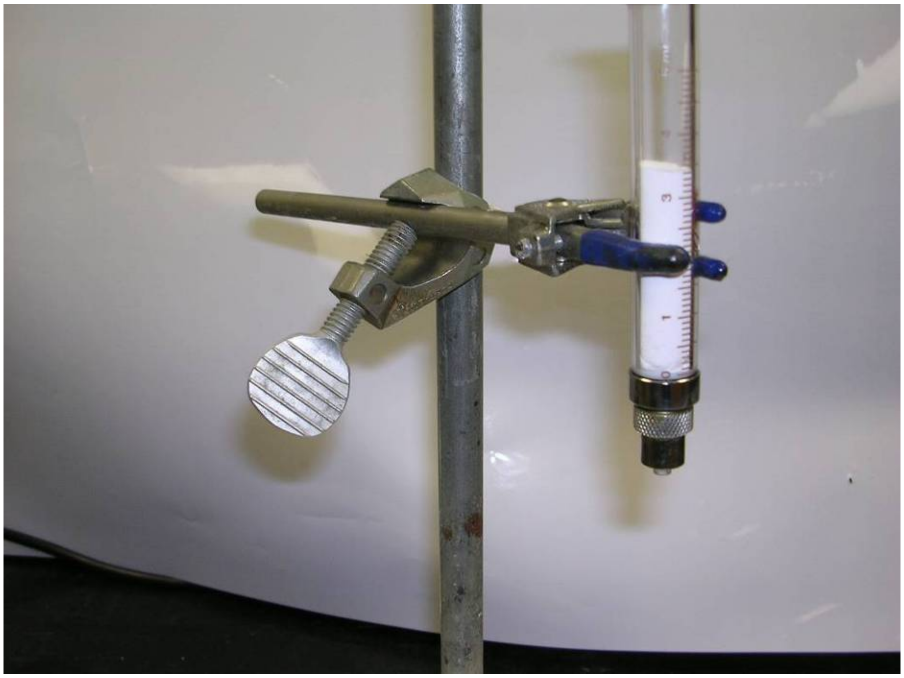

Figure 6. Initial set-up for conducting PCB adsorption experiments.

The super activated alumina was subjected to further tests to determine whether the PCBs could then be removed and degraded. A syringe was set up as in the prior study, and 5 mL of spiked Navy machine oil was passed through the material.

Table 1. Results from first experimental set-up for alumina adsorption followed by desorption with ethanol, followed by treatment with Mg/C.

Media Tested	PCB Conc (mg/kg)
Spiked Navy Oil	0.8591961
Oil after passing through alumina column	Below detection limit (0.035 mg/kg)
PCBs desorbed from alumina column	0.3643
PCBs after one day treatment with Mg/C and acidified ethanol	Below detection limit (0.035 mg/kg)

In our study, it was shown that the PCBs could be effectively removed by the super activated alumina, and sonication of the column alumina in 5 mL of ethanol for 30 minutes was found to remove and recover the adsorbed PCBs (see Table 1 above). This ethanol was acidified and combined with 0.05 g of 80% Mg/20% Fe, then put on the shaker table for 1 day. After shaking with an equal amount of toluene for 2 minutes to transfer the PCBs to the organic phase, the toluene layer was removed and acid-washed. The degradation was found to be successful. Negative concentrations in this table stem from the fact that the oil used for both this experiment and the calibration curve was from the Navy site, and already lightly contaminated with PCBs prior to spiking.

Once it was determined that the super activated alumina could be used in this fashion, a breakthrough point study was conducted. Syringes were set up as in the prior two experiments, but the volume of spiked oil passed through each syringe was varied. When 5 mL of oil was used, the results from the previous test were corroborated and the PCBs were observed to be completely removed. When 7 mL of oil was passed through the adsorbent bed, the PCB 1260 envelope was clearly visible in GC-ECD chromatograms of the eluent, and when 9 mL of the oil was used, the concentration increased, although it did not reach the concentration of the initial spike. When the column material was removed and sonicated in 5 mL ethanol, the same concentration of PCBs was found in the material from the 7-mL and 9-mL columns, confirming that the maximum concentration that can be adsorbed onto a given amount of alumina was reached.

A further breakthrough study was conducted on 10/22/08. A column was constructed using a glass wool plug and 7 mL of super activated alumina, and toluene was pumped through to wet down the column. The experimental set-up is shown in Figure 7. Navy machine oil spiked to 1 mg/kg with PCB 1260 was then pumped through the column at a rate of approximately 1 mL per 12 minutes. The first 4 mL to pass through the column was discarded, as it was just the toluene used to settle the column and would not contain any PCBs. After this, aliquots of 1 mL were collected to monitor the point at which the alumina could no longer adsorb any more PCBs. The breakthrough curve is shown in Figure 8.

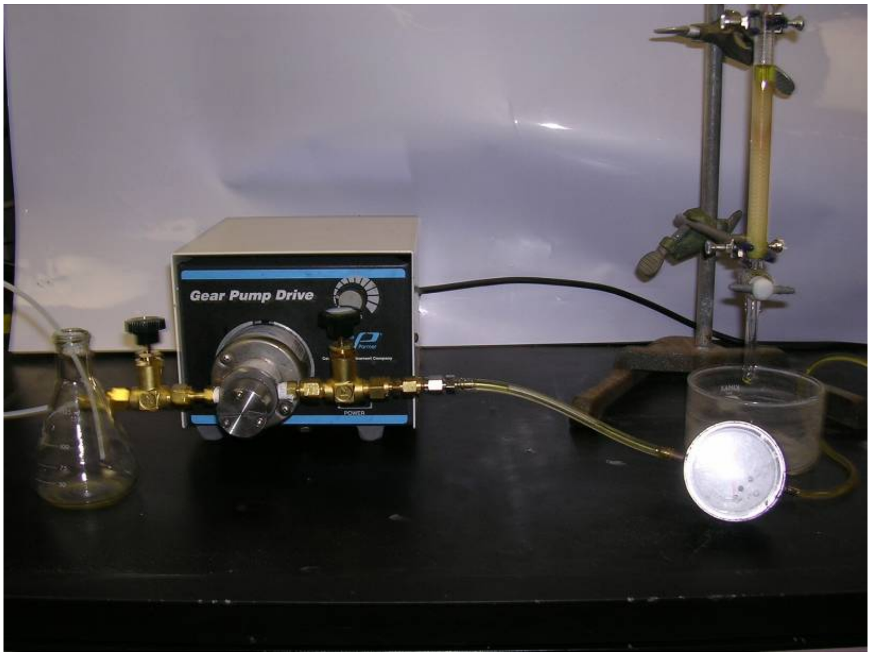

Figure 7. Column adsorption experiment for removing PCBs from oil.

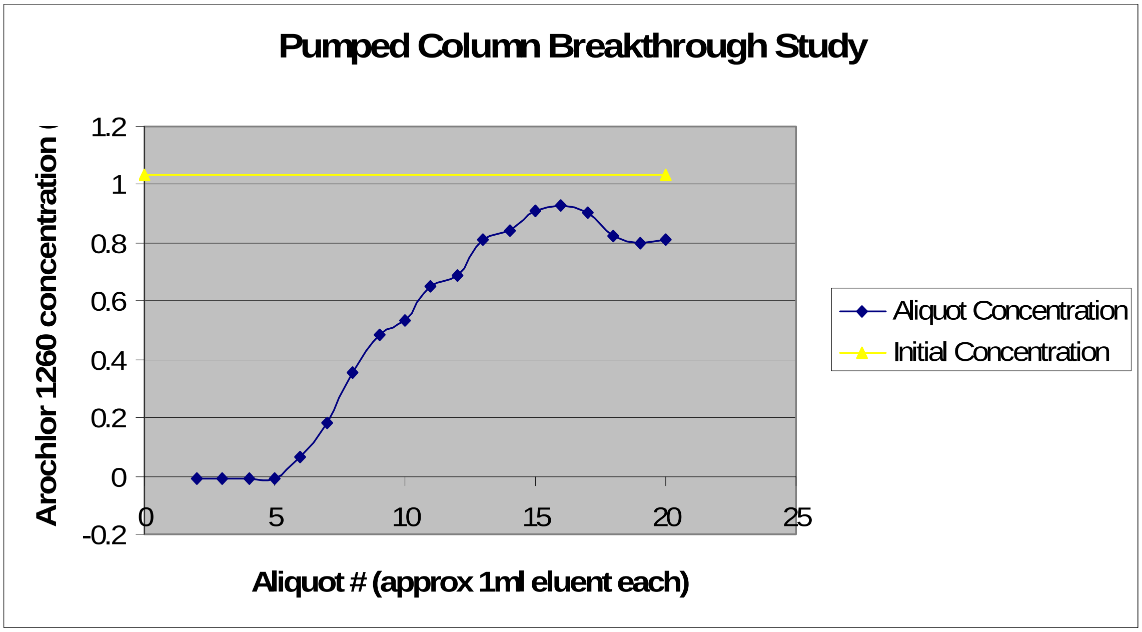

Figure 8. Breakthrough curve for PCB removal from oil.

The rate of PCB 1260 removal from the oil began at a peak of 1.044 µg/mL, but decreased gradually beginning after aliquot 5. The total amount of PCB 1260 found to have been removed from 20 mL of oil as calculated from the area above the elution curve, was 9.29 micrograms.

After collection, air was used to blow as much of the oil as possible out of the column, and absolute ethanol was passed through the column for the purpose of testing removal of PCBs from the adsorbent material without a sonication step. Twenty 1-mL aliquots were taken during this time, and it could be observed that the first two aliquots contained primarily oil that had not been dislodged by the air blown through the column, while those afterwards consisted of primarily ethanol. The PCB desorption data is shown in Figure 9.

Quantitation of the elution data documented that recovery of PCBs from the adsorbent bed was greater than 95% recovery. This confirms that any adsorbent columns used in the field tests do not require the removal of the adsorbent from the column for degradation of the PCBs.

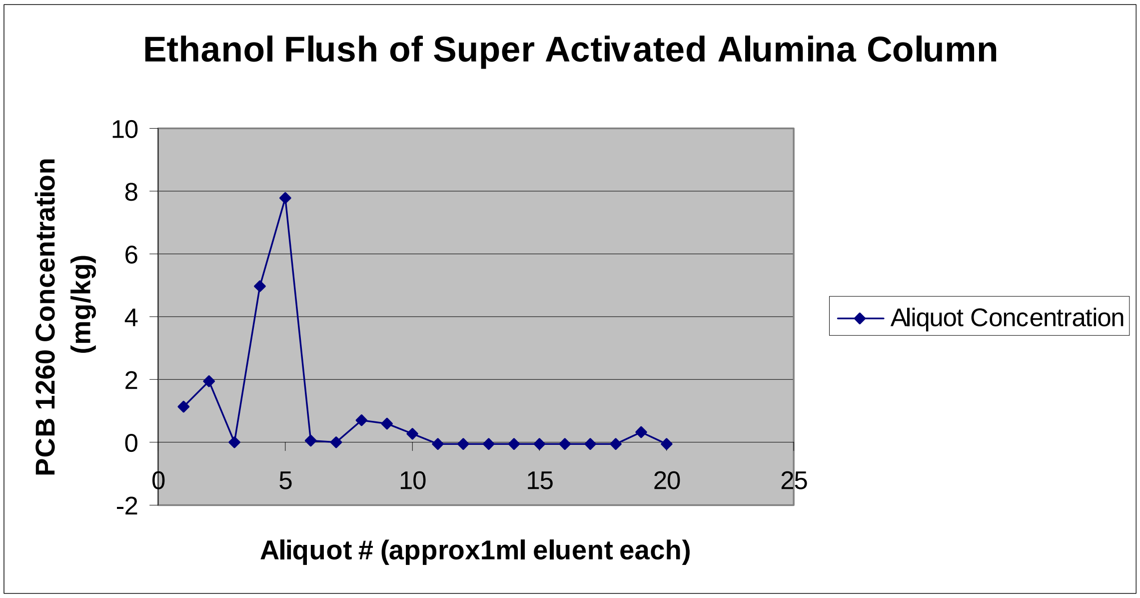

Figure 9. PCB desorption from the alumina column.

Another experiment using a smaller adsorbent bed volume to document almost complete closed-loop capture and degradation of PCBs(even though not optimized) is recorded in the following figures. Figures 10a-e show the Aroclor 1260 peak envelope (in oil received from Navy) at the initial concentration of 1.263 mg/kg (Figure 10a), after passing through the adsorption bed and the PCB concentration dropping to 0.183 mg/kg

(Figure 10b), in the ethanol adsorption bed rinse with concentration of 1.189 mg/kg(Figure 10c), in a second ethanol rinse with a concentration of 0.200 mg/kg(Figure 10d), and finally after exposure to Mg/C for one day (Figure 10e) with a concentration now below the detection limit. These chromatograms provide visual validation that the adsorbent is removing the PCBs from the oil (remember this system is not yet optimized and removal with one pass through the adsorbent column which for this experiment yielded about 85% removal), the adsorbent releases the PCBs when flushed with ethanol, and the PCBs can be degraded quickly with acidified ethanol and Mg/C.

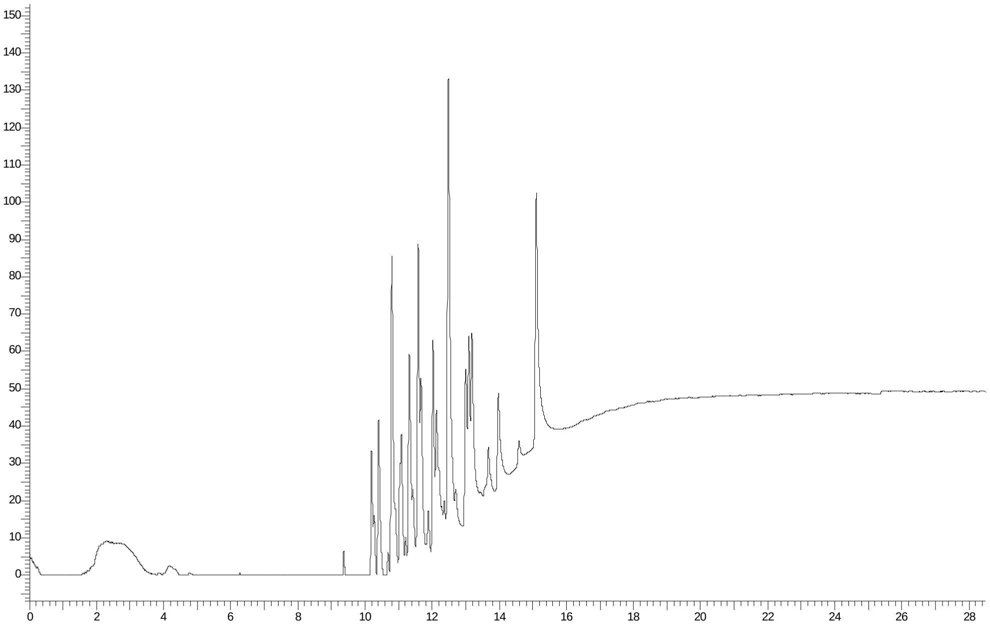

Figure 10a. Chromatogram (GC/ECD) of PCB envelope from Navy oil spiked to with addition ~1 mg/kgwith PCB 1260.
Concentration: 1.263 mg/kg.

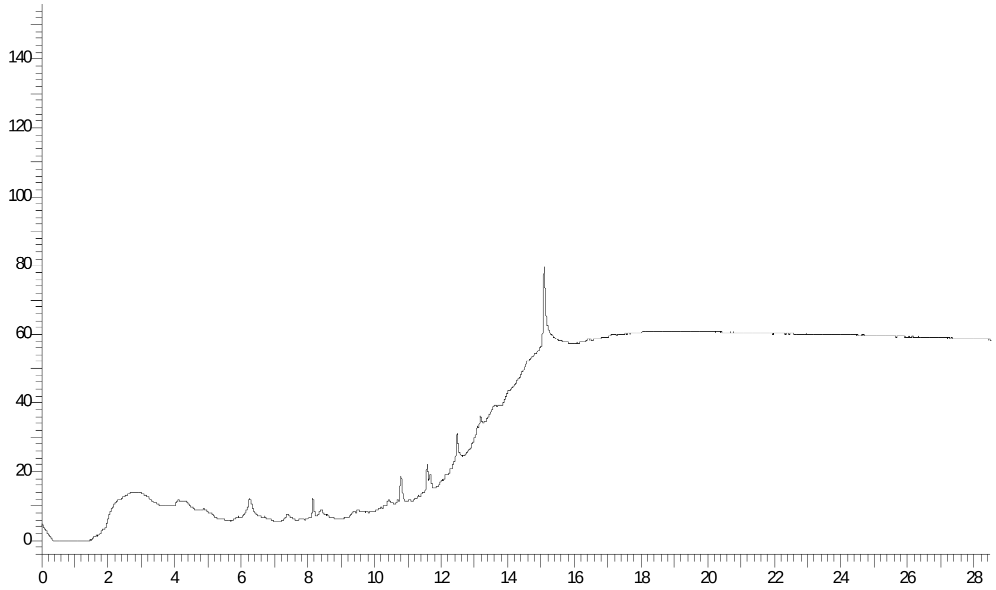

Figure 10b. Chromatogram (GC/ECD) of PCB envelope from Navy oil (5 mL) after passing through super activated alumina adsorption bed. Concentration: 0.183 mg/kg.

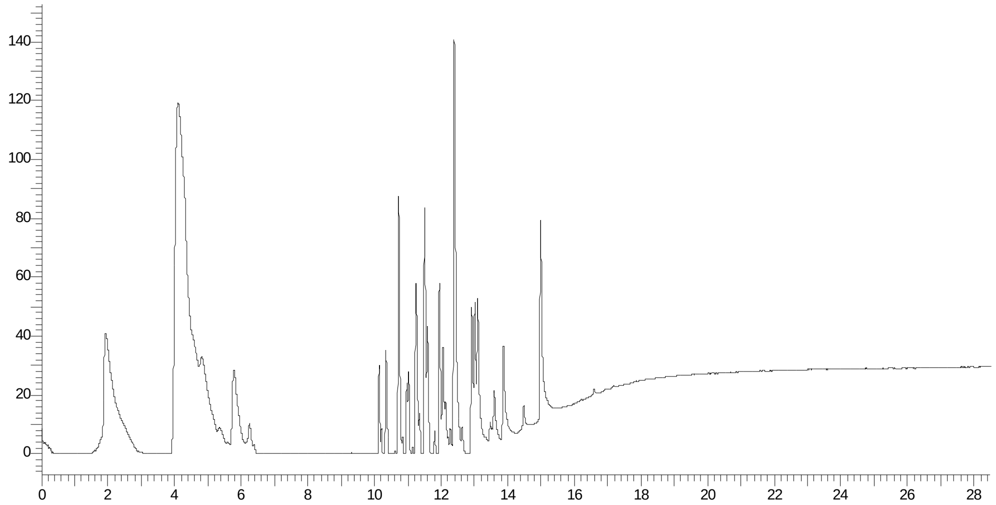

Figure 10c. Chromatogram (GC/ECD) of PCB envelope from alumina in adsorption bed, Concentration: 1.189 mg/kg.

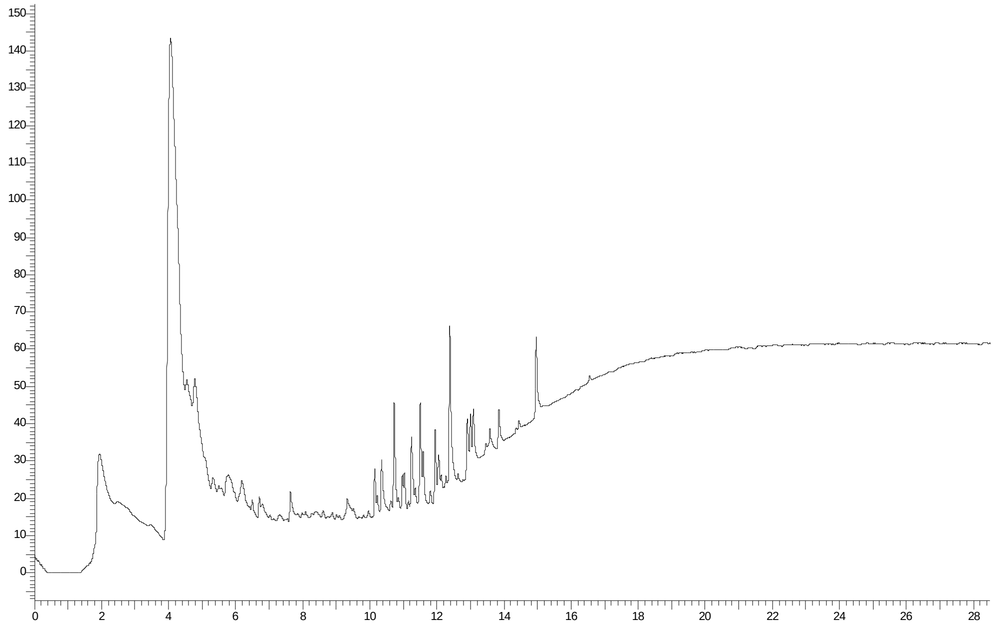

Figure 10d. Chromatogram (GC/ECD) of PCB envelope from alumina in adsorption bed, second 5 mL ethanol rinse.

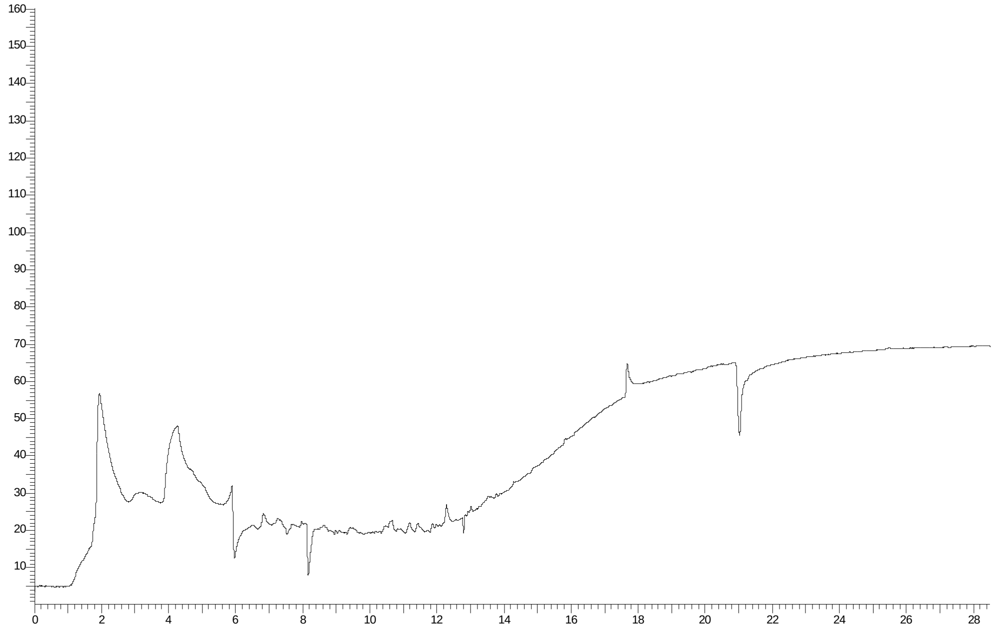

Figure 10e. Chromatogram (GC/ECD) of ethanol/PCBs from 1st sonication, acidified and degraded with Mg/C for 1 day.

Since the Alltech super activated alumina that was used for experiments up to this point is not a large scale commodity product, information about it was collected and some similar, less expensive products were tested for the ability to both remove PCBs from oil and to then desorb the PCBs from the alumina in an ethanol flush. Alumina C from Dynamic Adsorbents, Inc. was tested in syringe studies in the same manner as the super activated alumina, but it was found that the PCBs could not be removed from the alumina after they were adsorbed. This would only relocate the contamination instead of allowing the PCBs to be degraded. Thus, three new different types of alumina were obtained and tested. They consisted of Brockmann I activated alumina from Sigma-Aldrich in weakly acidic, acidic, and neutral varieties. Each of these alumina was capable of removing PCBs from oil and allowing the PCBs to be desorbed from the alumina with an ethanol flush.

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Appendix C: Sampling and Analysis Plan

TABLE OF CONTENTS

LIST OF TABLES

LIST OF ATTACHMENTS

1. INTRODUCTION

Geosyntec Consultants, Inc. (Geosyntec) has prepared this Sampling and Analysis Plan (SAP) for the Environmental Security Technology Certification Program (ESTCP) to evaluate the application of a Bimetallic Treatment System (BTS) to remove and degrade PCBs found on Department of Defense (DoD) facilities. The field portion of the Dem/Val addressed in this report will be conducted at the Badger Army Ammunition Plant (BAAP) Saulk County, Wisonsin. This site has had PCBs identified in the paint on structures and metal equipment, appropriate site conditions and a suitable on-site support network for execution of the Dem/Val. Baseline characterization and performance monitoring events are planned for April 2008 through July 2008, and will be conducted to meet the following field demonstration objectives:

1. Determine the protocol for formulating BTS for site-specific conditions to enhance applicability to various PCB-containing materials found across numerous DoD facilities while maximizing safety and efficacy with the ultimate goal of reducing PCB concentrations to less than 50 mg/kg.
2. Demonstrate the effectiveness of BTS on a wide range of actual contaminated structures at three DoD facilities. The relationships between dose applied, repeated applications and reaction kinetics will be evaluated with the intention of specifically identifying the factors influencing treatment and limiting reaction rates for a specific media (e.g. different painted structures). Environmental condition effects (temperature and humidity; weathering) and impact of BTS on material appearance and adhesion will also be evaluated.
3. Review BTS application and handling process and develop appropriate on-site safety protocols for institutions to implement during its application, including the handling of any site-specific waste products generated by BTS and the application process. The BTS process itself produces non-toxic waste.
4. Develop full-scale cost and performance reports for DoD facility end-users to utilize when addressing PCBs found on existing structures.

Specifically the sampling and monitoring results will be used to:

- Assess the starting PCB concentrations in the paint prior to BTS application;
- Evaluate the length of time that BTS needs to be applied to achieve extraction of the PCBs from the paint and degradation within the BTS paste to concentrations ≤ 50 mg/kg;
- Evaluate BTS paste distribution (even thickness after application) using two application methods;

- Evaluate the distribution and application of the sealant using two application methods; and
- Assess the changes in PCB concentration after BTS application.

This SAP presents:

- The scope of work to be conducted to satisfy the project objectives, including a discussion of the rationale (Section 2),
- The methods for sample collection (Section 3);
- The methods for sample analysis (Section 4);
- Experimental controls (Section 5);
- Data quality parameters (Section 6);
- Calibration procedures (Section 7); and
- Data quality indicators (Section 8).

2. SCOPE OF WORK

This section describes the scope of work for the sampling and analysis to be conducted as part of the field demonstration of application of a BTS to remove and degrade PCBs found in painted structures at CCAFS, identifies the data required to meet the objectives listed in Section 1, and presents the schedule to collect the required data.

2.1 Paint Sampling Program

Prior to BTS application to the test surfaces identified in Table 3-22 of the main document, a set of paint scraping samples will be collected from each of the locations. Five samples will be taken from the 10-ft^2 test surfaces and three samples will be collected from the 5-ft^2 test surfaces. This will result in a total of 52 paint samples being collected from pre-treatment locations. After a two-week treatment with the BTS paste, a similar set of post-treatment paint samples will be collected test sectors on the structural sections of material using the cold-scraping method with a metal chisel or equivalent scraping tool.

Samples will be collected from the painted structural sections to:

- Determine baseline and post-demonstration PCB concentrations and during the BTS reactivity phase to help determine the rate at which PCB extraction and degradation occurs ; and
- Evaluate BTS paste distribution on the structural components and evaluate the moisture content (solvent retention).

2.2 BTS Paste Sampling Program

The BTS paste will be applied using a textured spray apparatus like those used for drywall for larger sections or manual applicator on smaller sections. BTS will be applied to a thickness of no less than ¼ inch. Samples will be collected of the BTS after application to the structural sections to:

- Determine baseline and post-demonstration PCB concentrations and during the BTS reactivity phase to help determine the rate at which PCB extraction and degradation occurs ; and
- Evaluate BTS paste distribution on the structural components and evaluate the moisture content (solvent retention).

2.3 Sealant Sampling Program

The sealant will be applied using a high volume, low-pressure spray gun or using the aerosol spray can that the bedliner is sold in. Sealant will be lightly applied in multiple coats so as not to damage the paste's even distribution and to insure that the BTS is completely sealed. Samples will be collected from the sealant after application to the structural sections to:

- Confirm that the PCBs are not transitioned to the sealant from the paint or the BTS; and
- Evaluate the distribution and thickness of the sealant.

2.4 Sampling Program

Paint, BTS and sealant samples will be collected from each of the test sectors that will be set up on the structural components and scrap equipment of the BAAP test area. **Table B-1** lists the parameters that will be analyzed for each type of sample. The rationale for analysis of each of these parameters is summarized below.

<u>PCB and degradation products</u> - will be measured in the paint, the BTS and the sealant during the treatment phase and after removal from the structural sections to evaluate PCB extraction from the paint and degradation in the BTS.

<u>Moisture content </u>– Moisture content of the BTS paste will be evaluated prior to application , during application, after being sealed, during the treatment phase and after it is removed from the structural components to insure sufficient solvent is present in the paste for extraction and degradation of PCBs.

<u>Adhesion of paint</u> – The adhesion of the paint will be tested pre-BTS application and after the BTS and sealant has been removed to evaluate any changes in paint adhesion with BTS application.

<u>Sealant integrity</u> – The sealant will be inspected after application and during the treatment phase to evaluate the sealant is evenly distributed and does not have any holes or cracking. The sealant is integral to keeping sufficient solvent in BTS for it to extract and degrade PCBs.

3. SAMPLE COLLECTION METHODS

This section describes the schedule and procedures for applying the BTS and sealant and collecting and analyzing the sealant, BTS and paint samples. All data collection activities described in this section will be conducted in accordance with the site-specific Health and Safety Plan (Appendix D).

3.1 Sampling Schedule

The sampling schedule for the field demonstration will take place in three phases. The three phases, in order of occurrence, are:

1. Baseline sampling;
2. Performance monitoring (Treatment Phase); and
3. Post-demonstration sampling.

A grid will be established on each of the different structural components for an overall treatment area of 100 ft^2 to allow for the collection of ten pre-deployment samples for baseline sampling. Baseline samples will be collected and analyzed for PCB. Split samples will be analyzed in the NASA and UCF laboratories to independently evaluate the efficacy of the BTS application. The proposed performance monitoring sampling schedule is provided in **Table B-1**. After BTS has been applied and sealed, the performance monitoring phase begins. The amount of BTS applied is a function of the covering rate, and is not a function of the reaction kinetics, with the exception of needing to apply a second coat if degradation is not complete after approximately 3 weeks. Samples will be collected ten days after application and then again at 21 days after application with daily qualitative (visual and manual) evaluation of moisture levels. At the end of three weeks, the PCB concentrations in the paint and BTS paste samples will determine if a second coat of BTS is required. For inactive paste, the BTS will be removed at the end of three weeks and activated in a 55-gallon drum with Mg/Pd in an ethanol solution. If moisture levels decline significantly, additional solvent will be added to the BTS.

The number and location of BTS application area for each test structural sample may change based on the size and shape of the structural samples available for testing, and other site constraints.

3.2 Procedures for Sampling Paint

In accordance with ASTM E1729-05, paint samples will be collected for analysis using the cold-scraping method with a metal chisel or equivalent scraping tool. Because of the potential for collection of metal substrate during the scraping process, a magnet will be used to separate steel from the paint samples prior to weighing. This will be done to ensure the sample masses are not artificially high, leading to false low PCB concentrations.

3.3 Procedures for Sampling BTS and Evaluating Application Thickness and Moisture

BTS paste samples will be collected for analysis by scraping the paste from the treated painted metal surfaces with a rigid plastic or equivalent scraping tool. Care will be taken to insure that the paint is not scraped during the paste sampling and that no paint chips enter the paste sample. This will be confirmed through careful visual observation of both the painted surface and the paste. A small amount of ethanol in a spray wash bottle will be used to wash any remaining BTS from the painted surface and the ethanol wash will be collected and included in the paste sample.

The thickness of the BTS paste will be evaluated using a calibrated probe to measure the thickness of the paste on the surface to be treated. The thickness will be measured at a minimum of three locations across each sector of the treated surface prior to the application of the sealant. The moisture content of the paste will be evaluated qualitatively by manually pressing on the sealed BTS paste surface. The fresh BTS paste will be soft and malleable after application and the moisture content during the course of the treatment phase will be compared with initial texture. The moisture content will be evaluated at a minimum of three locations within each sector of the treated surface.

3.4 Procedures for Sampling Sealant and Evaluating Application Thickness

The sealant samples will be collected for analysis by pealing the sealant off the surface of the BTS and scraping the paste from the underside of the sealant using a scraping tool. Care will be taken to insure that all the BTS is removed from the sealant and does not enter the sealant sample. A small amount of ethanol in a spray wash bottle will be used to wash any remaining BTS from the sealant film.

The thickness of the sealant will be evaluated using a calibrated probe to measure the thickness on the paste on the surface to be treated. The thickness will be measured at a minimum of three locations across each sector of the treated surface before the area is sampled for BTS and paint.

3.5 Sample Labelling, Storage, Packaging and Transportation Procedures

Sample identifiers (IDs) will consist of the sample location name and distance or placement on the structural section (if applicable). Indelible ink pens will be used to label sample containers. In addition to the sample ID, all samples will be labeled with the following information:

- Project name;

- Name or initials of individual collecting the sample;
- Date and time of sample collection; and
- Analysis to be performed.

All samples will be placed in a sample cooler immediately following collection and stored on ice in accordance with the sample handling and custody procedures (see Attachment 2).

4. SAMPLE ANALYSIS

4.1 Analytical Laboratory Methods

Table B-2 identifies the laboratory analytical methods that will be conducted for the BTS, sealant and paint samples, and the associated standard quantitation limits and holding times. Laboratory reports will include results of all sample and quality assurance/quality control (QA/QC) analyses conducted, including any laboratory QC sample analyses (e.g., method blanks, spikes, surrogates).

5. EXPERIMENTAL CONTROLS

5.1 Field QA/QC Controls

Field QA/QC samples consisting of field duplicate samples will be collected to monitor sampling and laboratory analytical performance with respect to groundwater samples.

Field Duplicates: Field duplicates are co-located samples (collected at the same time from the same location using the same sampling procedure) that will be analyzed to evaluate the precision of the sampling and analysis system. Field duplicate samples submitted for laboratory analyses will be submitted without indication of which investigative sample the duplicate represents (i.e., blindly). Field duplicates will be analyzed for all parameters. Field duplicates will be collected and analyzed at a frequency of one field duplicate for every twenty field samples, or for each sampling round.

5.2 Other Controls

Field audits of sampling protocols will be performed to ensure that procedures outlined in this SAP are followed. The audits will include observation of sampling procedures, and review of chain-of-custody documentation and sample results. These audits will be conducted by the QA/QC Officer (Jacqueline Quinn), or her designee, at the Site within the first week of initiating field activities. Following this period, daily audits of field activities and documentation will be conducted via telephone during field activities. A QA/QC checklist (Attachment 1 in Appendix C) will be used to monitor field QA/QC procedures.

Sampling system failures shall be reported by the field personnel directly to the Field Study Leader-QA/QC Officer (Jacqueline Quinn) to specify the type of response action required, the method to evaluate effectiveness of the response action, and the methods for documenting the failure and response action implemented. The Field Leader-QA/QC Officer will review the cause of the failure and determine whether a future change to the sampling method is required.

6. DATA QUALITY PARAMETERS

Data quality objectives (DQOs) are based on the need to monitor the primary data quality indicators (DQIs): precision, bias, accuracy, representativeness, completeness, and comparability (often referred to as PARCC criteria).

- Precision is a measure of agreement among replicate measurements of the same property under prescribed similar conditions.
- Accuracy is the closeness of an individual measurement to the true value. This includes a combination of random error (precision) and systematic error (bias) components that result from sampling and analytical operations.
- Representativeness is the degree to which sample data accurately and precisely represent a characteristic of a population parameter at a sampling point.
- Completeness is a measure of the amount of the valid data obtained from the measurement system compared to the amount that should have been collected.
- Comparability is the confidence with which two data sets can contribute to a common analysis and interpretation.

Precision and bias will be measured by analysis of the field and laboratory QC samples described in Appendix C and comparison of statistics calculated using these results to the acceptance criteria specified. Completeness will be calculated and compared to the acceptance criteria. The representativeness and comparability of the sample results shall be assured through the design of the sampling procedures. In addition, required method detection limits (MDLs) have been established to ensure that a meaningful comparison of the concentration data to performance standards can be completed.

7. CALIBRATION PROCEDURES, QUALITY CONTROL CHECKS, AND CORRECTIVE ACTION

7.1 Calibration Procedures

All instruments will be calibrated as required by the standard USEPA methods or the instrument manufacturer's directions. Calibration verification will be conducted at least once per day or for each analytical run. All field equipment will be calibrated prior to use. Calibration checks using known standard solutions of the analyte of interest will be run as necessary during the day and at the end of each sampling session.

7.2 Waste Management

All solids and fluids generated during application, sampling and equipment cleaning will be containerized by the sampling personnel in approved Department of Transportation (DOT) drums that can be sealed, locked and transported to a designated storage area for characterization, as appropriate, and subsequent proper disposal by KSC in accordance with applicable regulations.

Solid waste such as unusable sampling equipment, gloves, and toweling will be packaged and disposed of according to KSC protocols.

7.3 Data Management

7.3.1 Data Recording

Data generation will be initiated in the field as personnel complete field forms. The field forms will include observations about weather conditions at the Site at the time of sample collection, the results of field measurements and analyses, the volume of materials used and sampled, date and time sampling, and the identification and order of samples collected. Examples of field forms to be used during execution of this SAP are contained in Attachment 1.

These forms shall be transmitted daily to the Field Study Leader-QA/QC Officer, who shall review the forms for clarity, completeness, and conformity with the SAP. The Field Study Leader-QA/QC Officer and field personnel shall communicate on a sufficient frequency in order to review the activities planned and to ensure the completion of the intended scope of work. The field forms shall be filled out as part of the project record.

7.3.2 Data Reduction

Data reduction will be minimal and will consist primarily of tabulating analytical data and field measurement results into electronic spreadsheets or databases. Electronic data transfer from the laboratory, where possible, will be utilized to minimize data transcription error.

7.3.3 Data Validation

Data validation is the process of evaluating data and accepting or rejecting it on the basis of the data quality objectives. The Quality Assurance Project Plan (QAPP; Appendix C) outlines the data validation procedures that will be completed on the data collected.

7.3.4 Data Reporting

The final report from the field demonstration will be the Technical Report, which will include a description of the experimental methods, observations of note, and the results from all chemical analyses in tabulated format. The assigned responsible parties will validate all analytical data, and the original data sheets will be retained with the project records. As appropriate, validated data will be archived using spreadsheet and/or database formats. The data will be stored in a spreadsheet file for further evaluation and calculations. In addition to the above items, the Technical Report will include the identification of outliers, details regarding the corrective actions taken, and a discussion of any necessary deviation from the protocols established in the referenced methods.

8. DATA QUALITY INDICATORS

DQIs for the field demonstration will be based on the analysis of samples collected and analyzed as outlined in Appendix C, the QAPP.

Field QC will consist of sample duplicates collected at a frequency of one field duplicate for every twenty field samples, or for each sampling round. A completeness goal of 80% has been set for all of the critical parameters. Because of the number of sampling events, and the number of samples collected during each sampling event, a completeness of 80% will allow attainment of project objectives.

9. REFERENCES

American Society for Testing and Materials (ASTM). 1997. *Standard Practice for Sampling Waste and Soils for Volatile Organics*. Designation: D 4547-91.

TABLE C-1: ANTICIPATED PERFORMANCE MONITORING SAMPLING SCHEDULE

Application Of A Bimetallic Treatment System (BTS) For PCB Removal From Older Structures On DOD Facilities

	Analyte	Specific Parameter of Interest	Frequency	Rationale/Use
PASTE	PCB and degradation products (1)	PCB, biphenyl	Weekly	PCB extraction from paint and degradation
	Moisture content	Estimate of amount of solvent remaining in the BTS paste	Every 2 days	Insure sufficient solvent present in BTS paste for extraction and degradation of PCBs
PAINT	PCB and degradation products (1)	PCB, biphenyl	Weekly	PCB extraction from paint and degradation
	Adhesion of paint	Adhesion of paint to metal surface post treatment	Post-BTS application and removal	Evalutate any changes in paint adhesion with BTS application
SEALANT	PCB and degradation products (1)	PCB, biphenyl	Weekly	To insure PCB and degradation products are not transferred to sealant
	Sealant integrity	Sealant thicknes and condition	Every 2 days	Sealant is evenly distributed and does not have any holes or cracking. Sealant integral to keeping sufficient solvent in BTS for it to extract and degrade PCBs.

Notes:

1 - This is a destructive analysis so will be done on one section of the test area one a weekly basis until all sections have been analyzed or complete PCB degradation has been confirmed in all components of the system (paint, paste, sealant)

TABLE C-2: SUMMARY OF SAMPLE HANDLING AND LABORATORY ANALYTICAL DETAILS

Application Of A Bimetallic Treatment System (BTS) For PCB Removal From Older Structures On DOD Facilities

	Parameter	Analytical Method	Method Number	Analytical Laboratory (1)	Quantitation Limit	Sample Container	Preservative	Holding Time
PASTE	PCB and degradation products in paste	Gas Chromatography	Modified EPA 8082	UCF/KSC	0.10 mg/kg	plastic bottle for collection/glass for extraction	none	N/A
	Moisture Content of BTS	Visual Inspection Manual Inpection	N/A	N/A	N/A	N/A	N/A	N/A
PAINT	PCB and degradation products in paint	Gas Chromatography	Modified EPA 8082	UCF/KSC	0.10 mg/kg	plastic bottle for collection/glass for extraction	none	n/a
	Adhesion of paint	Field Test of Pull-Off Strength of Coatings	ASTM - D4541-02	N/A	N/A	N/A	N/A	N/A
SEALANT	Sealant integrity	Visual Inspection	NA	NA	NA	NA	NA	NA
	PCB and degradation products in sealant	Gas Chromatography	Modified EPA 8082	UCF/KSC	0.10 mg/kg	plastic bottle for collection/glass for extraction	none	N/A

Notes:

N/A - Not Applicable

UCF - University of Central Florida

KSC - Kennedy Space Center

(1) - 10% of the samples will be sent to a conmerical laboratory (to be determined) for confirmatory analysis.

TBD - To be determined

ATTACHMENT 1

EXAMPLE FIELD FORMS

HEALTH & SAFETY TAILGATE MEETING

GEOSYNTEC CONSULTANTS
130 Research Lane, Suite 2
Guelph, Ontario, Canada N1G 5G3
(519)822-2230 Fax (519)822-3151

Division/Subsidiary ____________________ Facility ____________________
Date ____________________ Time __________ Job Number ____________________
Customer ____________________ Address ____________________
Specific Location ____________________
Type Work ____________________
Chemicals Used ____________________

Safety Topics Presented

Protective Clothing / Equipment ____________________
____
Chemical Hazards ____________________
____
Physical Hazards ____________________
____
Emergency Procedures ____________________
____
Hospital / Clinic ____________________ Phone ( ) ____________________ Paramedic Phone ( ) ____________________
Hospital Address ____________________
Special Equipment ____________________
Other ____________________

Attendees

Name Printed	Signature

Meeting Conducted By

____________________ ____________________
Name Printed Signature

Supervisor ____________________ Manager ____________________

INCIDENT REPORT

GEOSYNTEC CONSULTANTS
130 Research Lane, Suite 2
Guelph, Ontario, Canada N1G 5G3
(519)822-2230 Fax (519)822-3151

Project Name *

Project Number *

Date *

Site Health & Safety Officer *

Employee Name *

Company *

Job Title *

Type of Incident: *
*
*

Location of Incident: *
*
*

Specific Job at Time of Incident: *
*

Level of Protection Worn at Time of Incident: *
*
*
*

Summary of What Occured: *
*
*
*
*
*

Actions Taken to Correct Situation (PPE, etc .): *
*
*
*
*
*

Medical Care Administered: *
*
*
*

Employee Signature: Date: *

Site Health & Safety Officer Signature: Date: *

HEALTH & SAFETY AIR QUALITY MONITORING

GeoSyntec Consultants
130 Research Lane, Suite 2
Guelph, Ontario, Canada N1G 5G3
(519)822-2230 Fax (519)822-3151

Borehole ID ____________________
Project Name ____________________
Project Number ____________________
Site Location ____________________
Field Personnel ____________________
Recorded By ____________________
Date ____________________

Wind Direction ____________________
PID Model Number ____________________
Serial # ____________________
Combustible Gas Meter Model ____________________
Serial # ____________________
Oxygen Meter Model ____________________
Serial # ____________________

Time	Location / Depth	PID Reading (ppmV)	Combustible Gas Reading (%LEL)	Oxygen Reading (%)	Comments

FIELD CHECK LIST

GEOSYNTEC CONSULTANTS
130 Research Lane, Suite 2
Guelph, Ontario, Canada N1G 5G3
(519)822-2230 Fax (519)822-3151

Project Name ____________________ Site Contact ____________________

Project Number ____________________ Phone Number ____________________

Site History
- ☐ Site map
- ☐ Borehole logs
- ☐ Well Construction Details
- ☐ Water sampling and purging records

Supplies
- ☐ Permanent markers
- ☐ Pens, pencils, ruler, note book
- ☐ Packing tape
- ☐ Calculator
- ☐ Lighter
- ☐ Camera (if digital -disks, battery charger)

Personal Protective Equipment
- ☐ Hard hat
- ☑ Safety boots
- ☐ Work gloves
- ☐ Disposable gloves
- ☐ Safety glasses
- ☐ Hearing protection
- ☐ Cellular phone (office contact list)
- ☐ Fire resistent coveralls
- ☐ ____________ other PPE (ie. tyvek suits)
- ☐ Map to nearest medical facility

Site Safety Equipment
- ☑ First Aid Kit
- ☐ Fire Exinguisher
- ☑ HASP
- ☐ LEL meter
- ☑ OSHA - Regulations for Construction Projects

Monitoring Equipment
- ☐ Water level meter
- ☐ PID (Span Gas)
- ☐ FID (Standards)
- ☐ ORP electrode meter (Std. Solutions)
- ☐ DO meter (2 Std. Solutions)
- ☐ pH meter (3 Std. Solutions (4,7,10))
- ☐ Specific Conductance Meter (Std. Solutions)
- ☐ Thermometer
- ☐ Container for Std. Solutions (ie. Paper cups)
- ☐ Bailer

Tools
- ☐ Well Cracking Devices
- ☐ Site Specific Keys
- ☐ Locks
- ☐ Bolt Cutters
- ☐ Tool Box
- ☐ Flash Light

Field Forms
- ☐ Daily Field Report/Project Field Activity Logbook
- ☐ Meter Calibration Report
- ☐ Sample/Core Log of Boring
- ☐ Soil Head Space Measurements
- ☐ Well Construction
- ☐ Well Inspection
- ☐ Water Level Data
- ☐ Corrective Action Forms
- ☐ Incident Report
- ☐ Pump Recovery Test
- ☐ Groundwater Extraction and Treatment
- ☐ Monitoring Well Development/Purging & Sampling Records
- ☐ Laboratory Chain of Custody Forms
- ☐ Courier Way Bills (Fed-Ex Acct. # 2327-8603-5)
- ☐ ____________________

Decontamination Supplies
- ☐ deionized water
- ☐ distilled water
- ☐ alconox
- ☐ paper towels
- ☐ garbage bags
- ☐ squirt bottles
- ☐ methanol
- ☐ 10% nitric acid

Soil Sample Supplies
- ☐ mason jars
- ☐ tin foil
- ☐ saran wrap
- ☐ waste disposal (ie.drums)
- ☐ zip lock bags
- ☐ blue ice

Water Sample Supplies
- ☐ waterra tubing
- ☐ valves foot
- ☐ buckets
- ☐ flow through cell/beakers
- ☐ field filters
- ☐ waste disposal (ie. drum)
- ☐ zip lock bags
- ☐ blue ice

Laboratory Samples

Parameters	Bottles	Preservative

Are equipment blanks required? ☐ Yes ☐ No
Are field duplicates required? ☐ Yes ☐ No
Is a trip blank required? ☐ Yes ☐ No

Project Specific Notes:

DRUM TRACKING

GEOSYNTEC CONSULTANTS
130 Research Lane, Suite 2
Guelph, Ontario, Canada N1G 5G3
(519)822-2230 Fax (519)822-3151

Project Name ______________________
Project Number ______________________
Date ______________________
Field Personnel ______________________
Recorded By ______________________

Comments ______________________

Drum Number	Accum. Date	Waste Type	Depth Interval	Corresponding GeoSyntec Sample #	Date GeoSytnec Sample Analyzed	Waste Classification	Date of Transfer

DAILY FIELD REPORT

GEOSYNTEC CONSULTANTS
130 Research Lane, Suite 2
Guelph, Ontario, Canada N1G 5G3
(519)822-2230 Fax (519)822-3151

Project Name: ______________________ Date: ______________________ Page ___ of ___
Project Number: ______________________ Primary Activities: ______________________
Field Personnel: ______________________
____
Recorded By: ______________________
Weather: ______________________

Time	Description of activities - location of work, work performed, equipment & personnel used, incidental information

Daily Field Report

CORRECTIVE ACTION FORM

GEOSYNTEC CONSULTANTS
130 Research Lane, Suite 2
Guelph, Ontario, Canada N1G 5G3
(519) 822-2230 Fax (519) 822-3151

Project Name ____________________

Page ____ of ____

Project Number ____________________

Date ____________________

Initiator's Name & Title ____________________

Recorded By ____________________

Reviewed and Implemented by:

Project Manager ____________________

QA Manager ____________________

QA Officer ____________________

Problem Description

Corrective Action

ATTACHMENT 2

STANDARD FIELD PROCEDURES

Attachment 2 – Standard Field Procedures

LIST OF CONTENTS

EQUIPMENT DECONTAMINATION PROCEDURES

This document describes the procedures to be followed during the cleaning of non-disposable sampling and other field equipment. All sampling equipment shall be cleaned prior to arrival at the Site. Equipment will be free of petroleum oils and greases and any other substances that may compromise the chemical integrity of the structure surfaces or the samples that will be collected. All equipment will be stored in a clean location after cleaning. Where practicable, smaller pieces of equipment, such as chisels, probes and beakers, will be either wrapped in aluminum foil or sealed in a plastic bag following cleaning. Fluids generated during equipment decontamination shall be containerized in approved DOT drums that can be sealed and locked for later disposal. Care shall be taken to ensure that the fluids do not flow overland into nearby surface water bodies, sewer systems, or ditches.

1. Field Sampling Equipment

Non-dedicated sampling equipment (e.g., chisels, paint scrapers, sub sampling equipment, beakers) will be manually cleaned prior to initial use, after each use, and at the completion of sampling activities according to the following procedure.

- Manually scrub with a phosphate-free (e.g., Alconox™) soap solution.
- Rinse with distilled water.
- Rinse with reagent-grade isopropanol.
- Rinse thoroughly with distilled water.
- Allow equipment to air dry.

SAMPLE DESIGNATION, CUSTODY AND HANDLING PROCEDURES

This document describes the procedures to be followed for sample designation, handling, custody, and documentation.

1. Sample Designation

The laboratory will provide sample containers having a self-adhesive, non-removable label affixed to each sample container. Sample labels will contain the following information:

- Sample ID;
- Project name;
- Name or initials of individual collecting the sample;
- Date and time of sample collection
- Analysis to be performed; and
- Preservation chemical (if used).

Field duplicate or replicate samples will require special procedures for sample designation to ensure that they will be submitted blindly to the laboratory. The sample location will not be included in the sample ID and the time of collection will be fictitious. Both the fictitious and actual information will be documented in the field records.

2. Sample Handling and Storage

Each sample will be sealed in a separate plastic bag following collection. Samples will then be stored in an insulated cooler. Samples selected for laboratory analysis will be transferred to insulated coolers for overnight shipment to the laboratory. Each cooler will be packed in a manner that will prevent damage to sample containers during shipment. A chain-of-custody form (discussed below) will accompany each cooler.

3. Sample Custody and Documentation

Chain-of-custody forms will be used to trace the possession and handling of all samples, from their collection, through analysis, until their final disposition. These forms will document the names of the relinquishing and receiving parties, the time and date of the transfer of custody, and the reason for the transfer of custody. One chain-of-custody form will accompany each cooler shipped to the laboratory. The form will be placed in a sealed plastic bag inside the cooler. Shipping of samples to the laboratory will be accomplished by Federal Express, or equivalent, overnight service. Samples will remain in the custody of the sampling team until custody is relinquished to the courier service that will transfer the samples to the laboratory. Each sample shipment will be tracked via the courier waybill number to ensure that prompt delivery of the shipment to the laboratory has occurred.

Upon receipt by the Sample Custodian for the laboratory, the Sample Custodian will note on the form whether any damaged sample containers or discrepancies between the sample label and information on the form, and sign and date the form. A copy of the chain-of-custody form will then be transmitted to the Field Study Leader-QA/QC Officer for their records and so that proper action can be taken, if necessary.

Appendix D: Sampling Results

TABLE D-1
PRE/POST-TREATMENT CONCENTRATIONS FROM VARIOUS TEST PANELS

Pre-Treatment Concentrations in Paint		**Post Treatment Concentrations and % Removal in Paint**				**Post Treatment Concentrations in Paste**
Sample ID	**Conc (mg PCB/kg)**	**Application Time**	**Conc (mg PCB/kg)**	**% Removal**	**Active (Y/N)**	**Conc (mg PCB/kg)**
Sample 3-7	94	Week 1	33	65%	N	0.41
Sample 3-8	102	Week 1	31	70%	N	0.46
Sample 5-7	13	Week 1	7	48%	N	0.65
Sample 8-5	72	Week 1	16	78%	Y	--
Sample 8-6	65	Week 1	16	76%	Y	--
Sample 3-9	80	Week 2	14	82%	N	0.53
Sample 3-10	89	Week 2	14	84%	N	0.43
Sample 5-8	12	Week 2	6	46%	N	0.92
Sample 5-9	12	Week 2	6	52%	N	0.52
Sample 8-6	--	Week 2	27	58%	Y	--
Sample 8-7	70	Week 2	24	66%	Y	--
Sample 1-1	40	Week 3	13	67%	Y	--
Sample 1-2	31	Week 3	13	58%	Y	--
Sample 1-3	33	Week 3	BDL	100%	Y	--
Sample 2-1	47	Week 3	12	74%	Y	--
Sample 2-2	43	Week 3	25	42%	Y	--
Sample 2-3	44	Week 3	34	24%	Y	--
Sample 3-1	72	Week 3	2	97%	N	--
Sample 3-2	66	Week 3	2	98%	N	--
Sample 3-3	72	Week 3	3	96%	N	--
Sample 3-4	71	Week 3	3	96%	N	--
Sample 3-5	87	Week 3	2	98%	N	--
Sample 3-6	86	Week 3	2	98%	N	--
Sample 3-11	80	Week 3	1	98%	N	0.59
Sample 3-12	74	Week 3	2	98%	N	0.61
Sample 4-1	37	Week 3	BDL	100%	Y	--
Sample 4-2	41	Week 3	10	76%	Y	1.38
Sample 4-3	51	Week 3	17	67%	Y	1.38
Sample 4-4	35	Week 3	15	57%	Y	--
Sample 4-5	37	Week 3	10	73%	Y	1.67
Sample 5-1	58	Week 3	8	87%	N	1.29
Sample 5-2	59	Week 3	7	88%	N	--
Sample 6-1	49	Week 3	BDL	100%	Y	--
Sample 6-2	50	Week 3	12	76%	Y	--
Sample 6-3	23	Week 3	13	44%	Y	<0.1
Sample 6-4	25	Week 3	BDL	100%	Y	--
Sample 6-5	23	Week 3	13	46%	Y	<0.1
Sample 6-6	23	Week 3	BDL	100%	Y	--
Sample 6-9	25	Week 3	BDL	100%	Y	--
Sample 6-10	26	Week 3	BDL	100%	Y	--
Sample 6-11	23	Week 3	BDL	100%	Y	--
Sample 6-12	36	Week 3	12	65%	Y	<0.1
Sample 7-1	73	Week 3	15	79%	Y	0.25
Sample 7-2	72	Week 3	13	83%	Y	0.28
Sample 7-3	69	Week 3	12	82%	Y	0.19
Sample 8-1	73	Week 3	37	49%	N	--
Sample 8-7	--	Week 3	10	85%	Y	<0.1
Sample 8-8	50	Week 3	10	80%	Y	--
Sample 5-3	50	Week 4	13	75%	N	--
Sample 5-4	43	Week 4	12	73%	N	--

Notes:

*Test sample of inactive BTS left uncoated and checked for removal capability

% - percent

-- sample not taken

BDL - below detection limit

Conc - concentration

mg PCB/kg - milligrams of PCB per kilogram

N - no

PCB - Polychlorinated biphenyls

Y - yes

TABLE D-2
CONCENTRATION OF PCBs IN SAMPLES FROM BUILDING 6810-11

BUILDING 6810-11 PAINT									BUILDING 6810-11 PASTE		
Pre-Treatment					Post Treatment				Post Treatment		
Sample ID Location	Active (Y/N)	Conc (mg/kg)	TR AVG	STD DEV	Conc (mg/kg)	TR AVG	STD DEV	Average % Removal	Conc (mg/kg)	TR AVG	STD DEV
NM VP 1 ft	N	1,824	1,888	90	99	96	5	95%	32	32	1
NM VP 1 ft	N	1,951			92				33		
--	--	--			--				32		
MgPd sil 1 ft	Y	2,153	2,111	60	93	99	8	95%	18	13	7
MgPd sil 1 ft	Y	2,069			104				5		
--	--	--			--				17		
MgPd VP 1ft	Y	3,049	2,793	363	175	185	13	93%	25	23	2
MgPd VP 1ft	Y	2,536			194				21		
--	--	--			--				22		
NM VP 1 ft	N	3,884	3,262	881	352	167	38	95%	42	56	13
NM VP 1 ft	N	2,639			298				66		
--	--	--			--				60		
MgPd sil 1ft	Y	1,498	1,420	110	95	97	3	93%	23	23	2
MgPd sil 1ft	Y	1,343			99				25		
--	--	--			--				21		
MgPd VP 3 ft. A	Y	--	2,545	229	198	218	58	91%	19	36	11
MgPd VP 3 ft. A	Y	--			188				41		
MgPd VP 3 ft. A	Y	--			197				35		
MgPd VP 3 ft. B	Y	2,481			--				34		
MgPd VP 3 ft. B	Y	2,620			--				37		
MgPd VP 3 ft. C	Y	2,652			285				51		
MgPd VP 3 ft. C	Y	2,903			322				--		
MgPd VP 3 ft. C	Y	--			267				--		
MgPd VP 3 ft. D	Y	2,302			152				--		
MgPd VP 3 ft. D	Y	2,314			176				--		
MgPd VP 3 ft. D	Y	--			177				--		
NM VP 3 ft. A	N	2,224	2,138	201	--	169	56	92%	187	162	60
NM VP 3 ft. A	N	2,411			--				205		
NM VP 3 ft. B	N	1,782			270				93		
NM VP 3 ft. B	N	--			223				--		
NM VP 3 ft. B	N	--			208				--		
NM VP 3 ft. C	N	2,053			127				--		
NM VP 3 ft. C	N	2,110			110				--		
NM VP 3 ft. C	N	--			125				--		
NM VP 3 ft. C	N	--			92				--		
NM VP 3 ft. D	N	2,291			186				--		
NM VP 3 ft. D	N	2,093			175				--		
NM VP 3 ft. D	N	--			174				--		

TABLE D-2
CONCENTRATION OF PCBs IN SAMPLES FROM BUILDING 6810-11

BUILDING 6810-11 PAINT									BUILDING 6810-11 PASTE		
Pre-Treatment					**Post Treatment**				**Post Treatment**		
Sample ID Location	**Active (Y/N)**	**Conc (mg/kg)**	**TR AVG**	**STD DEV**	**Conc (mg/kg)**	**TR AVG**	**STD DEV**	**Average % Removal**	**Conc (mg/kg)**	**TR AVG**	**STD DEV**
MgPd sil 3 ft. A	Y	1,830	2,074	329	286	238	86	89%	66	61	7
MgPd sil 3 ft. A	Y	2,774			274				64		
MgPd sil 3 ft. A	Y	--			270				54		
MgPd sil 3 ft. B	Y	2,178			223				--		
MgPd sil 3 ft. B	Y	2,221			204				--		
MgPd sil 3 ft. B	Y	--			222				--		
MgPd sil 3 ft. C	Y	1,799			137				--		
MgPd sil 3 ft. C	Y	2,086			152				--		
MgPd sil 3 ft. C	Y	--			137				--		
MgPd sil 3 ft. C	Y	--			122				--		
MgPd sil 3 ft. D	Y	1,820			366				--		
MgPd sil 3 ft. D	Y	1,887			361				--		
MgPd sil 3 ft. D	Y	--			340				--		
MgPd VP 1ft	Y	2,165	2,862	986	258	251	9	91%	62	63	19
MgPd VP 1ft	Y	3,559			241				82		
MgPd VP 1ft	Y	--			253				45		

Notes:

Upper case letter (A,B, C, D) indicates that multiple areas (collected into separate containers) were sampled within the sample treatment area.

% - percent

-- sample not taken

Conc - concentration

mg/kg - milligrams per kilogram

Mg/Pd - magnesium/palladium

N - no

NM - no metal (inactive paste)

PCB - Polychlorinated biphenyls

SIL - silcone sealant

STD DEV - standard deviation of data set

TR AVG - Treatment Average: three to four samples were taken from the same sample container, extracted separately and analyzed, these number

VP - vinyl polymer sealant

wks - weeks

Y - yes

TABLE D-3
CONCENTRATION OF PCBs IN CONCRETE IN BUILDING 6810-11 AFTER BTS TREATMENT

BUILDING 6810-11 CONCRETE								
Pre-Treatment					Post Treatment			
Sample ID	Active (Y/N)	Conc (mg/kg)	TR AVG	STD DEV	Conc (mg/kg)	TR AVG	STD DEV	Average % Removal
NM VP 3ft concrete A	N	43	48	6	40	42	2	13%
NM VP 3ft concrete A	N	55			41			
NM VP 3ft concrete A	N	45			44			
NM VP 3ft concrete D	N	38	35	4	22	24	3	31%
NM VP 3ft concrete D	N	36			28			
NM VP 3ft concrete D	N	30			25			
NM VP 3ft concrete D	N	--			22			
MgPd sil 3ft concrete	Y	28	30	6	13	13	1	55%
MgPd sil 3ft concrete	Y	36			13			
MgPd sil 3ft concrete	Y	25			14			

Notes:

Upper case letter (A,D) indicates that multiple areas (collected into separate containers) were sampled within the sample treatment area.
AVG - three to four samples were taken from the same sample container, extracted separately and analyzed, these numbers are averaged.
BTS - Bimetallic Treatment System
STD DEV - standard deviation of data set.
NM - no metal (inactive paste)
mg/kg - milligrams per kilogram
Mg/Pd - magnesium/palladium
VP - vinyl polymer sealant
SIL - silcone sealant

TABLE D-4
PCB CONCENTRATIONS IN PAINT FROM BUILDING 6810-36

BUILDING 6810-36 PAINT									BUILDING 6810-36 PASTE			
Pre-Treatment					Post Treatment				Post Treatment			
Sample ID	Active (Y/N)	Conc (mg/kg)	TR AVG	STD DEV	Conc (mg/kg)	TR AVG	STD DEV	Average % Removed	Treatment Time (wks)	Conc (mg/kg)	TR AVG	STD DEV
NM VP 3 ft A	N	391	566	190		98	4	83%	1	59	56	4
NM VP 3 ft A	N	414							1	53		
NM VP 3 ft D	N	745			94				--	--		
NM VP 3 ft D	N	714			102				--	--		
NM VP 3 ft D	N	--			98				--	--		
MgPd sil 3 ft A	Y	855	789	108		82	21	90%	1	44	43	13
MgPd sil 3 ft A	Y	854			82				1	56		
MgPd sil 3 ft B	Y	908			112				1	29		
MgPd sil 3 ft B	Y	817							--	--		
MgPd sil 3 ft C	Y	590			66				--	--		
MgPd sil 3 ft C	Y	684			69				--	--		
MgPd sil 3 ft D	Y	741							--	--		
MgPd sil 3 ft D	Y	861							--	--		
MgPd VP 1 ft	Y	620	595	35	40	40	0	93%	1	39	40	6
MgPd VP 1 ft	Y	570			40				1	35		
--	--	--			--				1	47		
NM VP 1 ft	N	900	911	15	43	45	3	95%	2	114	95	18
NM VP 1 ft	N	921			47				2	78		
NM VP 1 ft	N				46				2	92		
MgPd sil 1 ft	Y	810	812	38	75	83	8	90%	2	28	25	12
MgPd sil 1 ft	Y	850			86				2	36		
MgPd sil 1 ft	Y	775			89				2	12		
MgPd VP 1 ft	Y	580	547	35	32	37	5	93%	2	20	39	17
MgPd VP 1 ft	Y	510			39				2	46		
MgPd VP 1 ft	Y	550			41				2	52		
NM VP 1 ft	N	780	815	49	115	110	7	87%	3	147	152	8
NM VP 1 ft	N	850			105				3	158		
MgPd sil 1 ft	Y	840	815	35	132	141	12	83%	3	57	50	11
MgPd sil 1 ft	Y	790			149				3	37		
--	--	--			--				3	56		

TABLE D-4
PCB CONCENTRATIONS IN PAINT FROM BUILDING 6810-36

BUILDING 6810-36 PAINT									BUILDING 6810-36 PASTE			
Pre-Treatment					Post Treatment				Post Treatment			
Sample ID	Active (Y/N)	Conc (mg/kg)	TR AVG	STD DEV	Conc (mg/kg)	TR AVG	STD DEV	Average % Removed	Treatment Time (wks)	Conc (mg/kg)	TR AVG	STD DEV
MgPd VP 3 ft A	Y	542	542	24	56	40	14	93%	3	52	34	17
MgPd VP 3 ft A	Y	508			55				3	18		
MgPd VP 3 ft A	Y				55				3	33		
MgPd VP 3 ft B	Y	--		--	26				--	--		
MgPd VP 3 ft B	Y	--		--	29				--	--		
MgPd VP 3 ft B	Y	--		--	26				--	--		
MgPd VP 3 ft C	Y	402		11	50				--	--		
MgPd VP 3 ft C	Y	387			52				--	--		
MgPd VP 3 ft C	Y				43				--	--		
MgPd VP 3 ft D	Y	724		25	25				--	--		
MgPd VP 3 ft D	Y	689			28				--	--		

Notes:

Upper case letter (A,B, C, D) indicates that multiple areas (collected into separate containers) were sampled within the sample treatment area.
% - percent
-- sample not taken
Conc - concentration
mg/kg - milligrams per kilogram
Mg/Pd - magnesium/palladium
N - no
NM - no metal (inactive paste)
PCB - Polychlorinated biphenyls
SIL - silcone sealant
STD DEV - standard deviation of data set
TR AVG - Treatment Average: three to four samples were taken from the same sample container, extracted separately and analyzed, these num
VP - vinyl polymer sealant
wks - weeks
Y - yes
Sealant failed almost immediately and paste dried out

TABLE D-5
PCB CONCENTRATIONS IN CONCRETE AT BUILDING 6810-36, PRE POST TREATMENT

BUILDING 6810-36 CONCRETE									
Pre-Treatment					Post Treatment				
Sample ID	Active (Y/N)	Conc (mg/kg)	TR AVG	STD DEV	Treatment Time (wks)	Conc (mg/kg)	TR AVG	STD DEV	Average % Removal
MgPd Sil 1wk concrete	Y	85	139	49	1	17	18	3	87%
MgPd Sil 1wk concrete	Y	110			1	16			
MgPd Sil 1wk concrete	Y	183			1	23			
MgPd Sil 1wk concrete	Y	178			1	17			
MgPd VP Concrete	Y	50	59	18	3	10	20	10	66%
MgPd VP Concrete	Y	60			3	37			
MgPd VP Concrete	Y	43			3	16			
MgPd VP Concrete	Y	39			3	15			
MgPd VP Concrete	Y	75			3	28			
MgPd VP Concrete	Y	84			3	14			

Notes:

AVG - four to six samples were taken from the same sample container, extracted separately and analyzed, these numbers are averaged.
Conc - concentration
mg/kg - milligrams per kilogram
Mg/Pd - magnesium/palladium
SIL - silcone sealant
STD DEV - standard deviation of data set.
TR AVG - treatment average
VP - vinyl polymer sealant
wks - weeks
Y - yes
N - no

TABLE D-6
CONCENTRATIONS IN PAINT AND PASTE SAMPLED FROM BUILDING 6657-02

6657-02 PAINT										6657-02 PASTE		
Pre-Treatment					Post Treatment					Post Treatment		
Sample ID	Active (Y/N)	Conc (mg/kg)	TR AVG	STD DEV	Treatment Time (wks)	Conc (mg/kg)	TR AVG	STD DEV	Average % Removal	Conc (mg/kg)	TR AVG	STD DEV
LARGE TANK												
NM VP 1ft	N	45,739	42,660	2,690	1	20,676	19,824	1,205	54%	284	298	61
NM VP 1ft	N	41,480			1	18,972				245		
NM VP 1ft	N	40,762			1					364		
MgPd VP 1ft	Y	33,863	36,173	2,080	1	15,118	11,788	4,709	67%	526	688	164
MgPd VP 1ft	Y	37,898			1	8,458				853		
MgPd VP 1ft	Y	36,759			1					684		
NM VP 1ft	N	34,607	33,263	1,901	2	14,238	14,016	415	58%	1,622	1,554	206
NM VP 1ft	N	31,918			2	13,538				1,303		
NM VP 1ft	N				2	14,273				1,793		
--	--				--					1,499		
MgPd VP 1ft	Y	27,671	43,046	13,497	2	13,066	15,825	2,875	63%	1,339	1,222	118
MgPd VP 1ft	Y	52,941			2	15,605				1,173		
MgPd VP 1ft	Y	48,526			2	18,804				1,159		
--	--				--					1,352		
--	--				--					1,085		
NM VP A 3ft	N	34,127	33,879	1,951	3	8,326	6,576	1,698	81%	2,054	1,781	386
NM VP A 3ft	N	35,695			3	5,827				1,508		
NM VP A 3ft	N	31,816			3	7,284				--		
NM VP A 3ft	N				3	7,447				--		
NM VP A 3ft	N				3	3,998				--		
NM VP B 3ft	N	36,523	28,051	11,981	3	10,632	11,503	1,232	59%	--		
NM VP B 3ft	N	19,579			3	12,375				--		
NM VP C 3ft	N	40,980	36,055	4,471	3	16,522	16,907	545	53%	--		
NM VP C 3ft	N	32,251			3	17,293				--		
NM VP C 3ft	N	34,934			3					--		
MgPd VP A 3ft	Y	33,234	35,303	2,926	3	22,705	21,536	2,121	39%	642	716	104
MgPd VP A 3ft	Y	37,372			3	19,088				789		
MgPd VP A 3ft	Y				3	22,814				--		
MgPd VP B 3ft	Y	33,107	35,532	3,430	3	14,433	14,847	919	58%	--		
MgPd VP B 3ft	Y	37,957			3	15,900				--		
MgPd VP B 3ft	Y				3					--		
MgPd VP C 3ft	Y	24,989	27,548	3,619	3	14,209				--		
MgPd VP C 3ft	Y	30,107			3					--		

TABLE D-6
CONCENTRATIONS IN PAINT AND PASTE SAMPLED FROM BUILDING 6657-02

6657-02 PAINT										6657-02 PASTE		
Pre-Treatment					Post Treatment					Post Treatment		
Sample ID	Active (Y/N)	Conc (mg/kg)	TR AVG	STD DEV	Treatment Time (wks)	Conc (mg/kg)	TR AVG	STD DEV	Average % Removal	Conc (mg/kg)	TR AVG	STD DEV
LARGE TANK												
MgPd LID VP	Y	24,111	27,154	4,304	3	8,522	10,605	2,947	61%	111	141	34
MgPd LID VP	Y	30,197			3	12,689				178		
										134		
SMALL TANK												
MgPd sil A 3ft	Y	21,130	20,089	1,028	1	8,666	8,687	434	57%	218	179	83
MgPd sil A 3ft	Y	19,676			1					84		
MgPd sil B 3ft	Y	20,097		288	1	9,131				235		
MgPd sil B 3ft	Y	19,689			1					--		
MgPd sil C 3ft	Y	21,242		1,798	1	8,263				--		
MgPd sil C 3ft	Y	18,698			1					--		
MgPd sil 1ft	Y	24,602	28,970	6,176	2	11,288	12,585	1,835	57%	390	464	69
MgPd sil 1ft	Y	33,337			2	13,882				475		
--	--				--					526		
MgPd sil 1ft	Y	15,728	15,319	1,201	3	9,441	10,053	4,422	34%	682	605	114
MgPd sil 1ft	Y	13,967			3	5,970				474		
MgPd sil 1ft	Y	16,263			3	14,749				658		

Notes:

Upper case letter (A, B, C) indicates that multiple areas (collected into separate containers) were sampled within the sample treatment area
% - percent
-- sample not taken
Conc - concentration
mg/kg - milligrams per kilogram
Mg/Pd - magnesium/palladium
N - no
NM - no metal (inactive paste)
PCB - Polychlorinated biphenyls
SIL - silcone sealant
STD DEV - standard deviation of data set
TR AVG - Treatment Average: three to four samples were taken from the same sample container, extracted separately and analyzed, these
VP - vinyl polymer sealant
wks - weeks
Y - yes

TABLE D-7
PCB CONCENTRATIONS IN PAINT TAKEN FROM EQUIPMENT IN THE STAGING AREA

STAGING AREA PAINT										STAGING AREA PASTE		
Pre-Treatment						Post Treatment				Post Treatment		
Sample ID	Treatment Time (wks)	Active (Y/N)	Conc (mg/kg)	TR AVG	STD DEV	Conc (mg/kg)	TR AVG	STD DEV	Average % Removed	Conc (mg/kg)	TR AVG	STD DEV
MgPd VP press #4		Y	85	93.0	10.8							
MgPd VP press #4		Y	101									
MgPd VP press #4	1	Y				17	13	3	85%	2.9	2.2	0.7
MgPd VP press #4	1	Y				13				2.3		
MgPd VP press #4	1	Y				10				1.5		
MgPd VP press #4	3	Y				6	7	0.5	93%	1.6	1.1	0.1
MgPd VP press #4	3	Y				7				1.4		
MgPd VP press #12		Y	156	160.3	5.9							
MgPd VP press #12		Y	164									
MgPd VP press #12	1	Y				3	3	0	98%		0.3	0.0
MgPd VP press #12	1	Y				3				0.3		
MgPd VP press #12	1	Y				3				0.3		
MgPd VP press #12	3	Y				4	4	0.1		0.4	0.4	0.0
MgPd VP press #12	3	Y				3			98%	0.4		
MgPd VP press #14		Y	8	8.7	0.9							
MgPd VP press #14		Y	9									
MgPd VP press #14	1	Y				3	2	1	75%	0.4	0.5	0.1
MgPd VP press #14	1	Y				2				0.6		
MgPd VP press #14	1	Y				2				0.5		
MgPd VP press #14	3	Y				12	13	0.7	-43%	0.3	0.2	0.0
MgPd VP press #14	3	Y				13				0.2		
MgPd VP press #15		Y	8	5.9	3.7							
MgPd VP press #15		Y	3									
MgPd VP press #15	1	Y				1				0.2	0.2	0.0
MgPd VP press #15	1	Y				bdl				0.2		
MgPd VP press #15	3	--								0.1	0.1	0.0
MgPd VP press #15	3	Y				bdl				0.1		
MgPd VP press #15	3	Y				bdl				0.1		
MgPd VP press #36		Y	bdl									
MgPd VP press #36		Y	bdl									
MgPd VP press #36	1	Y				bdl				0.1	0.1	0.0
MgPd VP press #36	1	Y				bdl				0.1		
MgPd VP press #36	3	Y				bdl				0.1	0.1	0.0
MgPd VP press #36	3	Y				bdl				0.1		

Notes:

Upper case letter (A, B, C, D) indicates that multiple areas (collected into separate containers) were sampled within the sample treatment area.

AVG - two to five samples were taken from the same sample container, extracted separately and analyzed, these numbers are averaged.

bdl - below detection limit

STD DEV - standard deviation of data set.

mg/kg - milligrams per kilogram

Mg/Pd - magnesium/palladium

VP - vinyl polymer sealant

Appendix E: Quality Assurance Project Plan

APPENDIX E

QUALITY ASSURANCE PROJECT PLAN

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LIST OF ATTACHMENTS

1. PURPOSE AND SCOPE OF THE PLAN

Geosyntec Consultants, Inc. (Geosyntec) has prepared this Quality Assurance Project Plan (QAPP) for the Environmental Security Technology Certification Program (ESTCP) to evaluate the application of a Bimetallic Treatment System (BTS) to remove and degrade PCBs found on Department of Defense (DoD) facilities. The field portion of the Dem/Val addressed in this report will be conducted at the Badger Army Ammunition Plant (BAAP), Saulk County, Wisconsin. This site has had PCBs identified in the paint on the structures and scrap equipment, appropriate site conditions and a suitable on-site support network for execution of the Dem/Val. Baseline characterization and performance monitoring events are planned for April 2008 through July 2008, and will be conducted to meet the following field demonstration objectives:

1. Determine the protocol for formulating BTS for site-specific conditions to enhance applicability to various PCB-containing materials found across numerous DoD facilities while maximizing safety and efficacy with the ultimate goal of reducing PCB concentrations to less than 50 mg/kg.
2. Demonstrate the effectiveness of BTS on a wide range of actual contaminated structures at three DoD facilities. The relationships between dose applied, repeated applications and reaction kinetics will be evaluated with the intention of specifically identifying the factors influencing treatment and limiting reaction rates for a specific media (e.g. different painted structures). Environmental condition effects (temperature and humidity; weathering) and impact of BTS on material appearance and adhesion will also be evaluated.
3. Review BTS application and handling process and develop appropriate on-site safety protocols for institutions to implement during its application, including the handling of any site-specific waste products generated by BTS and the application process. The BTS process itself produces non-toxic waste.
4. Develop full-scale cost and performance reports for DoD facility end-users to utilize when addressing PCBs found on existing structures.

The following sections describe the Quality Assurance (QA) procedures that will serve to ensure that the integrity of collected data is not compromised, and specify the QA responsibilities.

The QAPP is produced to document how QA and quality control (QC) activities will be implemented during the life cycle of a project. The QAPP will dictate how QA/QC procedures for sampling and analysis of paint, BTS paste, sealant and PCBs will be implemented.

2. QUALITY ASSURANCE RESPONSIBILITIES

Staff from Geosyntec, Kennedy Space Center (KSC), Naval Facilities Engineering Service Center (NFESC) and the University of Central Florida (UCF) will perform routine field sampling and analyses. The project team will consist of a Project Manager, Field Study Leader, QA/QC Officer, Project Technical Staff, and Analytical Laboratories. Their responsibilities are outlined in the following sections.

2.1 Project Manager

The primary function of the Project Manager will be to ensure that all of the activities planned are consistent with the overall strategy of the project, appropriate resources (human, technological) are employed, the scope, schedule and budget for activities are set consistent with project requirements, and progress on project activities is occurring at the appropriate rate to meet the scheduling and budget requirements. Suzanne O'Hara of Geosyntec and Nancy Ruiz of NFESC will perform the duties of the Project Manager.

2.2 Field Study Leader

The Field Study Leader will be responsible for scheduling and coordinating all project and field activities, in accordance with the scope and budget of the project. Duties will include:

- Verifying all field records for accuracy;
- Ensuring that all personnel have a good understanding of the objectives of the program, an understanding of their respective roles relative to one another, and an appreciation of the importance of the roles to the overall success of the program;
- Procuring subcontracted services such as analytical laboratories, and coordinating communication between the project team members and these subcontractors;
- Scheduling and coordinating analytical services;
- Directing data management, validation and reporting;
- Coordinating preparation of groundwater and soil monitoring reports; and
- Routing documentation and reports to the QA/QC Officer for review and approval.

Dr. Christian Clausen will act as the Field Study Leader. Dr. Clausen or her designee will be responsible for overseeing the operation, maintenance and monitoring program during the field demonstration. He will be responsible for any QC measures required for collection of field data.

2.3 QA/QC Officer

The QA/QC Officer will assure and control quality by assuring that the activities are implemented using a consistent, high-quality approach, the information and data collected are compiled accurately and effectively for all team members to access and use, tasks are conducted in a manner that is consistent with the project plans, a consistent format for the deliverables is followed, an appropriate level of scoping and review has been conducted by senior technical specialists, and appropriate QA/QC procedures are implemented during data collection and reduction and during preparation of the deliverables.

Suzanne O'Hara will also perform the duties of QA/QC Officer. She will be responsible for validating that all field data meet the specifications of the data quality objectives and will distribute validated data to the appropriate project team members.

2.4 Project Technical Staff

Project technical staff will perform the sampling tasks and data reduction and validation, under the direction of the Field Study Leader-QA/QC Officer.

2.5 Analytical Laboratories

Analytical laboratories to be used for each parameter are listed in **Table C-1**.

3. DATA QUALITY

3.1 Data Quality Objectives

Data quality objectives (DQOs) are statements that provide project specific goals that are required to achieve results that will accurately and representatively reflect the primary parameters measured in the Treatment Area (TA) during the field demonstration period.

The primary parameters that will be measured to evaluate the conditions in the TA are shown in **Table C-1**. To maintain consistent and quality results throughout the field demonstration, Data Quality Indicators (DQIs) will be used to express the various properties of the primary parameters. The DQIs required are: precision, bias, accuracy, representativeness, completeness, and comparability (often referred to as PARCC criteria) which are listed below.

- Precision is a measure of agreement among replicate measurements of the same property under prescribed similar conditions.
- Accuracy is the closeness of an individual measurement to the true value. This includes a combination of random error (precision) and systematic error (bias) components that result from sampling and analytical operations.
- Representativeness is the degree to which sample data accurately and precisely represent a characteristic of a population parameter at a sampling point.
- Completeness is a measure of the amount of valid data obtained from the measurement system compared to the amount that should have been collected.
- Comparability is the confidence with which two data sets can contribute to a common analysis and interpretation.

Precision and bias will be measured by analysis of the field and laboratory QC samples described later in this section and comparison of statistics calculated using these results to the acceptance criteria specified in Section 6. Completeness will be calculated and compared to the acceptance criteria (see Section 6). The representativeness and comparability of the sample results shall be assured through the design of the sampling procedures as described in Section 6. In addition, required method detection limits (MDLs) have been established to ensure that a meaningful comparison of the concentration data to performance standards can be completed.

3.2 Sample Handling and Custody Requirements

Sample handling and custody requirements are described in detail in the Sampling Analysis Plan (SAP) located in Appendix B.

3.3 Field QA/QC Requirements

QA/QC samples consisting of duplicate samples will be collected to monitor the precision and bias resulting from sampling and laboratory analyses with respect to paint, paste, and sealant samples collected from the Site. The QA/QC requirements are discussed in detail in the SAP (Appendix B). A QA/QC checklist (Attachment 1) will be used to monitor field QA/QC procedures.

3.4 Laboratory QA/QC Requirements

Table C-1 identifies the laboratory analytical methods that will be conducted for the samples and the associated standard quantitation limits and holding times. Laboratory reports will include results of all sample and QA/QC sample analyses conducted, including any laboratory QC sample analyses (e.g., method blanks, spikes, surrogates). A QA/QC check list (Attachment 1) will be used to monitor laboratory QA/QC procedures.

3.5 Equipment Maintenance and Decontamination

All sampling and field equipment shall receive routine testing, inspection and maintenance checks to minimize equipment breakdown. Routine daily maintenance to be conducted in the field is listed below.

- Remove surface dirt and debris from the exposed surfaces of all equipment.
- Store equipment away from the elements.
- Inspect equipment and instruments for possible problems daily, including cracked or clogged lines or tubing, weak batteries, and worn pump heads.
- Charge any equipment battery packs when not in use.

Spare and replacement parts will be stored in the field to minimize down time. These will include:

- Appropriately sized batteries and electrical cords;

- Extra sample containers and preservatives;
- Spare parts for sprayers and compressors;
- Extra coolers and packing materials; and
- Additional supply of health and safety equipment.

Cleaning of non-dedicated and non-disposable field sampling and measuring equipment and personal protective equipment (PPE) will be performed to minimize the potential for cross-contamination between sample locations and samples, and to minimize the potential for exposure of workers to Site-related chemicals. All sampling devices and reusable PPE will be decontaminated immediately after each use with a direct application of a soapy water solution (i.e. Alconox), followed by a rinse with distilled water. Expended decontamination fluids are to be containerized for proper disposal. Protective gloves will be discarded following each sampling event.

4. ANALYTICAL CHECKS

4.1 Calibration Procedures and Frequency

All field equipment will be calibrated prior to use. Calibration checks using known standards will be run as necessary during the day and at the end of each sampling session according to the manufacturers' instructions.

4.2 Data Recording

Data generation will be initiated in the field, as personnel complete field forms. The field forms will include observations about weather conditions at the Site at the time of sample collection, the results of field measurements and analyses, and equipment calibration and identification information. The results of the field measurements, date and time of sampling, and the identification and order of samples collected will be recorded in the field records.

These forms shall be transmitted daily to the Field Study Leader-QA/QC Officer, who shall review the forms for clarity, completeness, and conformity with the SAP. The Field Study Leader-QA/QC Officer and field personnel shall communicate on a sufficient frequency in order to review the activities planned and to ensure the completion of the intended scope of work. The field forms shall be filled out as part of the project record.

4.3 Data Reduction

Data reduction will be minimal and will consist primarily of tabulating analytical data and field measurement results into electronic spreadsheets or databases. Electronic data transfer from the laboratory, where possible, will be utilized to minimize data transcription error.

4.4 Data Validation

Data validation is the process of evaluating data and accepting or rejecting it on the basis of the DQOs shown in **Table C-2**. The data quality criteria outlined in Section 6 will be used for making these decisions. Validation procedures will include the following:

- Ensuring close adherence to the specified sampling, protocol, and analysis procedures;

- Ensuring the use of properly calibrated and maintained equipment and analytical instrumentation; and

- Examining the precision, accuracy, and other QC aspects of the data generated during the project.

The data will be validated using EPA data qualifiers for organic and inorganic data (EPA, 540-R-01-008 and 540/R-99-008). Records of all data will be maintained, including those judged to be outliers or of spurious value. The persons validating the data will have sufficient knowledge of the data collection objectives to identify questionable values.

Analytical data generated in this program will be considered useful if the QC data for spiked and duplicate samples achieve the precision and accuracy goals stated in **Table C-2**, and if the sample is analyzed within the maximum holding time. If the precision and accuracy goals are not achieved, then these data will be flagged and the impact of not meeting the QA objectives will be delineated.

4.5 Data Reporting

The final report from the field demonstration will be the Technical Report, which will include a description of the experimental methods, observations of note, and the results from all chemical analyses in tabulated format. The assigned responsible parties will validate all analytical data, and the original data sheets will be retained with the project records. As appropriate, validated data will be archived using spreadsheet and/or database formats. The data will be stored in a spreadsheet file for further evaluation and calculations. In addition to the above items, the Technical Report will include the identification of outliers, details regarding the corrective actions taken, and a discussion of any necessary deviation from the protocols established in the referenced methods.

5. DEMONSTRATION PROCEDURES

A complete description of the field demonstration set-up and performance monitoring is presented in Section 3.6 of the Technology Demonstration Plan.

5.1 Demonstration Problems and Corrective Actions

Sampling system failures shall be reported by the field personnel directly to the Field Study Leader-QA/QC Officer (Jacqueline Quinn-Suzanne O'Hara) to specify the type of response action required, the method to evaluate effectiveness of the response action, and the methods for documenting the failure and response action implemented. The Field Leader-QA/QC Officer will review the cause of the failure and determine whether a future change to the sampling method is required. Spare and replacement parts will be stored in the field to minimize down time.

6. CALCULATION OF DATA QUALITY INDICATORS

Quantitative QA objectives for the field demonstration will be based on the analysis of samples collected and analyzed as outlined in **Table C-2**. The QA objectives for precision, accuracy, method detection limit (MDL), and completeness for this test's critical measurements are given in **Table C-2**.

6.1 Precision

As mentioned previously, precision quantifies the repeatability of a given measurement. Precision will be based on the relative percent difference (RPD) of duplicate samples. Precision for most laboratory measurements is estimated by calculating the RPD of laboratory duplicates such that,

$$RPD(\%) = \frac{C_1 - C_2}{(C_1 + C_2)/2} x 100$$

where:

RPD is relative percent difference, and
C_1 and C_2 are the result of replicate analysis.

A precision goal of 20% has been set for all of the critical parameters.

6.2 Accuracy

Accuracy is calculated in terms of the percentage of a known amount of analyte or matrix spike recovered from a given matrix. Percent recovery for an analyte is estimated by:

$$\%\,\mathrm{R} = \left(\frac{\mathrm{S - U}}{\mathrm{C_{sa}}}\right) \times 100$$

where:

%R = percent recovery
S = measured concentration in spiked aliquot
U = measured concentration in unspiked aliquot
C_{sa} = actual concentration of spike added

Accuracy will be determined by the percentage of analyte recovered (percent recovery [%R]) from the matrix spikes. Soil-phase matrix spikes for laboratory analyses will be prepared by injecting a known mass of a standard solution containing all of the analytes of interest into a groundwater sample, then analyzing the spiked sample according to the methods used for the groundwater samples.

6.3 Method Detection Limit (MDL)

The MDL for all measurements is calculated according to:

$$\mathrm{MDL} = t_{(n-1,1-\alpha=0.99)} \times s$$

where:

s = standard deviation of replicate analyses of matrix spikes with concentrations near the MDL

$t_{(n-1,\ 1-\alpha = 0.99)}$ = Students' t value for a one-sided, 99% confidence level and a standard deviation estimate with n-1 degrees of freedom.

6.4 Completeness

Completeness refers to the percentage of valid data received based on completed analysis performed in the laboratory, and is calculated by:

$$\%\,C = \left(\frac{V}{T}\right) \times 100$$

where:

%C = percent completeness
V = number of measurements judged valid
T = total number of measurements

Field QC will consist of sample duplicates collected for 5% of the samples submitted for analysis. One trip blank consisting of clean water in the respective sample container will be included for QC purposes with each cooler of samples submitted to the lab for analysis. A completeness goal of 80% has been set for all of the critical parameters. Because of the number of sampling events, and the number of samples collected during each sampling event, a completeness of 80% will allow attainment of project objectives.

6.5 Comparability

Comparability is the degree to which one data set can be compared to another. To ensure comparability, samples will be collected at specified intervals and in a similar manner, and they will be analyzed within required holding times by accepted methods. All data and units used in reporting this study will be consistent with accepted practices for groundwater and soil analyses.

6.6 Representativeness

Representativeness is the degree to which a sample or group of samples is indicative of the population being studied. Over the course of this study, samples will be collected in a manner such that they represent both the chemical composition and the physical state of the samples at the time of sampling. The sample collection protocol will minimize contact with the atmosphere, thereby minimizing volatilization.

7. PERFORMANCE AND SYSTEM AUDITS

7.1 Field Audits

Field audits of sampling protocols will be performed to ensure that procedures outlined in the SAP are followed. The audits will include observation of sampling procedures, and review of chain-of-custody documentation and sample results. These audits will be conducted by the QA/QC Officer (Suzanne O'Hara), or her designee, at the Site within the first month of initiating field activities. Following this period, daily audits of field activities and documentation will be conducted via telephone during field activities. A QA/QC checklist (Attachment 1) will be used to monitor field QA/QC procedures.

7.2 Laboratory Audits

The laboratory will perform internal performance audits. The audits conducted by the contracted laboratory will be reviewed by Geosyntec for compliance with QC objectives and in terms of timeliness and communications by measuring the laboratory's performance against their contract requirements for each analytical report submitted to Geosyntec.

7.3 Response Actions

Potential errors in field or laboratory data will be assessed through the field and laboratory audits (Sections 7.1 and 7.2). If it appears that field or laboratory data are in error, the error(s) or potential errors will be documented and appropriate response action(s) will be taken. Response actions may include one or more of the following:

- Measurements may be repeated;
- Calibrations may be checked and/or repeated;
- Instrument(s) or measuring device(s) may be repaired or replaced;
- New samples may be collected and/or samples may be re-analyzed; and
- Prepared samples having known concentrations (e.g., spike samples or standards) may be submitted blindly to the laboratory for analysis.

Appropriate response actions will be determined on a case-by-case basis. If any project staff become aware of any problems, they will immediately notify the Study Leader-QA/QC Officer who will determine the appropriate action to correct the problem. The Study Leader-QA/QC

Officer will also be responsible for directing the implementation of the response action(s), and documenting and communicating the problem encountered and the action taken.

8. QUALITY ASSURANCE REPORTS

The QA/QC assessment and the results of audits will be summarized in the Technical Report. The summary will include the following information:

- Overall assessment of field and laboratory programs;
- Summary of performance and system audits;
- Summary of data validation for accuracy, precision, completeness, representativeness, and comparability;
- Summary of sample tracking reports;
- Corrective actions taken, if any; and
- Any significant field observations noted on the field forms during the sampling procedures.

Chain-of-Custody (CoC) records, related sample documentation and data validation reports will be reviewed on an as received basis so that data quality problems will be detected as soon as possible. The Field Study Leader-QA/QC Officer will maintain the repository of all documents generated for this project.

9. DATA FORMAT

Data generation will be initiated in the field, as personnel complete field forms. The field forms will include observations about weather conditions at the Site at the time of sample collection, the results of field measurements and analyses, and equipment calibration and identification information.

All data generated during the course of field activities, will be recorded directly, promptly, and legibly in ink. All data entries will be dated and signed or initialed by the person making the entry. Any changes in data entries will not obscure the original entry and the reason for the change shall be noted and initialed. In automated data collection systems, the individual responsible for direct data input shall be identified at the time of data input. Any changes in automated data entries shall not obscure the original entry. Updated entries will indicate the reason for the change, the date, and the person responsible for making the change.

Data for this project will also include miscellaneous correspondence and laboratory analytical reports. Analytical data will be obtained from the lab in both hardcopy and electronic data deliverable (EDD) formats (if available). The electronic data will be checked for consistency with the hardcopy, in accordance with Geosyntec procedure. Conformance with established data storage protocols will then be verified for a number of data fields, including:

- Parameter name;
- Units;
- Date format;
- EPA qualifier; and
- Analytical method.

The EDD shall subsequently be translated into the standard Geosyntec database format. Features of this format include data storage in a single location to minimize version conflicts, and a database log to record import events.

10. DATA STORAGE AND ARCHIVING PROCEDURES

The database is stored in a project-specific directory. The server on which the directory is located is backed up on a regular basis, with storage of backups occurring offsite. Geosyntec will use the reports as the basis for preparing the Technical Report and will archive the raw data for 1 year.

All raw data, documentation, records, protocols, and reports generated as a result of field demonstration activities shall be retained. Upon completion of the technical effort, the field notebooks will be archived at Geosyntec for 1 year, or turned over to ESTCP for long-term archiving.

TABLE E-1: SUMMARY OF SAMPLE HANDLING AND LABORATORY ANALYTICAL DETAILS
Application Of A Bimetallic Treatment System (BTS) For PCB Removal From Older Structures On DOD Facilities

	Parameter	Analytical Method	Method Number	Analytical Laboratory (1)	Quantitation Limit	Sample Container	Preservative	Holding Time
PASTE	PCB and degradation products in paste	Gas Chromatography	Modified EPA 8082	UCF/KSC	0.10 mg/kg	plastic bottle for collection/glass for extraction	none	N/A
	Moisture Content of BTS	Visual Inspection Manual Inpection	N/A	N/A	N/A	N/A	N/A	N/A
PAINT	PCB and degradation products in paint	Gas Chromatography	Modified EPA 8082	UCF/KSC	0.10 mg/kg	plastic bottle for collection/glass for extraction	none	n/a
	Adhesion of paint	Field Test of Pull-Off Strength of Coatings	ASTM - D4541-02	N/A	N/A	N/A	N/A	N/A
SEALANT	Sealant integrity	Visual Inspection	NA	NA	NA	NA	NA	NA
	PCB and degradation products in sealant	Gas Chromatography	Modified EPA 8082	UCF/KSC	0.10 mg/kg	plastic bottle for collection/glass for extraction	none	N/A

Notes:

N/A - Not Applicable

UCF - University of Central Florida

KSC - Kennedy Space Center

(1) - 10% of the samples will be sent to a conmerical laboratory (to be determined) for confirmatory analysis.

TBD - To be determined

TABLE E-2: QA OBJECTIVES FOR PRECISION, ACCURACY, METHOD DETECTION LIMIT, AND COMPLETENESS
Application Of A Bimetallic Treatment System (BTS) For PCB Removal From Older Structures On DOD Facilities

	Parameter	Analytical Method	Method Number	Analytical Laboratory (1)	Quantitation Limit	Precision (%)	Accuracry (%)	Completeness (%)
PASTE	PCB and degradation products in paste	Gas Chromatography	Modified EPA 8082	UCF/KSC	0.10 mg/kg	20	80	80
	Moisture Content of BTS	Visual Inspection Manual Inpection	N/A	N/A	N/A	N/A	N/A	N/A
PAINT	PCB and degradation products in paint	Gas Chromatography	Modified EPA 8082	UCF/KSC	0.10 mg/kg	20	80	80
	Adhesion of paint	Field Test of Pull-Off Strength of Coatings	ASTM - D4541-02	N/A	N/A	20	80	80
SEALANT	Sealant integrity	Visual Inspection	NA	NA	NA	NA	NA	NA
	PCB and degradation products in sealant	Gas Chromatography	Modified EPA 8082	UCF/KSC	0.10 mg/kg	20	80	80

Notes:
N/A - Not Applicable
UCF - University of Central Florida
KSC - Kennedy Space Center
(1) - 10% of the samples will be sent to a conmerical laboratory (to be determined) for confirmatory analysis.
TBD - To be determined

ATTACHMENT 1

FIELD AND LABORATORY QA/QC CHECKLIST

TABLE 1
FIELD AND LABORATORY QA/QC CHECKLIST
Application of a BTS for PCB Removal from Older Structures on DOD Facilities

SAMPLE DATE				
SAMPLE LOCATION				
CRITERIA ***Field Sampling***				
Samples collected as per protocols in SAP (Y/N)				
QA/QC samples meet frequency criteria in SAP (Y/N)				
Lab Report Requirements				
Lab Name				
Contact person and telephone number				
Date of sample analysis				
Analyses required specified				
Data qualifiers used in lab report explained (Y/N)				
Any method modifications described				
Complete tabulated conc.s & DLs provided				
Def'n of short forms & acronyms provided				
Units:				
ug/L or mg/L for liquids				
mg/kg or ug/kg per dry wt for solids				
Other Lab Report Requirements				
Analyzed within holding times (Y/N)				
Lab report number (list)				
Lab report signed and dated (name/date)				
Lab report formatted correctly				
Method blanks specified (if applicable)				
Case Narrative provided (Y/N)				
Sample IDs consistent with C-o-C (Y/N)				
Chain-of-Custody				
Project Name specified				
Project Number specified				
Sample IDs specified				
Sampling date and time specified				
Collector's name, title, affiliation specified				
Date and time of sample receipt				
Parameters indicated (specify)				
QC samples:				
Field Duplicate collected: list sample IDs:				
Duplicate of: list sample ID				
Duplicate parameter of: List parameter				
RPD within 20% (specify RPD)				
Number of sample containers for each test				
Preservative/filtration indicated				
Overnight delivery indicated				
Method of shipment indicated				
Sample Receipt				
Custody seal intact				
Sample vials/containers intact				
Samples arrived within 7 days of date of collection				