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Laboratory Study of Polychlorinated Biphenyl (PCB) Contamination and Mitigation in Buildings

Part 4. Evaluation of the Activated Metal Treatment System (AMTS) for On-site Destruction of PCBs

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Executive Summary

E.1 Background

Polychlorinated biphenyls (PCBs) were once used as a plasticizer in certain building materials such as caulking, sealants, and paints from the 1950s through the late 1970s. Because PCBs have a variety of adverse health effects in animals and human, federal regulations have specific requirements for use and disposal of PCB-containing materials (U.S. EPA, 2005; 2009). Briefly, building materials that contain 50 ppm or more PCBs are not authorized for use and must be disposed of as *PCB bulk product waste* according the Code of Federal Regulations 40 CFR §761.3 and §761.62. If PCBs have contaminated either the surrounding building materials or adjacent soil, these materials are considered *PCB remediation waste*, which is subject to the cleanup and disposal requirements according 40 CFR §761.61.

There are many methods for mitigating PCB contamination in buildings. Source removal is one of the most commonly used methods. It can be accomplished either by physically removing the sources (such as caulk and light ballasts) or by chemically transforming PCBs into non-hazardous reaction products (EH&E, 2012). One of the chemical destruction techniques for reductive dechlorination of PCBs is the use of zero-valent metals, a technique that uses the Bimetallic Treatment System (BTS) or the Activated Metal Treatment System (AMTS). Both treatment systems are paste-like materials that contain a reducing agent, organic solvents/hydrogen donor, and thickening agent. The BTS method uses elemental magnesium (Mg) powder coated with a small amount of a palladium (Pd) complex as the reducing agent while the AMTS method uses only magnesium powder. According to Krug et al. (2010), the organic solvents in the BTS or AMTS paste can penetrate into the PCB-containing material and extract the PCBs from the material. Then the reducing agent in the paste degrades the extracted PCBs rapidly and thoroughly. Scientists from the National Aeronautics and Space Administration (NASA) and the University of Central Florida have refined the technique and applied it to the Department of Defense (DoD) facilities that had PCB levels as high as 11,000 mg/kg in the painted surfaces of concrete and greater than 50 mg/kg in the painted surfaces of steel (Krug et al., 2010). According to the authors, the demonstration of the technique at the DoD facilities showed rapid dechlorination of PCBs to less than 50 ppm in painted steel and concrete surfaces in about a week.

To expand this technique to other buildings, such as school buildings, several questions must be addressed, including:

- What is the efficiency of this technique for removing PCBs from indoor sources such as PCB-containing caulk, masonry materials, and paint?
- For thick sources such as PCB-containing caulk and concrete, how deep can the treatment system penetrate into the substrate to effectively remove PCBs?
- After PCBs are removed from the top layer of thick material, the PCBs in the deep layer may slowly migrate into the top layer, which is sometimes referred to as “bleed-back.” Will PCB bleed-back occur after treatment?

- Do the waste products from application of this technique cause any environmental concerns?

This study was intended to address these questions by using a combination of laboratory testing and mathematical modeling approaches. The results will be used to support the development of guidelines and decision-making tools for PCB mitigation.

The BTS and AMTS methods are closely related. Only the AMTS method was evaluated in this study because it was developed more recently and it is more cost- effective than the BTS method.

The AMTS paste can be applied to the surface of a PCB source either using spray-on or wipe-on techniques. The solvents in the mixture extract the PCBs from the source. The extracted PCBs are then destroyed by reacting with the reducing agent. There are two types of AMTS pastes: the “active paste” and the “inactive paste”. The former contains a reducing agent (magnesium powder) whereas the inactive paste does not. When the active paste is used, the extraction and chemical reaction take place at the same time. When the inactive paste is used, the paste needs to be collected from the source after treatment and then placed in a container where the extracted PCBs are destroyed by reacting with the reducing agent.

E.2 Objectives

The main goal of this study was to evaluate the effectiveness and usefulness of the AMTS method for decontamination of PCBs in buildings. The objectives were to:

- Evaluate the performance of the AMTS method for laboratory-mixed PCB paint, caulk, and concrete, as well as field caulk, under laboratory conditions.
- Evaluate the dechlorination mechanism of the AMTS method.
- Use modeling tools to predict PCB bleed-back from the AMTS-treated PCB sources.

E.3 Methods

E.3.1 Preparation of the AMTS Pastes

The active and inactive AMTS pastes (Figure E.1) were made prior to use according to the formulas provided by the NASA scientists. The active paste contains magnesium powder, ethanol, glacial acetic acid, limonene, and several other chemicals. The inactive paste does not contain magnesium powder. The exact formulas of the AMTS pastes and the preparation procedures are proprietary information owned by NASA.

Figure E.1. AMTS Pastes (left – inactive paste; right – active paste)

E.3.2 Application of the AMTS Method to PCB-Contaminated Materials

The AMTS pastes were tested on the following PCB-containing materials:

- Three types of coatings, i.e., oil-based primer (“primer”), oil-based alkyd paint (“alkyd”), and solvent-free epoxy coating (“epoxy”). Separate samples of each type of coating were spiked with either a high or a low concentration of Aroclor 1254, approximately 0.7% and 0.3% by weight, respectively (Figure E.2)
- Two types of PCB-containing caulk from buildings (Figure E.3) and two laboratory-made polysulfide caulk samples spiked with different concentrations of Aroclor 1254
- Laboratory-made concrete substrates spiked with Aroclor 1254 (Figure E.4)

Figure E.2. Application of the inactive paste to a coating coupon

Figure E.3. Field caulk covered with the active paste

Figure E.4. Concrete pieces in a stainless steel mold for treatment by the AMTS method

The test coupons were treated for 5 to 10 days. After treatment, the AMTS paste was removed from the coupons. Then, the test coupons were placed in scintillation vials for sonication extraction and GC/MS analysis.

E.3.3 Using a Barrier Model

After treatment by the AMTS, PCBs are eliminated from the top layer of the source. This layer acts as a barrier to the migration of PCBs from the interior of the source to the surface. To evaluate the protective effect of this barrier layer, a fugacity-based, multi-layer model (Yuan et al., 2007) was used to predict the "bleed-back" of PCBs (i.e., migration of PCBs from the deep layer of the source into the treated top layer). The material/air partition coefficients and solid-phase diffusion coefficients, two parameters required by the model, were estimated from other experiments, and the effective penetration depths for field caulk and laboratory-mixed concrete obtained from this study were used as inputs to the model. The outputs from the model included the concentration profiles of a selected PCB in the source and barrier layers as functions of time and depth and the contribution of the AMTS treated source to indoor air pollution as a function of time. The modeling results allowed the calculation of the average PCB concentrations in the barrier layer and the concentrations of the PCB on the exposed surfaces.

E.4 Findings

E.4.1 Efficiency of the AMTS Method

The test results presented in Section 4 demonstrate that the AMTS method removed PCBs from the primer and the alkyd paint effectively. The removal efficiencies were greater than 80% after seven days of treatment. These results are consistent with those reported by Krug et al. (2010). The removal efficiency was higher for the primer (> 95%) than for the alkyd paint (> 81%).

The removal efficiencies for the caulk and concrete samples were much lower than those for the coating materials, i.e., 12 to 36% of PCB congeners (4 to 36% in terms of Aroclor 1254) for the field caulk and 39 to 54% of PCB congeners (27 to 52% in terms of Aroclor 1254) for the laboratory-made concrete (Figure E.5).

The AMTS method was evaluated at two different PCB concentration levels for each type of material. The results showed that there was not much difference in the PCB removal efficiencies for the two concentrations for paint, field caulk and laboratory-made concrete.

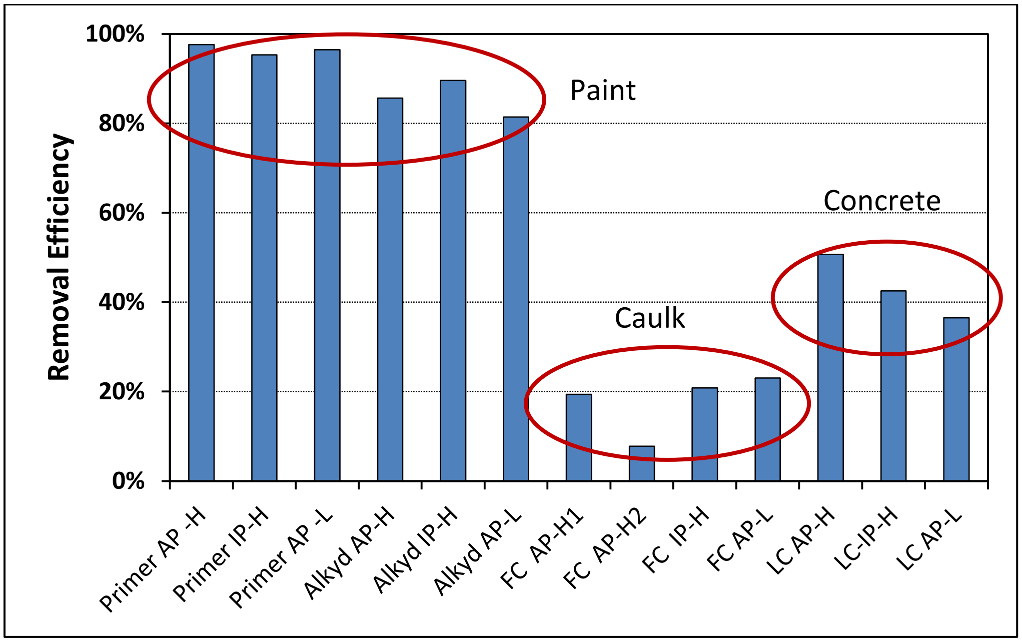

Figure E.5. Efficiency of the AMTS method in removing PCBs from different materials

(AP = active paste; IP = inactive paste; H = high PCB concentrations; L = low PCB concentrations; FC = field caulk; LC = laboratory-mixed concrete)

E.4.2 Effective Penetration Depths

The percent removal efficiency is a good indicator of the effectiveness of the AMTS method for coating materials, but the removal efficiency may be misleading for thick sources such as caulk and concrete because of the limited ability of the solvents in the pastes to penetrate the source substrate completely. The concept of “effective penetration depth” was introduced to determine the effectiveness of the AMTS for thick sources. The effective penetration depth is the thickness of a layer of the source material near the treated surface in which all PCBs are removed and beyond which the PCBs remain intact. This parameter is independent of the thickness of the source that is being treated (Section 5.2). The test results yielded an average effective penetration depth of approximately 1 mm for field caulk with 50% relative standard deviation (RSD) and approximately 3 mm for the laboratory-made concrete with 16% RSD.

E.4.3 Mathematical Modeling

After the AMTS treatment, the top layer of a thick source from which the PCBs has been removed acts as a temporary barrier to the migration of the PCBs from the untreated portion of the source substrate to the treated surface. It is important to know whether this barrier can effectively reduce the PCB concentration in room air and how long the protective effect lasts. To obtain a basic understanding of these issues, a fugacity-based barrier model was used to predict the "bleed-back" of PCBs for caulk and concrete after treated by AMTS. This model requires the material/air partition coefficient and solid-phase diffusion coefficient for the substrates. These parameters were obtained from the authors' previous studies (Guo et al., 2011, 2012a). The simulation results demonstrated that, when the AMTS method is used to treat thick sources that contain have very high concentrations of PCBs, the potential effect of PCB bleed-back on the PCB concentrations at the treated surface and in room air must be considered. Figures E.6 shows the simulation results for the concentration profiles of congener #110, the most abundant congener in Aroclor 1254, for treated concrete as a function of time and the depth of the test materials. It was assumed that the thickness of the PCB-containing layer of the concrete is 10 mm, that the initial concentration of congener #110 in the concrete is 196 μg/g, and that the AMTS creates an effective penetration depth of 3 mm.

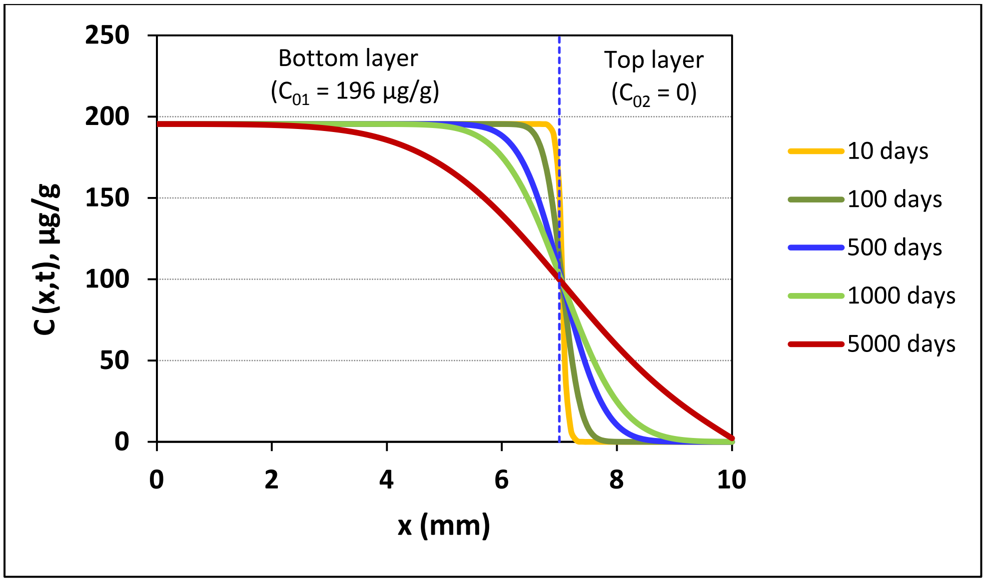

Figure E.6. Concentration profiles for congener #110 in the PCB-containing concrete as a function of time and depth, C(x,t), after the AMTS treatment

(C_{01} and C_{02} are the initial concentrations in the bottom and top layers, respectively; the effective penetration depth is 3 mm, i.e., between x = 7 and x = 10 mm in the x-axis.)

In general, the effect of PCB bleed-back for thick sources treated by AMTS on the PCB concentrations in room air is dependent on several factors, including the initial PCB concentration in the source, the effective penetration depth, the resistance of the substrate to PCB migration, and time. It could be a limiting factor for treating sources with very high PCB content. Therefore, post-treatment environmental monitoring, such as periodical air and wipe sampling, is necessary. Mathematical modeling can help understand the general behavior of the bleed-back phenomenon. Details are presented in Section 5.3 of the main body.

E.4.4 Summary of Findings

Overall, the AMTS method has the potential to become a viable method for mitigating PCB contamination in buildings. This method is promising for treating contaminated masonry materials near the expansion joints after the caulking material is removed because the AMTS can treat sources that contain several thousand ppm PCBs, for which the encapsulation method may not be effective. However, more research is needed because the current method has limited effective penetration depths for thick sources, including masonry materials. In addition, the reaction mechanism needs to be verified. The potential effect of the method on indoor air quality during the treatment also needs further evaluation.

E.5 Study Limitations

This study was conducted in a relatively short period of time with a limited scope. The main focus was the performance of the AMTS method as expressed in PCB removal efficiency and effective penetration depth. There are several important areas that this study did not investigate, including (1) the effectiveness of multiple treatments; (2) the properties and conditions of the source materials after being treated with AMTS; (3) the PCB residual concentration in the active paste after treatment. Furthermore, the study on the reaction mechanism of the AMTS method is inconclusive, and the modeling results for PCB bleed-back are semi-quantitative.

This study evaluated the AMTS technology that was available in early 2011. Since then, the developer of this method has modified the formulation and application procedure aimed to improve the performance of the method for contaminated masonry materials. This study did not evaluate this new method.

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Acronyms and Abbreviations

AMTS	Activated Metal Treatment System
BTS	Bimetallic Treatment System
CAS#	Chemical Abstracts Service Registry Number
CFR	Code of Federal Regulations
DCC	daily calibration check
DoD	Department of Defense
DQI	data quality indicator
EH&E	Environmental Health & Engineering, Inc.
EPA	U. S. Environmental Protection Agency
GC	gas chromatography
GC/MS	gas chromatography/mass spectrometry
IAP	internal audit program
IDL	instrument detection limit
IS	internal standard
IUPAC	International Union of Pure and Applied Chemistry
MSD	mass selective detector
NASA	National Aeronautics and Space Administration
NIST	National Institute of Standards and Technology
NRMRL	National Risk Management Research Laboratory
ORD	Office of Research and Development
PCB	polychlorinated biphenyl
ppm	parts per million
PQL	practical quantification limit
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
RCS	recovery check standard
RRF	relative response factor
RSD	relative standard deviation
SIM	selected ion monitoring
TMX	tetrachloro-*m*-xylene
TSCA	Toxic Substances Control Act

1. Introduction

1.1 Background

Polychlorinated biphenyls (PCBs), marketed as "Aroclors" and other trade names, were commonly used in public and commercial building-construction materials from the 1950s through the late 1970s in the United States (Herrick et al., 2004; Robson et al., 2010; Erickson & Kaley, 2011). Caulk, sealants, paint, fluorescent light ballasts, and other products manufactured with PCBs are the primary sources of PCBs in buildings. Because of their persistence (Brown et al., 1994), toxicological effects (ATSDR, 2000; U.S. EPA, 2008a), and the presence of high concentrations of PCBs in certain building materials in a wide range of old buildings, PCB-containing building materials are regulated by the Code of Federal Regulations 40 CFR §761 (U.S. EPA, 2005; 2009). Building materials with PCB concentrations greater than 50 ppm are not authorized for use and must be disposed of as *PCB bulk product waste* according to §761.3 and §761.62 or, otherwise, must be approved by EPA under a risk-based disposal according to §761.62(c). If PCBs have contaminated either the surrounding building materials or adjacent soil, these materials are considered *PCB remediation waste*, which is subject to the cleanup and disposal requirements according to §761.61.

The main goal of mitigating PCB contamination in buildings is to reduce human exposure to PCBs caused by emissions from and contact with PCB sources. The EPA recommended public health levels of PCBs in school indoor air are between 70 and 600 ng/m^3 depending on the age groups (U.S. EPA, 2012). In general, the existing methods for managing and remediating PCBs in buildings include: (1) physical removal of the sources, such as bulk removal, blasting, and cutting; (2) source modification, such as chemical extraction and chemical degradation; and (3) management solutions, such as encapsulation, physical barriers, ventilation, air cleaning, and administrative controls (EH&E, 2012 and references therein). Mitigating PCB contamination in buildings is complex and costly. The process generally involves: (1) obtaining site-specific information; (2) understanding regulations, regulatory implications, and social and economic impacts; (3) analyzing cost-effectiveness, cost-benefits, and short-term vs. long-term benefits; (4) assessing and managing risk and establishing health and safety protocols; (5) establishing acceptance criteria and verification methods; (6) pre-mitigation and post-mitigation monitoring; and (7) managing waste. The cost of mitigation ranges from $0.85 to $18 per square foot of building space and from $30 to more than $100 per linear foot of caulk, depending on the mitigation methods that are used (EH&E, 2012).

One of the chemical destruction techniques for PCBs is the use of zero-valent metals for reductive dechlorination. The technique uses the Bimetallic Treatment System (BTS) or the Activated Metal Treatment System (AMTS) to treat the PCB-contaminated materials (Agarwal et al., 2007; Kume et al., 2008; DeVor et al., 2008; DeVor et al., 2009; Maloney et al., 2011). Both treatment systems are paste-like materials that contain a reducing agent, organic solvents/hydrogen donor, and thickening agent. The BTS method uses elemental magnesium (Mg) powder coated with a small amount of a palladium (Pd) complex as the reducing agent while the AMTS method uses only magnesium powder. According to Krug et al. (2010), the organic solvents in the BTS or AMTS paste can penetrate into the PCB-containing material and extract the PCBs from the material. Then the reducing agent in the paste degrades the extracted PCBs rapidly and thoroughly. Scientists from National Aeronautics and Space Administration (NASA) and the University of Central Florida have refined the technique and applied it to actual contaminated structures (Krug et al., 2010).

The BTM and AMTS are prepared prior to use, and are in the form of "pastes." There are two types of pastes: the active paste and the inactive pastes. Their formulations are the same except that the latter does not contain the reducing agent. Consequently, the extraction and degradation of PCBs can be accomplished in either a one-step process by using the active paste or a two-step process by using the inactive paste. In the latter case, the inactive paste is applied to the surface of the PCB-containing material to extract and remove the PCBs from the source and then the extracted PCBs are degraded by the active metal reducing agent in a separate container (Krug et al., 2010).

The proposed reaction mechanism for PCB dechlorination is shown below:

$$Mg + 2\ ROH \rightarrow Mg\ (RO)_2 + H_2 \qquad (1.1)$$

$$n\ H_2 + 2\ C_{12}H_{10-n}Cl_n \rightarrow 2\ C_{12}H_{10} + n\ HCl \qquad (1.2)$$

In the reactions, Mg is a reducing agent and ROH is the proton donor (alcohol, acid, or water). The formula $C_{12}H_{10-n}Cl_n$ represents the PCB molecule, and $C_{12}H_{10}$ is biphenyl, a non-hazardous reaction product. The first reaction generates hydrogen, and the second reaction transforms the PCBs into non-chlorinated biphenyl through a dechlorination process. The method has been claimed to be applicable to painted structures, concrete surfaces contaminated by PCB-laden transformer oil, caulks and other adhesives, electrical equipment, soils, and other PCB-contaminated debris (Krug et al., 2010).

The BTS and AMTS methods have been applied in Department of Defense (DoD) facilities with PCB levels as high as 11,000 mg/kg in painted surfaces of concrete and greater than 50 mg/kg in painted surfaces of steel (Krug et al., 2010). The methods reduced the PCB concentration in treated paint to less than 50 mg/kg after one week of treatment. During this treatment process at DoD, a protocol was developed for formulating BTS and AMTS to enhance their applicability to various PCB-containing materials throughout DoD facilities, and their effectiveness was demonstrated on a wide range of structures that were contaminated with different PCB concentrations. According to the authors, the potential advantages of the chemical degradation techniques, including both the BTS and AMTS methods, are that it can be conducted on-site, that it is non-destructive, efficient, safe, and economically competitive, and that it eliminates long-term liabilities. However, it does have some limitations, i.e.: (1) it may be difficult to apply to irregular surfaces; (2) the treated surfaces may require reapplication of an additional coating after the first application; and (3) the active paste was unable to degrade all the PCB concentrations to less than 50 mg/g when the original PCB concentrations were very high (> 20,000 mg/kg). Also, an extremely simplified cost model was used to conduct the cost analysis. Application of BTS/AMTS to a concrete building prior to demolition appears to be more cost effective than to demolish the building and dispose of the waste in a Toxic Substances Control Act (TSCA) landfill.

The BTS and AMTS methods are closely related. Only the AMTS method was evaluated in this study because it is more recent and more cost-effective than the BTS method.

1.2 Goals and Objectives

The main goal of this study was to evaluate the effectiveness and usefulness of the AMTS method for treating PCB sources in buildings. The study was designed to: (1) evaluate the performance of the ATMS method for laboratory-mixed PCB-containing paint, caulk, and concrete, as well as PCB-containing caulk obtained from buildings; (2) use modeling tools to predict the PCB bleed-back (i.e., migration of PCBs from the deep layer of the source into the treated top layer); and (3) evaluate the AMTS reaction mechanism. The performance of the AMTS method was evaluated based on the PCB removal efficiency and the effective penetration depth. The information and data collected from this study can be used to support the development of risk-reduction strategies and decision-making tools regarding further recommendations for long-term measures to eliminate available PCBs on building surfaces and to minimize exposure to protect public health. The results should be useful to mitigation engineers, building owners and managers, decision-makers, researchers, and the general public.

1.3 About This Report

This is the fourth report in the publication series entitled *Laboratory Study of Polychlorinated Biphenyl (PCB) Contamination and Mitigation in Buildings*, produced by EPA's Office of Research and Development (ORD), National Risk Management Research Laboratory (NRMRL). The first report (Guo et al., 2011) was a characterization of the primary sources of PCBs that was focused on PCB-containing caulking materials and light ballasts. The second report (Guo et al., 2012a) focused on the transport of PCBs from secondary sources to interior surfaces and settled dust. The third report (Guo et al., 2012b) evaluated the encapsulation method as an interim measure for remediation of PCB contamination in buildings. This report summarizes the research results for evaluation of a chemical removal method for PCB sources. The study was limited to a laboratory investigation.

2. Experimental Methods

2.1 Activated Metal Treatment System Pastes

2.1.1 Preparation of AMTS Pastes

The exact formulas of the AMTS pastes and their preparation procedures are proprietary information that is owned by NASA. Active AMTS paste was prepared using the chemicals shown in Table 2.1. The inactive AMTS paste made up of all of the components shown in the table except the magnesium powder. Figure 2.1 shows the final active and inactive pastes.

Table 2.1. Components of the AMTS paste

Component	CAS#	Supplier
Ethanol	1634-01-4	Pharmco-APPER
Glacial acetic acid	--	Fisher Scientific
Limonene	5989-27-5	Sigma Aldrich
Calcium Stearate	--	ACROS
Carbomax PEG 8000	--	Fisher Scientific
Glycerin (glycerol)	--	Sigma Aldrich
Sodium Polyacrylate (5'100)	9003-04-7	Sigma Aldrich
Magnesium Powder	7439-95-4	Sigma Aldrich

Figure 2.1. AMTS Pastes (left – inactive paste; right – active paste)

2.1.2 Application and Removal of the AMTS Pastes

The paste was evenly applied to the substrates using a flexible putty knife. After application, a coating material (Dupli-Color® Professional Undercoat and Sound Eliminator, Product No. UC102) was sprayed over the paste and the substrate. This rubberized Dupli-Color® Undercoat encapsulates the paste, thereby preventing the evaporation of the ethanol (Krug et al., 2010). As the coating material dries, it may contract and form cracks. The substrates were inspected several times throughout the day on which the applications were applied, and the coating was reapplied, if necessary.

2.2 Test Procedures

The materials selected for tests are listed in Table 2.2. Mention of trade names is only for product identification. The laboratory tests that were conducted to evaluate the AMTS method are summarized in Table 2.3.

Table 2.2. Substrate materials tested

Product Name	Short Name	Binder or Base Material	Recommended Use	Recommended Application Method
All-Surface Enamel Oil-Based Primer	Oil-based primer	Oil-based primer	Wood, metal, drywall, interior/exterior	Brush, roller, airless sprayer
All-Surface Enamel Oil-Based Gloss	Alkyd paint	Oil-based enamel	Wood, metal, drywall, interior/exterior	Brush, roller, airless sprayer
Sikagard 62 [a]	Epoxy	Solvent-free epoxy	Concrete, steel	Brush, roller, airless sprayer
Quikrete® Sand/Topping Mix	Laboratory-prepared concrete	Cement and sand	Flooring, walls, driveways	Trowel and level
Industrial Polysulfide Joint Sealant [b]	Laboratory-prepared caulk	Polysulfide resin	Concrete expansion joints	Bulk caulk gun
Field caulk 1	N/A	Unknown	N/A	N/A
Field caulk 2	N/A	Unknown	N/A	N/A

[a] Two-part coating system.

[b] Two-part caulk.

Table 2.3. Summary of AMTS evaluation tests

Test ID	Material category	Matrix	Aroclor 1254 (w/w)	Number of samples		Application duration (days)
				Active paste	Inactive paste	
AMTS-E1 (E1)	Coating	Oil-based primer	0.72%	2	2	7
AMTS-E2 (E2)	Coating	Oil-based primer	0.36%	2	0	7
AMTS-E3 (E3)	Coating	Alkyd paint	0.72%	2	1	7
AMTS-E4 (E4)	Coating	Alkyd paint	0.31%	2	0	7
AMTS-E5 (E5)	Coating	Epoxy	0.79%	2	1	7
AMTS-E6 (E6)	Coating	Epoxy	0.32%	2	0	7
AMTS-E7 (E7)	Caulk	Field caulk 1	9.62%	5	1	5, 10
AMTS-E8 (E8)	Caulk	Field caulk 2	1.52%	3	0	5
AMTS-E9 (E9)	Caulk	Lab caulk 1	0.37%	2	0	5
AMTS-E10 (E10)	Caulk	Lab caulk 2	1.01%	4	2	5, 10
AMTS-E11 (E11)	Concrete	Laboratory-prepared concrete 1	0.30%	2	2	7
AMTS-E12 (E12)	Concrete	Laboratory-prepared concrete 2	0.08%	2	0	7
AMTS-E13 (E13) [a]	Coating	Oil-based primer	0.30% [b]	4	2	1, 3

[a] Test for degradation mechanism study.

[b] Concentration of PCB-209.

2.2.1 Tests for Coating Materials

The AMTS paste was tested on three different types of coatings, i.e., oil-based primer (“primer”), oil-based alkyd paint (“alkyd”), and solvent-free epoxy coating (“epoxy”). Each coating was spiked with two different concentrations of Aroclor 1254, approximately 0.7% and 0.3%, respectively. To add PCBs to the primer and alkyd paint, the stock paint was shaken for 15 minutes in a paint shaker (Red Devil, Model #54100H). The weight of an empty 60-mL amber jar was recorded. A calculated amount of Aroclor 1254 was added to the jar, and the jar was re-weighed. Then, a calculated amount of paint was added to the jar. The epoxy consisted of two components, i.e., Part A, the epoxy resin and Part B, the activator. The same process was used to add PCBs to the epoxy, except that Aroclor 1254 was added only to Part B. The final epoxy product was created by mixing Part A with Part B in a 1:1 ratio according to the manufacturer’s instructions. Table 2.4 shows the concentrations of Aroclor 1254 in formulation of each coating material and component.

Table 2.4. Laboratory preparation of Aroclor 1254-spiked coating

Source ID	Test ID	Matrix	Aroclor 1254 Source (g) [a]	Coating (g)	Concentration of Aroclor 1254 (w/w)[f]
S15900	AMTS-E1	Oil-based primer	0.289	40.038	0.72%
S16000	AMTS-E2	Oil-based primer	12.842 [b]	12.814	0.36%
S16100	AMTS-E3	Alkyd paint	0.283	39.137	0.72%
S16200	AMTS-E4	Alkyd paint	10.998 [c]	14.853	0.31%
S16300	AMTS-E5	Epoxy, Part B	0.354	24.865 [d]	0.79%
S16400	AMTS-E6	Epoxy, Part B	0.142	23.635 [e]	0.32%

[a] The source was pure Aroclor 1254 unless indicated otherwise.
[b] To make 0.3% Aroclor 1254 primer, 12.842 g of S15900 was diluted with the stock primer.
[c] To make 0.3% Aroclor 1254 alkyd, 10.998 g of S16100 was diluted with stock alkyd paint.
[d] Later, Part B was mixed with 19.5117 g of Part A of the epoxy system to obtain the final concentration.
[e] Later, Part B was mixed with 19.6743 g of Part A of the epoxy stem to obtain the final concentration.
[f] Values are based on wet paint.

A 21.9 × 28.6-cm^2 piece of free-film release paper (Paul N. Gardner Co., Inc., Item #PC-RP-1K) was painted using each of the paints listed in Table 2.4. The release paper, with one side coated with silicone, was designed for easy stripping of the dried paint film. Paint was applied using an artist’s brush that was 0.75 in (1.9 cm) wide. Two coats of paint were applied, except for the epoxy. Since the epoxy is thicker than the alkyd and primer, only one coat was necessary to cover the piece of release paper uniformly. There was a two-hour time interval between the two coats of the primer and at least seven hours for the alkyd. These curing times met the minimum curing time requirements recommended by the manufacturers. Each piece of painted release paper was cured for three months prior to use.

Test coupons (i.e., paint film on release paper) were created from the release paper using a 48-mm diameter arch punch (C.S. Osborne & Co., Arch Punch No. 149). To determine the initial PCB concentrations in the coupons, duplicate paint films were peeled off from the release paper and extracted for GC/MS analysis. For treatment tests, a small piece of tape was placed on the back of each coupon, and the coupons were attached to a 15.24 × 7.62-cm^2 aluminum panel (Figure 2.2).

Active paste (i.e., with magnesium powder) and inactive paste (i.e., without magnesium powder) were applied to the appropriate coupons (Figure 2.3). The Dupli-Color® Undercoat was then sprayed over the paste to prevent the evaporation of the ethanol (Figure 2.4). Touch-ups were made whenever necessary to ensure that there were no cracks in the coating layer. Since significant PCB degradation was reported in the AMTS paste even after only three days of treatment (Krug et al., 2010), we selected 5 to 10 days as treatment time for the method evaluation for different test materials. All paints were treated for seven days. After treatment, the Dupli-Color® Undercoat and the paste were removed from the coupons. The paint was then peeled off the release paper and placed in a scintillation vial for extraction.

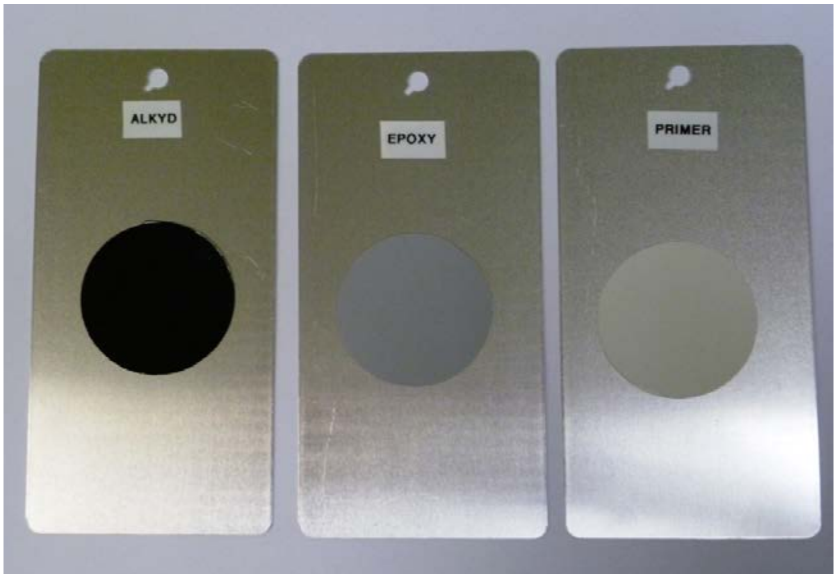

Figure 2.2. Coupons for paint tests (left: alkyd; center- epoxy; right: primer)

Figure 2.3. Application of inactive AMTS paste to a paint coupon

Figure 2.4. Paint coupon covered with the Dupli-Color® Undercoat

2.2.2 Tests for Field Caulk

Two types of interior caulk, collected from an unoccupied building scheduled for demolition, were used for this test. The concentrations of Aroclor 1254 in these two types of caulk differed by a factor of 10.

A cube of caulk was inserted into a vise-like holder (Figure 2.5). The apparatus held the caulk firmly in place and exposed only the top surface of the cube. Active and inactive pastes were applied to the designated caulk pieces (Figures 2.6), and the pastes were then covered with the Dupli-Color® Undercoat (2.7). After either five or ten days, the paste was removed from the caulk. Two slices of caulk, each with an approximate thicknesse of 2 mm, were cut from the treated caulk sample using the apparatus shown in Figure 2.8. These slices were cut perpendicular to the paste-treated side. Each slice was then diced (Figure 2.9) and extracted via sonication (Section 2.3).

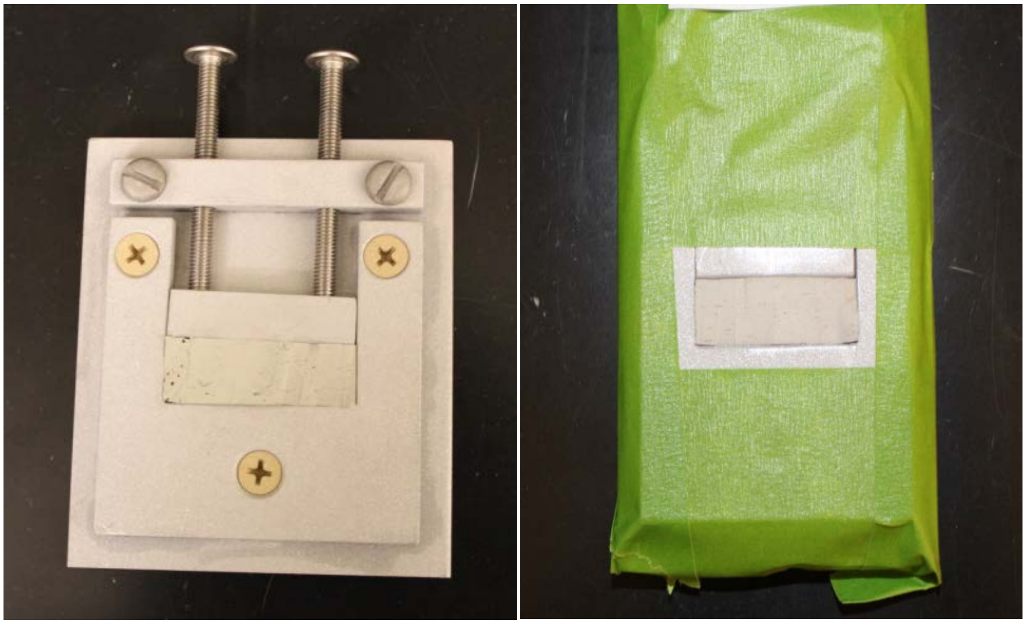

Figure 2.5. Field caulk in the holder before treatment

Figure 2.6. Field caulk covered with AMTS active paste

Figure 2.7. AMTS paste covered with the Dupli-Color® Undercoat

Figure 2.8. Caulk being cut into slices for extraction

Figure 2.9. Caulk slices for extraction

2.2.3 Tests for Laboratory-made Caulk

The caulking material used was Industrial Polysulfide Joint Sealant (THIOKOL 2235M, PolySpec, Houston, TX). The caulking material consisted of a resin (Part A) and a hardener (Part B).

An aliquot of Aroclor 1254 was added to Part B of the polysulfide caulk system since Part B was less viscous and more suitable for mixing than Part A. Then, Part B was added to Part A, and they were mixed until they were homogenized (Table 2.5). The caulk was then transferred to a 114 × 12.8 × 6.6-mm^3 (L × W × D) polyurethane foam mold (LAST-A-FOAM® FR-7100, General Plastics Manufacturing Co., Tacoma, WA) with a spatula (Figure 2.10).

After curing for five days, the caulk was removed from the mold and placed into a vise-like apparatus (Figure 2.11). The apparatus held the caulk firmly in place and exposed only the top surface of the piece. Active or inactive pastes were applied to the designated caulk pieces, and the paste was then covered with Dupli-Color® Undercoat. After seven days, the paste was removed from the caulk. Two slices of caulk, each with an approximate thickness of 2 mm, were cut from the treated caulk, using the apparatus shown in Figure 2.8. These slices were cut perpendicular to the paste application side. Then, each slice was diced and extracted via sonication.

Table 2.5. Preparation of Aroclor 1254-spiked laboratory-mixed caulk

Source ID	Test ID	Caulk		Aroclor 1254	
		Part B (g)	Part A (g)	Weight (g)	Concentration (w/w)
S16700	AMTS-E9	2.211	27.847	0.113	0.37% (3700 ppm)
S16600	AMTS-E10	4.405	55.65	0.613	1.01% (10100 ppm)

Figure 2.10. Laboratory-made caulk (left: caulk curing in the mold; right: caulk used in tests)

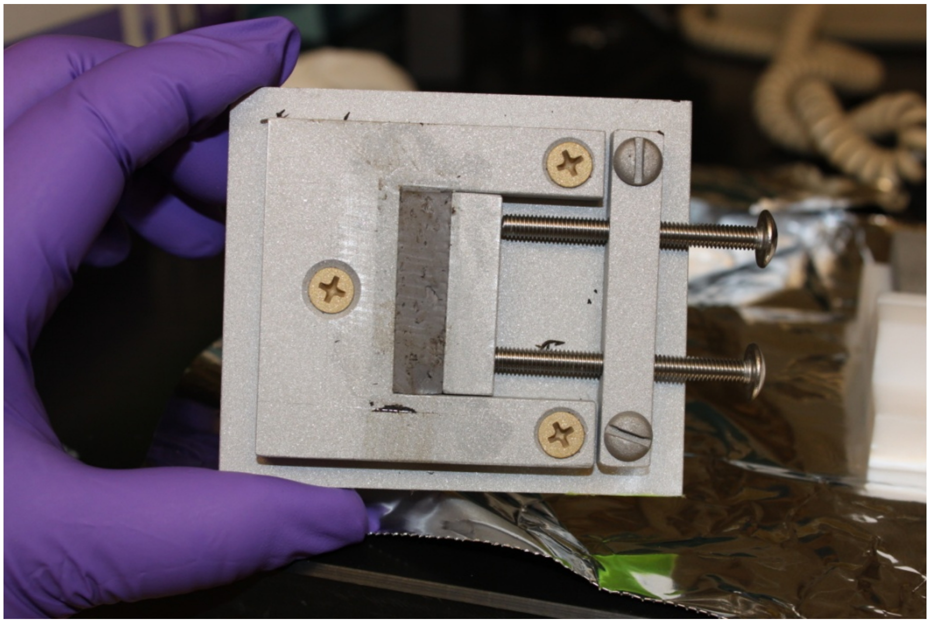

Figure 2.11. Laboratory-made caulk after AMTS treatment

2.2.4 Tests for Laboratory-made Concrete

Quikrete® Sand/Topping Mix (Product No. 1103) was used to create the concrete substrate that contained Aroclor 1254 (Table 2. 6). This ready-to-use concrete mixture consisted of Portland cement and commercial-grade sands. Since PCBs are not soluble in water, Aroclor 1254 was not added directly to the wet concrete. Instead, the desired amount of Aroclor 1254 was measured into a 60-mL wide-mouth jar. Concrete was weighed in an aluminum weighing dish and transferred to the jar containing the PCBs. Ten milliliters of hexane were mixed with the components to distribute the PCBs evenly throughout the concrete. The mixture was left standing in the fume hood for a minimum of one hour to ensure that all of the hexane had evaporated from the concrete. Once the concrete was completely dry again, 10 mL of water was mixed into the concrete. The concrete was poured into a cylindrical (12.5-mm diameter, 6-mm deep) stainless steel mold to make test coupons (Figure 2.12).

Table 2.6. Preparation of Aroclor 1254-spiked laboratory-made concrete

Source ID	Test ID	Concrete (g)	Aroclor 1254	
			Weight (g)	Concentration (w/w)
S16800	AMTS-E11	25.006	0.074	0.30% (3000 ppm)
S16900	AMTS-E12	25.006	0.021	0.08% (800 ppm)

Figure 2.12. Concrete pieces (left: in the stainless steel mold; right: individual coupons)

The concrete coupons were cured for seven days. With the coupons still in the mold, either active or inactive paste was applied to the top of the concrete and sprayed with Dupli-Color® Undercoat (Figures 2.13 and 2.14). After seven days, the paste and Dupli-Color® Undercoat were removed from the coupons. The concrete pieces were then removed from the mold and placed in scintillation vials. The vials were shaken vigorously in order to completely break the concrete coupon apart (Figure 2.15) for extraction.

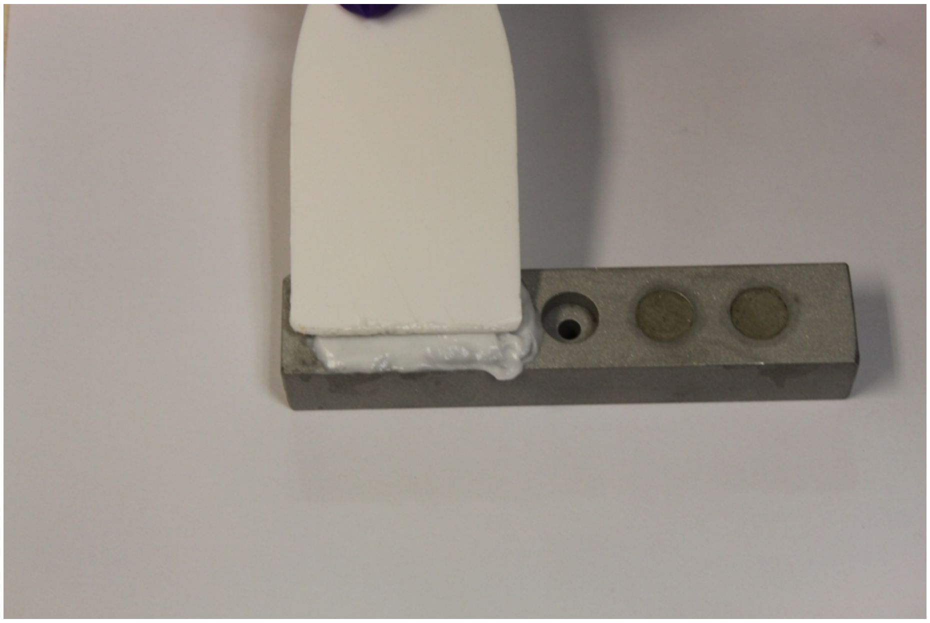

Figure 2.13. Application of inactive AMTS paste on the laboratory-made concrete

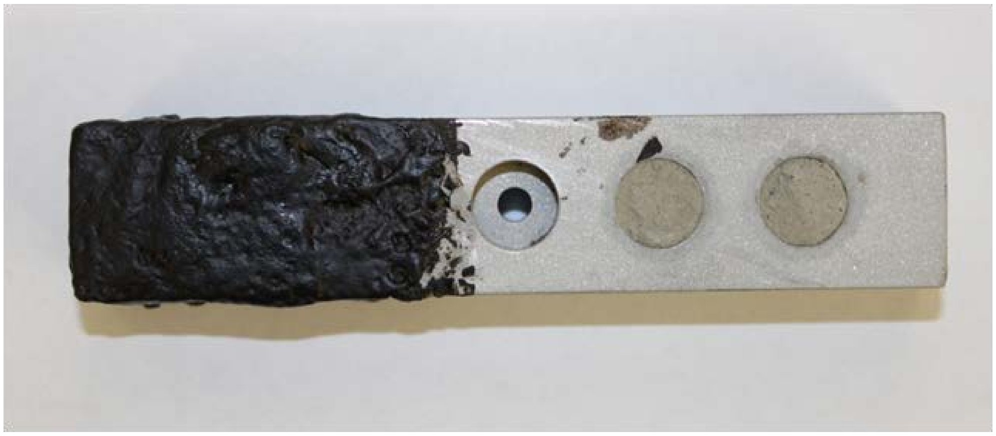

Figure 2.14. Laboratory-made concrete covered with AMTS paste and coating

Figure 2.15. Crushed concrete ready for extraction

2.2.5 Reaction Mechanism Study

The proposed reaction mechanisms for the AMTS method reduce PCBs to biphenyl (Equations 1.1 and 1.2 in Section 1). The possibility that the active paste would form partially-dechlorinated homologues was examined by applying the active paste to a primer coating that contained 0.30% PCB-209. Using PCB-209, which contains 10 chlorine atoms, allows detection of PCB congeners with lower chlorine numbers, if they are produced. A 21.9 × 28.6-cm^2 piece of free film release paper was painted with the primer (Table 2.7) using a 0.75 in (1.9 cm) wide artist's brush. A second coat was applied two hours later. The painted release paper was cured for two days prior to use. Coupons of the substrate were created by using a 48-mm diameter arch punch. A small piece of tape was placed on the back of each coupon, and the coupons were attached to a 15.2 × 7.62-cm^2 aluminum panel for treatment.

Table 2.7. Preparation of PCB-209-spiked paint for the reaction mechanism study

Source ID	Test ID	Primer (g)	Congener #209	
			Weight (g)	Concentration (w/w)
S16500	AMTS-E13	44.937	0.135	0.30% (3000 ppm)

Active and inactive pastes were applied to the appropriate coupons. The Dupli-Color® Undercoat was then sprayed over the paste. After either 24 or 72 hours, the Dupli-Color®Undercoat and the paste were removed from the coupons. The paint was peeled off the release paper and placed in a scintillation vial for extraction. In addition, approximately 500 mg of paste from each sample coupon were transferred to a vial for extraction.

2.3 Sample Extraction and Analysis

2.3.1 Sample Extraction

In our previous studies (Guo et al., 2011; 2012a), we concluded that the sonication method for extraction of caulk samples is comparable with the Soxhlet extraction method. Thus, the sonication method was selected for all samples in the AMTS evaluation. Ten milliliters of hexane was pipetted into the 20-mL amber scintillation vial that contained the sample. The recovery check standard (RCS, 100 µL) was added to the vial using a gas-tight syringe. The vial was sonicated for 30 minutes. Once the solution was cooled to room temperature, the hexane was decanted from the substrate and placed into a new scintillation vial, which contained approximately 500 mg of sodium sulfate (anhydrous grade or equivalent, Fisher, Pittsburgh, PA).

An aliquot (~1.5 mL) of the solution was then transferred from the amber scintillation vial into a screw-top vial that contained 3 mL of sulfuric acid. The sample was shaken for 30 seconds and then remained still until separation occurred. An aliquot of the top layer (hexane) was transferred into a 2-mL vial, and the sulfuric acid layer was discarded. This sulfuric acid wash was not used on the samples for determining the reaction products in Section 2.2.5.

Finally, 900 µL of the acid-washed extraction solution was added to a 1-mL volumetric flask. Using a gas-tight syringe, 100 µL of internal standard were inserted into the flask. The solution was brought to volume using the extract and mixed thoroughly with a vortex mixer (Fisher Scientific). A portion of the final solution was transfered to a GC vial for analysis.

2.3.2 Analytical Instrument and Reagents

The analytical instrument used for the quantitative analysis of PCB congeners was the Agilent 6980/5973N GC/MS (Agilent, Santa Clara, CA) with CTC PAL Auto Sampler (LEAP Technology, Carrboro, NC). Recovery check standards (RCSs) and internal standards (ISs) were spiked before extraction and GC analysis (Table 2.8). The internal standard solution for spiking contained 10 µg/mL of each IS. The RCS solution for spiking contained 5 µg/mL of each RCS. The certified PCB homologue standard (in isooctane) was purchased from AccuStandard, Inc. (New Haven, CT). The certified ^{13}C-labeled internal standards and recovery check standards (in nonane) were purchased from Wellington Laboratories, Inc. (Shawnee Mission, KS). The certified TMX standard (in acetone) was purchased from ULTRA Scientific (North Kingstown, RI).

Table 2.8. Chemical names and CAS Registration Numbers for the internal standards and recovery check standards

Purpose	Short Name	IUPAC Name	CAS#
Internal Standard	^{13}C-PCB-4	2,2'-Dichloro[$^{13}C_{12}$]biphenyl	234432-86-1
	^{13}C-PCB-52	2,2',5,5'-Tetrachloro[$^{13}C_{12}$]biphenyl	208263-80-3
	^{13}C-PCB-194	2,2',3,3',4,4',5,5',-Octachloro[$^{13}C_{12}$]biphenyl	208263-74-5
Recovery Check Standard	TMX	1,2,3,5-Tetrachloro-4,6-dimethylbenzene	877-09-8
	^{13}C-PCB-77	3,3',4,4'-Tetrachloro[$^{13}C_{12}$]biphenyl	105600-23-5
	^{13}C-PCB-206	2,2',3,3',4,4',5,5',6-Nonachloro[$^{13}C_{12}$]biphenyl	208263-75-6

2.3.3 Sample Analysis for AMTS Efficiency Tests

The target compounds for analysis are listed in Table 2.9.

Table 2.9. Chemical names and CAS Registration Numbers for the PCB congeners analyzed

Congener #	Short Name	IUPAC Name	CAS#
17	PCB-17	2,2',4-Trichlorobiphenyl	37680-66-3
52	PCB-52	2,2',5,5'-Tetrachlorobiphenyl	35693-99-3
66	PCB-66	2,3',4,4'-Tetrachlorobiphenyl	32598-10-0
77	PCB-77	3,3',4,4'-Tetrachlorobiphenyl	32598-13-3
101	PCB-101	2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2
105	PCB-105	2,3,3',4,4'-Pentachlorobiphenyl	32598-14-4
110	PCB-110	2,3,3',4',6-Pentachlorobiphenyl	38380-03-9
118	PCB-118	2,3',4,4',5-Pentachlorobiphenyl	31508-00-6
154	PCB-154	2,2',4,4',5,6'-Hexachlorobiphenyl	60145-22-4
187	PCB-187	2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0

The analytical method used for this project was a modification of EPA Method 8082A (U.S. EPA, 2007) and EPA Method 1668B (U.S. EPA, 2008b). The GC/MS was calibrated with PCB congeners in the range of 5 to 200 ng/mL. The GC/MS calibration and quantitation were performed using the relative response factor (RRF) method. The conditions of the analytical instrument were detailed in Part 1 of this report series (Guo et al., 2011).

2.3.4 Sample Analysis for the Reaction Mechanism Study

The analytical method used for the reaction mechanism study was a modification of EPA Method 680 (EPA, 1985). The GC/MS was calibrated using the relative response factor (RRF) method with PCB congeners from each homologue group, except for nonachlorobiphenyl, in the range of 5 to 600 ng/mL (Table 2.10). The RRF for decachlorobiphenyl was used as the calibration congener for both the nonachlorobiphenyl and decachlorobiphenyl homologue groups. The operating conditions of the instrument are presented in Table 2.11. The mass selective detector (MSD) selected ion monitoring (SIM) parameters were changed over time during analysis to achieve the best sensitivity, and these parameters are presented in Table 2.12.

Table 2.10. Chemical names and CAS Registration Numbers for EPA Method 680

Congener #	Short Name	IUPAC Name	CAS#
1	PCB-2	2-Chlorobiphenyl	2051-60-7
5	PCB-5	2,3-Dichlorobiphenyl	16605-91-7
29	PCB-29	2,4,5-Trichlorobiphenyl	15862-07-4
50	PCB-50	2,2',4,6-Tetrachlorobiphenyl	62796-65-0
87	PCB-87	2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8
154	PCB-154	2,2',4,4',5,6'-Hexachlorobiphenyl	60145-22-4
188	PCB-188	2,2',3,4',5,6,6'-Heptachlorobiphenyl	74487-85-7
200	PCB-200	2,2',3,3',4,5',6,6'-Octachlorobiphenyl	52663-73-7
209	PCB-209	2,2',3,3'4,4',5,5',6,6'-Decachlorobiphenyl	2052-24-3

Table 2.11. Operating conditions for the Agilent 6890/5973N GC/MS for EPA Method 680

Parameters	Settings
Injector	CTC PAL
Injection volume	1 µL
Inlet temperature	250 °C
Inlet mode	Splitless
Inlet Flow	1.0 mL/min measured at 100 °C
Carrier gas	Helium
GC column	SGE BPX5 30 m with 0.25-mm ID and 0.25-µm film thickness
Oven temperature program	100 °C for 2 min, to 150 °C at 15 °C/min, to 200 °C at 3 °C/min, to 300 °C at 8 °C/min, hold for 4 min, total time = 38.50 min
Transfer line temperature	280 °C
Acquisition Mode	SIM
Solvent delay	6 min

Table 2.12. SIM acquisition parameters for the Agilent 6890/5973N GC/MS for the analysis of PCB homologues

Homologues	Internal Standard	Retention Time Windows (min)	Primary Ion (m/z)	Confirmation Ions (m/z)
Monochlorobiphenyls	^{13}C-PCB-4	6.00 – 20.60	188	190
Dichlorobiphenyls	^{13}C-PCB-4	6.00 – 20.60	222	224
Trichlorobiphenyls	^{13}C-PCB-4	6.00 – 26.30	256	258
Tetrachlorobiphenyls	^{13}C-PCB-4	6.00 – 26.30	292	290
Pentachlorobiphenyls	^{13}C-PCB-52	20.60 – 28.25	326	324
Hexachlorobiphenyls	^{13}C-PCB-52	20.60 – 33.05	360	358
Heptachlorobiphenyls	^{13}C-PCB-52	20.60 – 33.05	394	392
Octachlorobiphenyls	^{13}C-PCB-52	28.25 – 38.50	430	428
Nonachlorobiphenyls	^{13}C-PCB-194	28.25 – 38.50	464	462
Decachlorobiphenyl	^{13}C-PCB-194	33.05 – 38.50	498	500
TMX (RCS) [a]	^{13}C-PCB-4	6.00 – 20.60	244	246
^{13}C-PCB-77 (RCS)	^{13}C-PCB-52	23.7	304	306
^{13}C-PCB-206 (RCS)	^{13}C-PCB-194	31.0	476	478
^{13}C-PCB-4 (IS) [b]	—	10.2	234	236
^{13}C-PCB-52 (IS)	—	17.8	304	306
^{13}C-PCB-194 (IS)	—	30.2	442	444

[a] TMX is tetrachloro-*m*-xylene; RCS is recovery check standard.

[b] IS is internal standard.

2.4 Environmental Parameters

The tests were performed under laboratory conditions, which were about 23 °C and 30-40% relative humidity

2.5 Measurement of the Dimensions of Test Coupons

Calculating the effective penetration depths for treated caulk and concrete coupons (described in Section 5.2) requires measurements of the sample dimensions. The measurements were made with AutoCAD LT 2005 (Autodesk, Inc., San Rafael, CA). The objects were photographed on a sheet of quad-rule paper with square grids that measured 0.5 cm, and a NIST-calibrated caliper (Mitutoyo Corp. Kawasaki, Japan) was used to obtain the measurements. The photographs were edited using Microsoft Office Picture Manager and then copied into AutoCAD LT 2005 to determine the area. In AutoCAD, the objects were scaled to the grid measurement of the paper, and a series of polylines was then connected to outline the object and closed to form the shape of the object. The area and perimeter of the shape were calculated based on the initial scale factor using the “list” command. To check the accuracy of the scale factor, a polyline drawing was made around four squares of the paper grid to determine the area. The area measurement was deemed acceptable if the grid area measurement, made using the NIST caliper, was within 5% of the physical measurement of the four square grids. This method of measuring area made it possible to determine the surface areas of the small caulk slices and the concrete coupons.

3. Quality Assurance and Quality Control

Quality assurance (QA) and quality control (QC) procedures were implemented in this project by following guidelines and procedures detailed in the approved Category II Quality Assurance Project Plan (QAPP), *Polychlorinated Biphenyls (PCBs) in Caulk: Evaluation of coatings for encapsulating building materials contaminated by polychlorinated biphenyls (PCBs) and a NASA method for PCB destruction*. Quality control samples consisted of method blank, extraction method blank, solvent blank, and duplicates. Daily calibration check samples were analyzed on each instrument on each day of analysis. Results of QA/QC activities are described in the following subsections.

3.1 Data Quality Indicator Goals for Critical Measurements

Data quality indicator (DQI) goals for the measurement parameters and validation methods are listed in Part 1 of this report series (Guo et al., 2011).

3.2 GC/MS Instrument Calibration

The GC/MS calibration and quantitation of PCBs were performed by using the relative response factor (RRF) method based on peak areas of extracted ion profiles for target analytes relative to those of the internal standard. For PCB congeners in Aroclor 1254, the calibration standards (in hexane) were prepared at six levels in the range from approximately 5 to 200 ng/mL. For EPA Method 680, PCB congener concentration varied because the concentrations of the congeners in the solution that was purchased (Accustandard, catalog # M-680A) were different. Three internal standards were added in each standard solution for different PCB congeners. The calibration curve was obtained by injecting 1 μL of the prepared standards in triplicate at each concentration level. Table 3.1 summarizes all GC/MS calibrations conducted for the project. The percent relative standard deviation (RSD) of the average RRFs meets the DQI goal of 25%.

The Internal Audit Program (IAP) was implemented to minimize any systematic errors. The IAP standards contained three calibrated PCB congeners, and IAP standards were analyzed after the calibration was completed. The certified IAP standards for PCB congeners from Aroclor 1254 were purchased from ChemService (West Chester, PA) and those of the PCB homologue were purchased from Ultra Scientific (North Kingstown, RI). The certified IAP standards were different from the supplier of the standards used for calibration. Table 3.2 presents the results of the analysis of the IAP standards for each calibration. The analytical results indicated that the recoveries of the IAPs ranged from 78% to 115% and that the percent RSDs ranged from 0.01% to 2.86%. All of these results meet the criteria for IAP analysis, which are 100 ± 25% recovery with percentage RSD of triplicate analyses within 25%.

Table 3.1. GC/MS calibration for PCB congeners [a]

For Aroclor 1254					For PCB Homologues (Congener Groups)						
Analytes	**9/29/2011**		**Range (ng/mL)**		**Analytes**	**11/11/2011**		**11/18/2011**		**Range (ng/mL)**	
	RRF	**%RSD**	**PQL[b]**	**Hi Cal [c]**		**RRF**	**%RSD**	**RRF**	**%RSD**	**PQL**	**Hi Cal [b]**
PCB-17	0.76	4.71	5.00	200	PCB-1	2.51	6.76	2.58	5.12	5.00	200
PCB-52	1.07	3.32	5.01	200	PCB-5	1.71	9.90	1.79	6.88	4.97	199
PCB-101	0.85	5.47	5.01	200	PCB-29	1.27	14.4	1.33	9.90	5.02	201
PCB-154	0.77	6.23	4.98	199	PCB-50	1.11	15.6	1.11	13.0	10.0	400
PCB-110	1.04	11.4	5.01	200	PCB-87	1.09	19.8	1.10	16.3	9.98	399
PCB-77	1.09	16.5	5.01	200	PCB-154	1.46	23.8	1.38	20.9	9.95	398
PCB-66	1.13	5.01	5.03	201	PCB-188	1.51	22.7	1.42	20.9	14.9	595
PCB-118	0.89	9.04	5.05	202	PCB-200	1.13	24.9	1.08	24.2	14.9	596
PCB-105	0.75	14.6	5.00	200	PCB-209	2.90	22.8	2.17	21.2	25	500
PCB-187	0.48	7.88	4.98	199	TMX (RCS)	0.42	5.15	0.43	5.68	5.01	201
TMX (RCS)	0.43	2.22	5.01	201	^{13}C-PCB-77 (RCS)	0.95	22.8	0.93	18.8	5.00	200
^{13}C-PCB-77 (RCS)	0.99	14.9	5.00	200	^{13}C-PCB-206 (RCS)	1.00	16.1	0.88	15.9	5.00	200
^{13}C-PCB-206 (RCS)	0.97	6.25	5.00	200	—	—	—	—	-—	—	—

[a] The DQI goal for %RSD was 25%; RRF data are for n = 3.

[b] PQL is practical quantification limi. It is the lowest calibration concentration.

[c] Hi Cal is the highest calibration concentration.

Table 3.2. IAP results for each calibration of PCB congeners [a]

Calibration Date	Analyte	IAP Concentration (ng/mL)	Recovery (n = 3)	
			Average %	%RSD
9/29/2011	PCB-52	36.0	115	0.01
	PCB-101	36.0	101	0.78
	PCB-77	36.0	80.9	0.95
11/11/2011	PCB-1	62.5	89.2	0.34
	PCB-5	62.5	84.3	0.68
	PCB-29	62.5	77.8	0.72
	PCB-50	125	89.8	0.76
	PCB-87	125	83.8	1.84
	PCB-154	125	89.3	1.60
	PCB-188	188	89.7	2.86
	PCB-200	188	88.7	1.61
	PCB-209	313	90.6	1.40
11/18/2011	PCB-1	62.5	88.3	0.56
	PCB-5	62.5	86.2	0.84
	PCB-29	62.5	82.1	0.86
	PCB-50	125	89.7	1.31
	PCB-87	125	86.0	0.88
	PCB-154	125	86.7	1.66
	PCB-188	188	87.1	1.28
	PCB-200	188	89.0	0.35
	PCB-209	313	90.4	1.52

[a] The DQI goal for %RSD was 25%.

3.3 Detection Limits

After each calibration, the instrument detection limit (IDL) was determined by analyzing the lowest calibration standard seven times and then calculating three standard deviations from the measured concentrations of the standard. IDLs are listed in Table 3.3 for all calibrated PCB congeners.

The detection limits for the sonication method were reported in the report entitled *Laboratory Study of Polychlorinated Biphenyl (PCB) Contamination and Mitigation in Buildings, Part II. Transport from Primary Sources to Building Materials and Settled Dust* (Guo et al., 2012a), Section 5.2.

Table 3.3. Instrument detection limits (IDLs) for PCB congeners on GC/MS (ng/mL)

For Aroclor 1254		For PCB Homologues (Congener Groups)		
Analytes	**9/29/2011**	**Analytes**	**11/11/2011**	**11/18/2011**
PCB-17	0.46	PCB-1	0.18	0.11
PCB-52	0.32	PCB-5	0.33	0.19
PCB-101	0.80	PCB-29	0.19	0.26
PCB-154	0.51	PCB-50	0.35	0.34
PCB-110	0.74	PCB-87	0.42	0.38
PCB-77	0.55	PCB-154	0.26	0.45
PCB-66	0.71	PCB-188	0.42	0.63
PCB-118	0.67	PCB-200	0.47	1.00
PCB-105	0.83	PCB-209	1.29	0.79
PCB-187	0.78	TMX (RCS)	0.77	0.53
TMX (RCS)	0.34	^{13}C-PCB-77 (RCS)	0.15	0.31
^{13}C-PCB-77 (RCS)	0.57	^{13}C-PCB-206 (RCS)	0.57	1.01
^{13}C-PCB-206 (RCS)	1.40	—	—	—

3.4 Quality Control Samples

Data quality control samples discussed here include method blank, extraction method blank, solvent blank and duplicates.

The method blank was carried through the complete sample preparation and analytical procedure, from AMTS paste application to GC/MS analysis, but there were no PCBs in the materials. A typical method blank sample showed the contribution of the contamination in the whole test process. The results are summarized in Table 3.4. The method blank samples were shared within each type of tests. No method blank samples were collected for mechanism study test E13. Most of the method blank samples were below PQL with the exception of tests E11 and E12, in which PCB-101, PCB-110, PCB-118, and PCB-105 had higher amounts per sample, most likely due to high carryover from the GC/MS analysis.

Table 3.4. Summary of method blank for tests (ng/sample) [a]

Analytes	Test ID						
	E1 & E2	**E3 & E4**	**E5 & E6**	**E7 & E8**	**E9**	**E10**	**E11 & E12**
PCB-17	~~0.00~~	~~0.00~~	~~0.00~~	~~0.00~~	~~0.00~~	~~0.00~~	~~0.00~~
PCB-52	~~0.90~~	~~0.00~~	~~0.00~~	~~0.00~~	~~3.91~~	~~9.74~~	~~34.3~~
PCB-101	~~2.12~~	~~0.00~~	~~0.00~~	~~0.00~~	~~7.62~~	~~22.1~~	85.5
PCB-154	~~1.71~~	~~0.00~~	~~0.00~~	~~0.00~~	~~0.74~~	~~2.60~~	~~9.50~~
PCB-110	~~2.86~~	~~0.00~~	~~0.00~~	~~0.00~~	~~11.2~~	~~32.1~~	128
PCB-77	~~4.27~~	~~2.89~~	~~0.00~~	~~0.00~~	~~0.00~~	~~0.00~~	~~0.23~~
PCB-66	~~1.65~~	~~0.00~~	~~0.00~~	~~0.00~~	~~1.96~~	~~6.23~~	~~14.2~~
PCB-118	~~4.47~~	~~0.00~~	~~0.00~~	~~0.00~~	~~20.3~~	~~49.6~~	151
PCB-105	~~4.39~~	~~0.00~~	~~1.60~~	~~0.00~~	~~13.7~~	~~30.9~~	92.8
PCB-187	~~2.44~~	~~0.00~~	~~0.00~~	~~0.00~~	~~0.00~~	~~2.09~~	~~4.11~~

[a] Values in strikethrough are below the PQL; each sample was extracted in 10 mL of hexane.

The extraction method blank was generated using hexane solvent following exactly the same extraction and GC/MS analysis procedures as the samples. The extraction method blank documented the contamination during solvent extraction and GC/MS analysis. When the samples were extracted at the same time, only one extraction blank was prepared. The results are presented in Table 3.5. Concentrations of PCBs in the extraction method blank that was shared by tests E1 through E6 were lower than the PQL, but the RCS test failed, so the data were not reported.

Table 3.5. Summary of extraction method blanks for tests (ng/sample) [a]

Analytes	Test ID				
	E7 & E8	E9 & E10	E11 & E12	Analytes	E13
PCB-17	~~0.00~~	~~0.00~~	~~0.00~~	PCB-1Cl	~~0.00~~
PCB-52	~~0.00~~	~~1.10~~	~~0.29~~	PCB-2Cl	~~0.00~~
PCB-101	~~0.00~~	~~3.13~~	~~0.28~~	PCB-3Cl	~~0.00~~
PCB-154	~~0.00~~	~~0.00~~	~~0.00~~	PCB-4Cl	~~0.00~~
PCB-110	~~0.00~~	~~4.25~~	~~1.36~~	PCB-5Cl	~~0.00~~
PCB-77	~~0.00~~	~~0.00~~	~~0.55~~	PCB-6Cl	~~0.00~~
PCB-66	~~0.00~~	~~0.00~~	~~0.42~~	PCB-7Cl	~~0.00~~
PCB-118	~~0.00~~	~~5.91~~	~~1.88~~	PCB-8Cl	~~0.00~~
PCB-105	~~0.00~~	~~0.00~~	~~1.36~~	PCB-9Cl	~~0.00~~
PCB-187	~~0.00~~	~~0.00~~	~~0.00~~	PCB-10Cl	~~0.00~~

[a] Values with strikethrough are below the PQL; each sample was extracted using 10 mL of hexane.

The solvent blank was used to determine whether there was any contamination in the solvent used for sample extraction. The blank was prepared by adding 10 mL of extraction solvent to a 20-mL vial while conducting solvent extraction. The solvent (1 mL) was then spiked with internal standards and analyzed by GC/MS with the other samples. The results are shown in Table 3.6. No solvent blank samples were prepared for Test E11, Test E12, or Test E13. All data shown in Table 3.6 are below the PQL.

Table 3.6. Summary of solvent blank for tests (ng/sample) [a]

Analytes	Test ID		
	E1, E2, E3, E4, E5, E6	E7, E8	E9, E10
PCB-17	~~0.00~~	~~0.00~~	~~0.00~~
PCB-52	~~0.00~~	~~0.00~~	~~0.00~~
PCB-101	~~0.00~~	~~0.00~~	~~1.42~~
PCB-154	~~0.00~~	~~0.00~~	~~0.00~~
PCB-110	~~0.00~~	~~0.00~~	~~2.20~~
PCB-77	~~0.00~~	~~0.00~~	~~0.28~~
PCB-66	~~0.00~~	~~0.00~~	~~1.63~~
PCB-118	~~0.00~~	~~0.00~~	~~4.87~~
PCB-105	~~0.00~~	~~0.00~~	~~1.34~~
PCB-187	~~0.00~~	~~0.00~~	~~0.00~~

[a] Values in strikethrough are below the PQL; each sample was extracted using 10 mL of hexane.

Duplicate samples were used to estimate the precision of the sampling and analysis methods. The DQI was set to be RSD < 25%. Duplicate samples were prepared for each AMTS evaluation test condition. The results are summarized in the Result Section of this report. Table 3.7 summarizes the number of duplicate PCB congeners analyzed in each test and shows the number of duplicate PCB congeners that failed. The data quality was not necessarily unacceptable if the data did not meet the DQI for the tests because failing to meet the DQI might imply that the concentrations of the PCB congeners in the test materials were uneven.

Table 3.7. Summary of duplicate samples for PCB congeners

Test ID	Matrix	Number of duplicate samples	Number of duplicate congeners	Number of duplicate congeners failed
AMTS-E1	Oil-based primer	3	26	8
AMTS-E2	Oil-based primer	2	17	1
AMTS-E3	Alkyd paint	2	19	9
AMTS-E4	Alkyd paint	2	18	0
AMTS-E5	Epoxy	2	14	1
AMTS-E6	Epoxy	2	12	5
AMTS-E7	Field caulk 1	4	70	0
AMTS-E8	Field caulk 2	3	40	0
AMTS-E9	Lab caulk 1	2	27	9
AMTS-E10	Lab caulk 2	4	70	1
AMTS-E11	Laboratory-prepared concrete 1	3	30	0
AMTS-E12	Laboratory-prepared concrete 2	2	20	0
AMTS-E13	Oil-based primer	3	19	11

The depth of each piece of caulk and concrete was measured multiple times. The precision of the multiple measurements is reported in the Results section of this report.

3.5 Daily Calibration Check

On each day that analyses were conducted, at least one daily calibration check (DCC) sample was analyzed to document the performance of the instrument. DCC samples were analyzed at the beginning of and during the analysis sequence on each day. Table 3.8 summarizes the average recovery of DCCs for the tests. The recoveries meet the laboratory criterion of 75 to 125% recovery for acceptable GC/MS instrument performance.

Table 3.8. Average recoveries of DCCs for AMTS method evaluation tests

Test Type	DCC Analytes	Average %Recovery	SD	%RSD	N [a]
For Aroclor 1254 [b]	PCB-17	99.7	0.053	5.34	37
	PCB-52	100	0.024	2.37	37
	PCB-101	96.0	0.028	2.93	37
	PCB-154	96.5	0.028	2.86	37
	PCB-110	102	0.045	4.40	37
	PCB-77	103	0.063	6.10	37
	PCB-66	96.4	0.055	5.67	37
	PCB-118	98.8	0.046	4.62	37
	PCB-105	103	0.056	5.48	37
	PCB-187	104	0.052	5.02	37
	TMX (RCS)	98.8	0.044	4.43	37
	^{13}C-PCB-77 (RCS)	104	0.065	6.25	37
	^{13}C-PCB-206 (RCS)	101	0.039	3.84	37
For Method 680 [c]	PCB-1	104	0.023	2.24	8
	PCB-5	109	0.060	5.53	8
	PCB-29	109	0.082	7.52	8
	PCB-50	104	0.016	1.54	8
	PCB-87	104	0.053	5.14	8
	PCB-154	101	0.045	4.51	8
	PCB-188	100	0.055	5.50	8
	PCB-200	105	0.071	6.81	8
	PCB-209	94.8	0.105	11.1	8
	TMX (RCS)	103	0.036	3.52	8
	^{13}C-PCB-77 (RCS)	107	0.093	8.62	8
	^{13}C-PCB-206 (RCS)	98.5	0.035	3.60	8

[a] N is the number of DCCs analyzed.

[b]. Modified EPA Method 8082A and EPA Method 1668B were used for analysis of congeners in Aroclor 1254

[c]. Modified EPA Method 680 was used for the reaction mechanism study

3.6 Recovery Check Standards

Three recovery check standards (RCSs), i.e., TMX, ^{13}C-PCB-77, and ^{13}C-PCB-206, were spiked in each of the samples before extraction to serve as the laboratory controls. When the measured concentrations of PCBs in the sample were above the highest calibration level, which happened mostly during bulk analysis, the extract was diluted to re-analyze the sample. In that case, recoveries of RCSs were not reported. The analytical results are considered acceptable if the percent recovery of laboratory controls was in the range of 60-140% for at least two of the three recovery check standards. The results of the recovery check standards are listed in Table 3.9.

Table 3.9. Results summary of the recovery check standards

Test ID	Matrix	Number of samples	Number of RCS failed
AMTS-E1	Oil-based primer	8	0
AMTS-E2	Oil-based primer	6	0
AMTS-E3	Alkyd paint	6	0
AMTS-E4	Alkyd paint	6	0
AMTS-E5	Epoxy	7	3
AMTS-E6	Epoxy	6	1
AMTS-E7	Field caulk 1[a]	15	0
AMTS-E8	Field caulk 2[a]	9	0
AMTS-E9	Lab caulk 1[a]	8	0
AMTS-E10	Lab caulk 2[a]	16	0
AMTS-E11	Laboratory-prepared concrete 1	7	0
AMTS-E12	Laboratory-prepared concrete 2	6	0
AMTS-E13	Oil-based primer	9	3

[a] two slices were extracted and analyzed for each caulk piece.

4. Results

4.1 Terminology and Definitions

In this study, the effectiveness of the AMTS method for removing PCBs from test materials was expressed as percent removal efficiency (or % efficiency), as defined by Equation 4.1:

$$\%Efficiency = \left(1 - \frac{C_1}{C_0}\right) \times 100 , \tag{4.1}$$

where C_0 is the concentration of PCB congeners or Aroclor 1254 in the original, untreated test materials, and C_1 is the concentration of PCB congeners or Aroclor 1254 in the materials after the treatment by AMTS. Both C_0 and C were measured by GC/MS.

The "average efficiency for PCB congeners" is the average efficiency for the ten quantified PCB congeners in each sample.

The concentration of Aroclor 1254 in the samples was calculated following the adaptation of Method 8082A, which calculates the Aroclor concentration in three steps. The details were described in the report entitled *Laboratory Study of Polychlorinated Biphenyl (PCB) Contamination and Mitigation in Buildings, Part I. Emissions from PCB-Containing Caulking Materials and Light Ballasts* (Guo et al., 2011), Section 4.1.10.

The weight fractions of selected PCB congeners, F_i, in the Aroclor 1254 calculation are summarized in Table 4.1.

Table 4.1. Weight fractions (F_i) of selected PCB congeners used to calculate Aroclor 1254 concentration

PCB Congeners	W_{si} (ng) [b]	W_s (ng) [b]	F_i [b]
PCB-52	43.43	1002	0.043
PCB-101	66.32	1002	0.066
PCB-154, PCB-110, PCB- 77 [a]	74.66	1002	0.075
PCB-118	53.31	1002	0.053
PCB-105	22.12	1002	0.022

[a] PCB-154, PCB-110, and PCB-77 co-eluted. [b] W_{si} is the content of congener i in the Aroclor standard that was injected, W_s is the amount of the Aroclor standard that was injected, and F_i is the weight fraction of congener i in the Aroclor standard injected, given by the ratio W_{si}/W_s.

4.2 Tests for Coating Materials

Three types of coating materials prepared in the laboratory with different concentrations of PCBs were tested to evaluate the AMTS method. The samples were treated with the AMTS paste for seven days. The concentrations of PCB congeners in the samples were measured before and after the treatment. The initial concentrations in the materials before the treatment are summarized in Table 4.2.

Table 4.2. Summary of the concentrations of PCB congener (µg/g) and Aroclor 1254 (w/w) measured by GC/MS in the untreated test materials [a]

Analytes	**Test ID and Matrix**					
	E1 Primer	**E2** Primer	**E3** Alkyd	**E4** Alkyd	**E5** Epoxy	**E6** Epoxy
Aroclor 1254 (% w/w) [b]	0.717	0.359	0.718	0.306	0.792	0.324
Aroclor 1254 (µg/g) [b]	7170	3590	7180	3060	7920	~~3240~~
PCB-17 (µg/g)	3.18	1.41	4.90	1.85	0.01	~~0.01~~
PCB-52(µg/g)	268	142	462	193	0.77	NR
PCB-101 (µg/g)	535	221	595	316	1.20	NR
PCB-154 (µg/g)	53.4	27.1	91.6	40.6	0.10	~~0.03~~
PCB-110 (µg/g)	567	251	652	354	1.34	NR
PCB-77 (µg/g)	0.96	0.53	1.47	0.68	0.00	~~0.00~~
PCB-66 (µg/g)	55.2	24.1	80.1	29.4	0.22	0.07
PCB-118 (µg/g)	389	198	482	262	1.62	NR
PCB-105(µg/g)	260	96.9	354	121	0.77	NR
PCB-187 (µg/g)	17.5	6.66	23.5	10.3	0.03	~~0.01~~
Aroclor 1254 (% w/w)	0.84	0.37	1.10	0.50	0.002	0.001
Aroclor 1254 (µg/g)	8400	3700	11000	5000	200	100

[a] Average of duplicate samples; values in strikethrough are below the PQL; NR means “not reported” due to DQI failure; measured concentrations are for cured coatings.

[b] Spiked concentration, calculated by wet weight.

The efficiency of the AMTS treatment was calculated using Equation (4.1), and the results are summarized in Tables 4.3 (average percent efficiency for the target congeners) and 4.4 (percent efficiency for Aroclor 1254). The difference between the two sets of results is rather minor.

Tests AMTS-E5 and AMTS-E6 were conducted to determine the efficiency of the AMTS on an epoxy coating, which consisted of two components, Part A and Part B. Aroclor 1254 was added to Part B, then Part B was mixed with Part A to form solid epoxy (after curing). When the epoxy was extracted with hexane solvent, the extraction efficiency was very low, and the precision of the data was very poor. Most of the data did not meet the DQI goals and, therefore, the results are not reported here.

Table 4.3. Percent removal efficiencies for the sum of the target PCB congeners for coating materials after the AMTS treatment [a]

Test ID	Coating	Aroclor 1254 (% w/w) [b]	Sample ID [c]	%Efficiency [d]	Average	SD	%RSD
E1	Primer	0.717	AP-A	96.8	96.9	0.01	0.01
			AP- B	96.9			
			IP-A	92.6	94.5	2.59	2.74
			IP-B	96.3			
E2	Primer	0.359	AP-A	96.8	96.2	0.96	1.00
			AP- B	95.5			
E3	Alkyd	0.718	AP-A	82.4	85.9	4.89	5.70
			AP- B	89.4			
			IP	89.7	--	--	--
E4	Alkyd	0.306	AP-A	80.9	80.6	0.48	0.60
			AP- B	80.3			

[a] Treated for seven days.

[b] Spiked concentration, calculated based on the wet weight of the coating materials.

[c] AP (active paste); IP (inactive paste).

[d] Average efficiency for ten PCB congeners.

Table 4.4 Percent removal efficiencies for Aroclor 1254 after AMTS treatment of coatings [a]

Test ID	Coating	%Aroclor 1254 (w/w) [b]	Sample ID [c]	Concentration (μg/g)	%Efficiency	Average	SD	%RSD
E1	Primer	0.717	Untreated	8345	--	--	--	--
			AP-A	194	97.7	97.6	0.06	0.06
			AP- B	202	97.6			
			IP-A	510	93.9	95.3	2.05	2.15
			IP-B	267	96.8			
E2	Primer	0.359	Untreated	3692	--	--	--	--
			AP-A	112	97.0	96.4	0.75	0.77
			AP- B	151	95.9			
E3	Alkyd	0.718	Untreated	10952	--	--	--	--
			AP-A	1991	81.8	85.6	5.37	6.27
			AP- B	1159	89.4			
			IP	1138	89.6	--	--	--
E4	Alkyd	0.306	Untreated	4998	--	--	--	--
			AP-A	903	81.9	81.4	0.70	0.86
			AP- B	952	80.9			

[a] Treated for seven days (concentrations of the untreated coating materials are the averages of duplicate samples.).

[b] Spiked concentrations, calculated by wet weight.

[c] AP (active paste); IP (inactive paste).

4.3 Tests for Caulk Materials

The two samples of field caulk used in this study were collected from an unoccupied building scheduled for demolition. Based on the previous measurements (Guo et al., 2011), the concentrations of Aroclor 1254 in these samples differed by an order of magnitude. Preparation of the laboratory-made caulk is described in Section 2.3.2. After treatment, two slices of caulk were cut from each sample (Figure 4.1). Then, they were extracted and analyzed for PCBs separately.

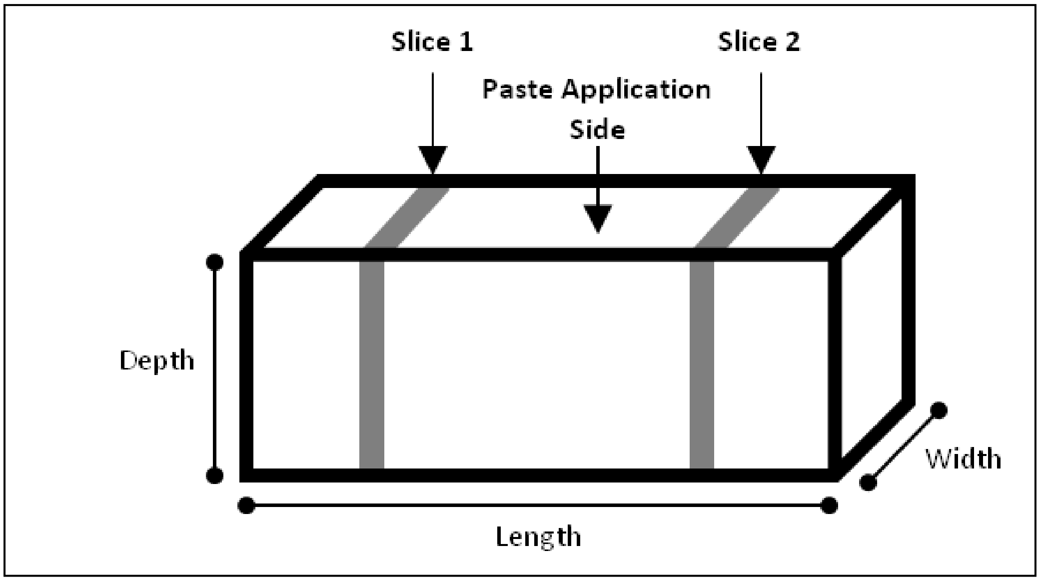

Figure 4.1. Caulk slices for extraction and analysis

Table 4.5. Summary of the concentrations of PCB congeners (µg/g) and Aroclor 1254 (w/w) measured by GC/MS in the original caulk [a]

Analytes	**Test ID and Matrix**			
	E7 **Field Caulk 1**	**E8** **Field Caulk 2**	**E9** [b] **Lab-prepared Caulk 1**	**E10** [b] **Lab-prepared Caulk 2**
PCB-17 (µg/g)	25.4	2.38	NR	2.02
PCB-52 (µg/g)	3224	463	NR	161
PCB-101 (µg/g)	6233	925	NR	237
PCB-154 (µg/g)	724	124	NR	26.1
PCB-110 (µg/g)	6692	1031	NR	241
PCB-77 (µg/g)	19.0	3.16	NR	0.63
PCB-66 (µg/g)	710	121	NR	27.9
PCB-118(µg/g)	5433	893	NR	206
PCB-105 (µg/g)	2440	424	NR	92.3
PCB-187 (µg/g)	239	38.1	NR	10.1
Aroclor 1254 (% w/w)	9.62	1.52	NR	0.38
Aroclor 1254 (µg/g)	96200	15200	NR	3800

[a] Average of four slices from duplicate samples; NR means “not reported” due to DQI failure.

[b] Data for cured laboratory-made caulk.

All pieces of caulk samples were treated with AMTS paste for five to ten days (See Section 2.2.1). The efficiency of the AMTS treatment was calculated using Equation (4.1), and the results for the field-caulk tests are summarized in Tables 4.6 and 4.7.

Table 4.6. Percent removal efficiencies for PCB congeners for field caulk after AMTS treatment [a]

Test ID	%Aroclor 1254 (w/w) [b]	Sample ID [c]	%Efficiency [e]	Average	SD	%RSD
E7	9.62	AP 5-day A [d]	16.2	20.0	0.03	17.2
		AP 5-day B	23.0			
		AP 5-day C	20.7			
		AP 10-day A	6.45	9.45	0.04	9.45
		AP 10-day B	12.5			
		IP 5-day	19.3	--	--	--
E8	1.52	AP 5-day A	18.8	24.2	0.10	40.6
		AP 5-day B	18.3			
		AP 5-day C	35.6			

[a] Treated for five to ten days (Two slices were analyzed for each sample.) .

[b] Concentration in the untreated samples.

[c] AP (active paste application); IP (inactive paste application).

[d]. 5-day means that that treatment time was 5 days.

[e] Average efficiency of ten target congeners.

Table 4.7. Percent removal efficiencies for Aroclor 1254 for field caulk after AMTS treatment [a]

Test ID	%Aroclor 1254 (w/w)	Sample ID [b]	Concentration (μg/g)	%Efficiency	Average	SD	%RSD
E7	9.62	Untreated	96200	--	--	--	--
		AP 5-day A [c]	83086	13.6	19.4	0.05	25.6
		AP 5-day B	74715	22.3			
		AP 5-day C	74914	22.1			
		AP 10-day A	92817	20.8	7.8	0.06	77.5
		AP10-day B	84615	3.5			
		IP5-day	76150	12.0	--	--	--
E8	1.52	Untreated	15240	--	--	--	--
		AP 5-day A	12376	18.8	23.1	0.11	48.9
		AP 5-day B	13025	14.5			
		AP 5-day C	9778	35.8			

[a] Treated for five to ten days (Two slices were analyzed for each sample.). Concentrations of untreated field caulk are the averages of duplicate samples.

[b] AP (active paste application); IP (inactive paste application).

[c]. 5-day means that the treatment time was 5 days.

For each slice of the field caulk, four measurements were made to obtain the average depth of the slice. Two slices were cut from one piece of field caulk and analyzed for PCBs. Thus, the average depth of two slices was used to represent the depth of each piece of the field caulk. The AutoCAD measurements of the depths of the caulk samples are summarized in Table 4.8. The difference in the depths for each piece of field caulk was less than 8% of RSD with the exception of one case in which the difference was 21% of RSD. However, the difference between different pieces of the caulk samples was larger because the pieces of field caulk were not as uniform as the laboratory-made caulk samples.

Most of the data collected from tests of the laboratory-made caulk did not meet the DQI, so they were not reported. The failed data led to a negative value of %Efficiency for the AMTS method when the measured concentration after the treatment was higher than the measured concentration before the treatment.

Table 4.8. Summary of depth (height) measurements of pieces of field caulk

Test ID	Sample ID [a]	Depth (mm) [b]	Average (mm)	SD	%RSD
E7	AP 5-day A Slice1 [c]	4.56	4.65	0.13	2.74
	AP 5-day A Slice2	4.74			
	AP 5-day B Slice 1	5.27	5.15	0.18	3.40
	AP 5-day B Slice 2	5.03			
	AP 5-day C Slice 1	5.41	5.53	0.17	3.10
	AP 5-day C Slice 2	5.65			
	AP 10-day A Slice 1	4.80	4.17	0.89	21.3
	AP 10-day A Slice 2	3.54			
	AP 10-day B Slice 1	7.29	6.92	0.52	7.56
	AP 10-day B Slice 2	6.55			
	IP 5-day Slice 1	5.67	5.99	0.46	7.61
	IP 5-day Slice 2	6.31			
E8	AP 5-day A Slice 1	5.11	5.31	0.28	5.33
	AP 5-day A Slice 2	5.51			
	AP 5-day B Slice 1	3.28	3.33	0.07	2.10
	AP 5-day B Slice 2	3.38			
	AP 5-day C Slice 1	6.26	5.97	0.40	6.75
	AP 5-day C Slice 2	5.69			

[a] AP (active paste application); IP (inactive paste application). [b] Average of four measurements from one slice. [c] 5-day means that the treatment time was 5 days.

4.4 Tests for Concrete

Tests AMTS-E11 and AMTS-E12 were conducted to determine the efficiency of the AMTS method for laboratory-made concrete. The procedure for preparing the concrete samples is described in Section 2.2.4. The initial concentrations of the target congeners and Aroclor 1254 are summarized in Table 4.9.

Table 4.9. Summary of the concentrations of the PCB congeners and Aroclor 1254 in the untreated, laboratory-made concrete measured by GC/MS [a]

Analytes	Units	Test ID	
		E11	E12
PCB-17	µg/g	1.25	0.25
PCB-52	µg/g	106	28.7
PCB-101	µg/g	184	53.2
PCB-154	µg/g	21.4	5.87
PCB-110	µg/g	196	56.3
PCB-77	µg/g	0.46	0.12
PCB-66	µg/g	18.1	4.90
PCB-118	µg/g	159	46.1
PCB-105	µg/g	72.0	20.3
PCB-187	µg/g	0.44	1.68
Aroclor 1254	% (w/w)	0.29	0.082
Aroclor 1254	µg/g	2900	820

[a] Average of four slices from duplicate samples.

All laboratory-made concrete coupons were treated with AMTS paste for seven days. The efficiency of the AMTS treatment was calculated using Equation (4.1), and the results are summarized in Tables 4.10 and 4.11.

Table 4.10. Average percent removal efficiencies of the AMTS treatment for PCB congeners in laboratory-made concrete [a]

Test ID	%Aroclor 1254 (w/w) [b]	Sample ID [c]	%Efficiency [d]	Average	SD	%RSD
E11	0.30	AP 7-day A[e]	55.8	54.4	0.02	3.67
		AP 7-day B	53.0			
		IP 7-day A	41.3	46.9	0.08	17.1
		IP 7-day B	52.6			
E12	0.08	AP 7-day A	31.1	39.1	0.11	28.8
		AP 7-day B	47.0			

[a] Treated for seven days. [b] Concentration calculated by wet weight. [c] AP (active paste), IP (inactive paste).
[d] Average efficiency of ten PCB congeners. [e] 7-day means that the treatment time was 7 days.

The dimensions of each of the concrete coupons were about the same because they were prepared by using the stainless steel mold (Figure 2.12). The depths of the concrete coupons were determined by measuring seven blank coupons using the AutoCAD software. The average of the seven measurements was 6.13 mm, and this value was used as the depth of all of the concrete coupons. The standard deviation of these seven measurements was 0.20 mm.

Table 4.11. Removal efficiencies of Aroclor 1254 after AMTS treatment of laboratory-made concrete [a]

Test ID	Aroclor 1254 % (w/w) [b]	Sample ID [c]	Concentration (μg/g)	Removal Efficiency			
				%Efficiency	Average	SD	%RSD
E11	0.30	Untreated	2879	--	--	--	--
		AP 7-day A [d]	1380	52.1	50.7	0.02	3.89
		AP 7-day B	1460	49.3			
		IP 7-day A	1839	36.1	42.5	0.09	21.2
		IP 7-day B	1472	48.9			
E12	0.08	Untreated	818	--	--	--	--
		AP 7-day A	594	27.4	36.5	0.13	35.3
		AP 7-day B	445	45.6			

[a] Treated for seven days (Concentrations of the original laboratory-made concrete are the averages of duplicate samples.).

[b] Concentration calculated by wet weight.

[c] AP (active paste application), IP (inactive paste application).

[d]. 7-day means that the treatment time was 7 days.

4.5 Partial Dechlorination Investigation

The potential formation of partially-dechlorinated PCB congeners after treatment by the active paste was examined in Test AMTS-E13. The test was designed to determine whether there were any PCB-209 degradation products in the primer after the AMTS treatment. PCB congeners were quantified as homologues. The original concentrations of the PCB homologues in the primer are listed in Table 4.12

Table 4.12. Concentrations of PCB homologues in the untreated primer measured by GC/MS [a]

Analytes	Concentration (µg/g)
PCB-209 spiked	2980 [b]
PCB-Cl#1	~~0.00~~
PCB-Cl#2	~~0.08~~
PCB-Cl#3	~~0.02~~
PCB-Cl#4	~~0.00~~
PCB-Cl#5	~~0.00~~
PCB-Cl#6	~~0.14~~
PCB-Cl#7	5.63
PCB-Cl#8	~~0.00~~
PCB-Cl#9	29.56
PCB-Cl#10 (PCB-209)	1924

[a] Test E13; average of duplicate samples; values in strikethrough are below the PQL.

[b] Equivalent to 0.298% (w/w).

After treatment with active and inactive pastes, there was a significant reduction of PCB-209 in all samples. The PCB-209 concentration reduced by more than 90% after 1 day of treatment with active paste and 95% after 3 days of inactive paste treatment. However, the duplicate samples did not meet the DQI (i.e., ≤ 25%), so no data were reported here. The compounds detected during the test, using the primer as an example, are presented in the chromatograms in Figure 4.2. Neither biphenyl nor major PCB homologues were detected in the primer and the paste after the AMTS treatment. Biphenyl's absence could not be explained by the results, be it due to biphenyl's further degradation or due to biphenyl not being a major reaction product. More study is needed to better understand the reaction mechanism.

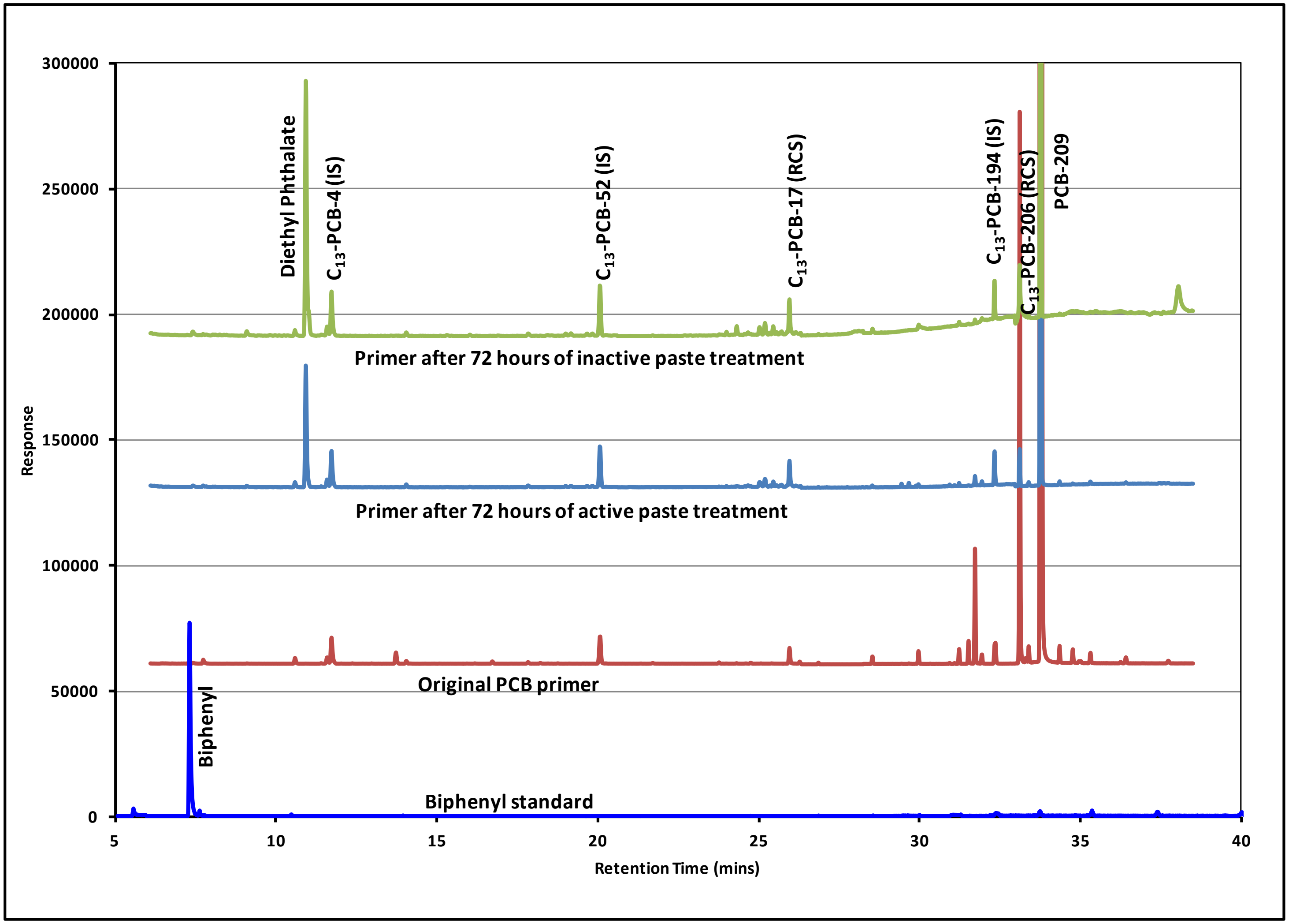

Figure 4.2. Chromatograms (SIM mode) of compounds detected in the reduction of PCB-209 by the AMTS method

(The peak for PCB-209 is off scale with response = 1.76×10^7)

5. Discussion

5.1 General Performance of the AMTS Method

The results of laboratory testing for the AMTS method, presented in Section 4, demonstrate that the method removed PCBs from coating materials, i.e. primer and alkyd, efficiently (Figure 5.1). The removal efficiency was greater than 80% for all samples after seven days of treatment. This result is consistent with the results in a previous report on this issue (Krug et al., 2010). Our test results indicated that the AMTS method is more efficient for removing PCBs from the primer than from the alkyd paint.

The percent removal efficiencies were lower for thicker materials including caulk and concrete. For the field caulk that was tested, the AMTS method removed 12 to 36% of the PCB congeners (4 to 36% in terms of Aroclor 1254). For the laboratory-made concrete, the removal efficiency ranged from 39 to 54% for the PCB congeners (27 to 52% in terms of Aroclor 1254).

For each substrate, the AMTS method was evaluated at two different PCB concentration levels. The results showed that the removal efficiencies for paint, field caulk, and laboratory-mixed concrete were not affected significantly by the concentrations of the PCBs.

Due to a problem that occurred during the tests for epoxy paint and the laboratory-mixed caulk, we were unable to assess the performance of the AMTS method for these materials.

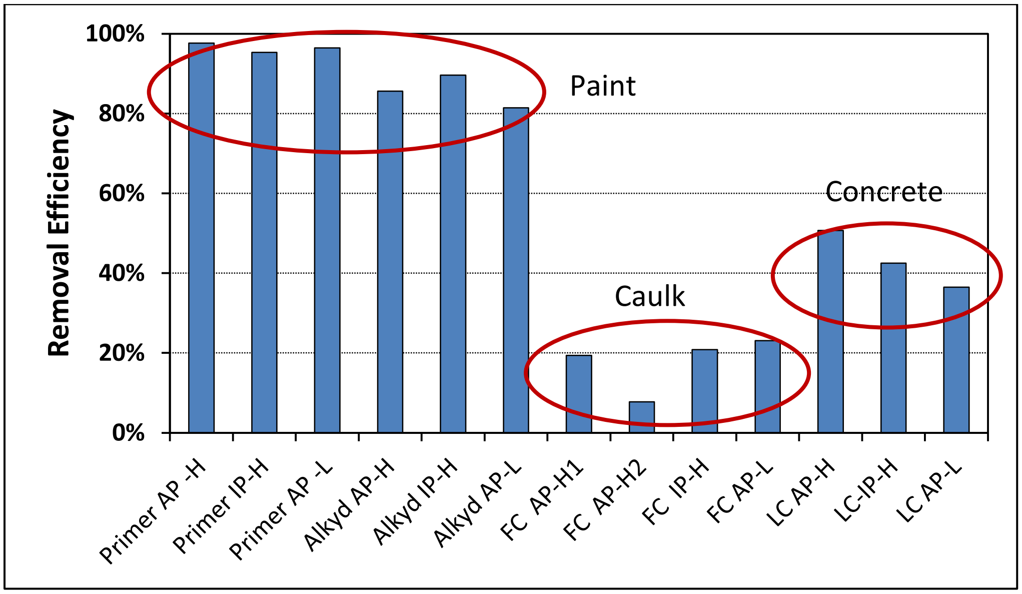

Figure 5.1. Efficiency of the AMTS method used on different materials

(AP-active paste, IP-inactive paste, H-high PCB concentrations, L-low PCB concentrations, FC-field caulk, LC-laboratory-prepared concrete)

5.2 Effective Penetration Depth

The percent removal efficiency is an appropriate indicator for the performance of the AMTS method for coating materials, but it may be misleading for thick sources such as caulk and concrete, because the depths to which the solvents in the paste can penetrate are limited. As a result, the percent removal efficiency is affected by the thickness of the source. To resolve this problem, the concept of "effective penetration depth" is introduced. The effective penetration depth is the thickness of a layer of the source material near the treated surface in which all PCBs are removed and beyond which the PCBs remain intact (Figure 5.2).

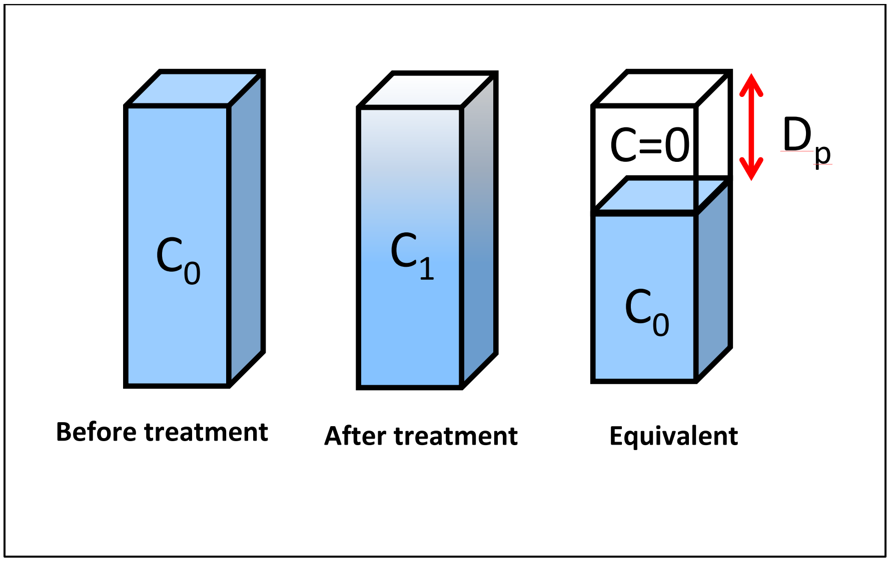

Figure 5.2. Schematic diagram of effective penetration depth (D_p)

As an idealized case, we assume that the PCB concentration in the caulk or concrete is uniform initially and that the AMTS treatment is 100% effective within the effective penetration depth and it has no effect beyond the effective penetration depth. If only one side of the source is treated by the AMTS, the effective penetration depth is defined by Equation 5.1:

$$D_p = L\left(1 - \frac{C_1}{C_0}\right), \tag{5.1}$$

where: D_p = effective penetration depth (mm)

L = thickness of the source (mm)

C_0 = uniform concentration of PCBs in the source before treatment (µg/g)

C_1 = average concentration of PCBs in the source after treatment (µg/g)

Although calculating the effective penetration depth (D_p) requires the thickness of the material (L), the effective penetration depth itself is not a function of the thickness. In fact, a major advantage of employing this parameter as a measure of removal efficiency over other parameters, such as percent reduction, is that the effective penetration depth is independent of the thickness of the caulk. As illustrated in Table 5.1, if two pieces of caulk are of the same type, same length, and same width but have different thicknesses, the percent reduction after treatment will be different (see the second row from bottom in the table), but the effective penetration depth will be the same (the bottom row). In this report, the concept of the effective penetration depth serves two purposes: (1) to evaluate the performance of the AMTS method for thick sources, and (2) To use this parameter in the barrier model as described in Section 5.3. This parameter is not designed for use in the field.

Table 5.1. Comparison of removal efficiencies expressed as percent efficiency and effective penetration depth using hypothetical values [a]

Parameters	Sample 1	Sample 2
Sample thickness , L (cm)	0.5	1.0
Sample area for treatment, A (cm^2)	1	1
Density of material, ρ (g/cm^3)	1	1
Concentration of PCBs before treatment, C_0 (μg/g)	100	100
Amount of PCBs before treatment, W_0 (μg) [b]	50	100
Amount of PCBs removed (μg) after treatment	10	10
Amount of PCBs remaining after treatment, W (μg)	40	90
Average concentration of PCBs after treatment, C_1 (μg/g) [c]	80	90
Percent removal efficiency, $[(C_0 - C_1) / C_0] \times 100$	20	10
Effective penetration depth, $D_p = L (C_0 - C_1) / C_0$, (mm)	1.0	1.0

[a] Two pieces of caulk of the same type and area but with different thicknesses.

[b] $W_0 = C_0 \times \rho \times L \times A$.

[c] $C_1 = W / \rho / (L \times A)$.

The effective penetration depths for field caulk and the laboratory-made concrete were calculated using Equation (5.1), and the results are summarized in Tables 5.2 and 5.3.

Table 5.2. Effectiveness of the AMTS method expressed as effective penetration depth for the field caulk

Test ID	%Aroclor 1254 (w/w) [a]	Sample ID [b]	Sample Thickness (mm)	Effective penetration depth (D_p)			
				D_p (mm)	Average	SD	%RSD
E7	9.62	AP 5-day A	4.65	0.63	1.00	0.32	32.0
		AP 5-day B	5.15	1.15			
		AP 5-day C	5.53	1.22			
		AP 10-day A	6.92	0.24	0.48	0.34	70.1
		AP 10-day B	5.99	0.72			
		IP 5-day	4.17	0.87	--	--	--
E8	1.52	AP 5-day A	5.31	1.00	1.21	0.85	70.2
		AP 5-day B	3.33	0.48			
		AP 5-day C	5.97	2.14			

[a] Concentration determined by GC/MS. [b] AP (active paste), IP (inactive paste).

Table 5.3. The effectiveness of the AMTS method expressed as effective penetration depth for the laboratory-made concrete

Test ID	%Aroclor 1254 (w/w) [a]	Sample ID[b]	Sample Thickness (mm)	Effective penetration depth (D_p)			
				D_p (mm)	Average	SD	%RSD
E11	0.30	AP 7-day A	6.13	3.19	3.11	0.12	3.89
		AP 7-day B	6.13	3.02			
		IP 7-day A	6.13	2.22	2.61	0.55	21.2
		IP 7-day B	6.13	3.00			
E12	0.08	AP 7-day A	6.13	1.68	2.24	0.79	35.3
		AP 7-day B	6.13	2.80			

[a] AP (active paste), IP (inactive paste).

5.3 Predicting the "Bleed-back" of PCBs from Treated Sources

5.3.1 Model Description

After treatment by the AMTS method, PCBs are essentially removed from the top layer of the source, and this layer acts as a barrier to the migration of PCBs from the untreated portion of the source, thereby reducing the concentrations of PCBs at the surface of the source and in the room air. As a practical matter, it is important to know whether the "bleed-back" of PCBs is a concern for the AMTS method. A fugacity-based barrier model, developed by Yuan et al. (2007), was used to predict the bleed-back of PCBs from treated sources. The details of the model are described in Section 5 of Part 3 of this report series, entitled *Laboratory Study of Polychlorinated Biphenyl (PCB) Contamination and Mitigation in Buildings, Part 3. Evaluation of the Encapsulation Method* (Guo et al., 2012b). When the AMTS treatment process is applied, only the treated side of the source is exposed to room air, and the distance from inside the PCB source to the surface of the top layer is defined as x (Figure 5.3).

A classroom scenario was assumed for the simulation, and the parameters are listed in Table 5.4 and Figure 5.3.

Table 5.4. Classroom scenario for the simulation using the fugacity-based barrier model

Parameter	Symbol	Value	Units	Notes
Classroom volume	V	100	m^3	
Exposed surface area of the PCB caulk	A_1	0.5	m^2	
Thickness of the original PCB caulk	L_{T1}	0.02	m	20 mm
Exposed surface area of the PCB concrete	A_2	10	m^2	
Thickness of the original PCB concrete	L_{T2}	0.01	m	10 mm
Concentration of PCBs in the inlet air	y_{in}	0	$\mu g/m^3$	
Air change flow rate	Q	2.78×10^{-2}	m^3/s	= 100 m^3/h

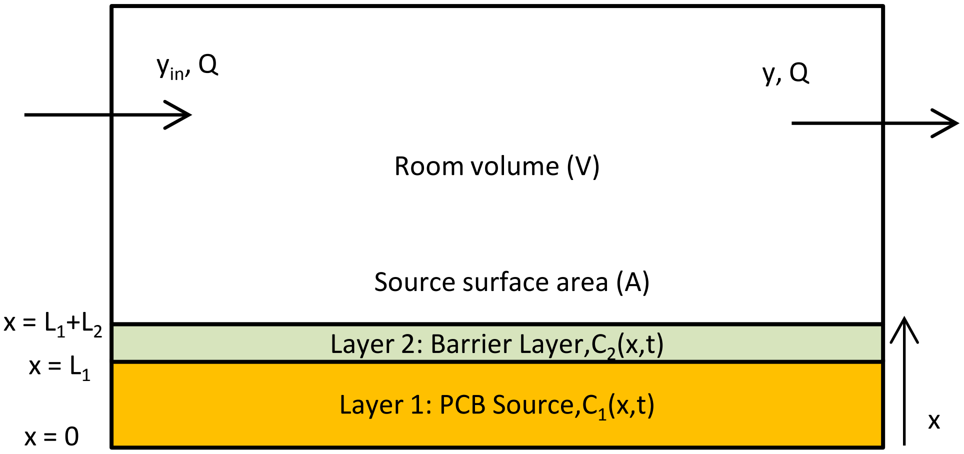

Figure 5.3. Schematic classroom scenario for the fugacity-based barrier model (Yuan et al., 2007)

5.3.2 Simulations for Treated Caulk

In addition to the average effective penetration depth obtained from the experiments (Section 5.2) for five days of active AMTS paste treatment, other parameters used for the simulation by the model are listed in Table 5.4 and 5.5. The values are for congener #110, the most abundant congener in Aroclor 1254. The effective penetration depths presented in Section 5.2 were used as the thickness of the barrier layer.

Table 5.5. Input parameters for the fugacity-based barrier model for PCB caulk (PCB-110)

Parameter	Symbol	Value	Units	Notes
Thickness of the source layer (L_{T1}-D_p)	L_1	0.019	m	19 mm [a]
Thickness of the barrier layer (D_p)	L_2	0.001	m	1 mm [b]
Molecular weight of the contaminant (congener #110)	MW	3.27×10^8	μg/mol	= 327 g/mol
Initial concentration in the source layer	C_{01}	8.03×10^9	$\mu g/m^3$	= 6692 μg/g [c]
Initial concentration in the barrier layer (AMTS treated)	C_{02}	0	$\mu g/m^3$	
Diffusion coefficient	D_1, D_2	2.98×10^{-15}	(m^2/s)	[d]
Material/air partition coefficients	K_1, K_2	3.64×10^7	dimensionless	[d]

[a] $L_{T1} = L_1 + L_2$ is the original thickness of the AMTS-treated caulk sample. [b] Based on experimental data.
[c] Assuming the density of the caulk is 1.2 g/cm^3; concentration based on experimental data for field caulk 1.
[d] Data from Guo et al. (2011); in this case, $D_1 = D_2$, $K_1 = K_2$.

Figure 5.4 shows the concentration profile of congener #110 in the PCB source caulk as a function of time. The x-axis is the direction of the PCB concentration from the bottom of the PCB source caulk to the interface of the source and the layer from which the PCBs have been removed.

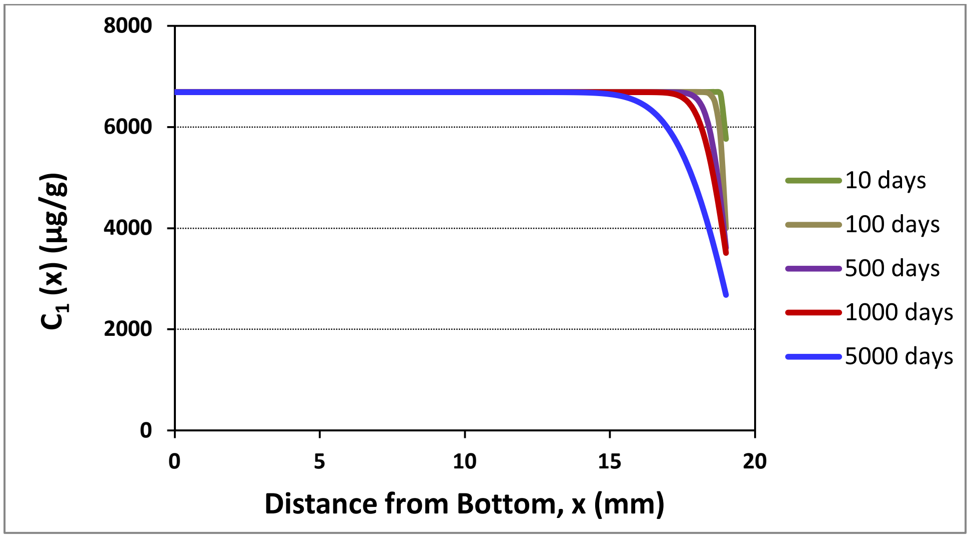

Figure 5.4. Concentration profiles [C_1(x)] for congener #110 in the bottom layer (i.e., below the effective penetration depth) of the PCB caulk after treatment by the active AMTS paste

(The effective penetration depth is between x = 19 mm and x = 20 mm)

The profile of the congener #110 in the top layer of the caulk from which the PCBs have been removed is shown in Figure 5.5. The x-axis is the direction from the source-barrier interface to the surface of the barrier. The concentration of the PCB at x = 0 mm (i.e., the interface of the source and barrier) decreases over time. The calculations performed by the model showed that the concentration of the PCB at x = 1 mm (i.e., at the surface of the barrier that is exposed to air) increases at first and then decreases over time.

The average concentration of PCBs as a function of time in the top layer from which the PCBs had been removed is shown in Figure 5.6. The contaminant accumulates in the barrier in the early days, after which there is a slow decrease. The decrease is caused mainly by the concentration gradient formed at the interface of the source and the barrier.

Figure 5.7 shows the concentration of PCBs at the exposed surface of the caulk from which PCBs had been removed by the AMTS treatment. This concentration at the surface is directly linked to the contribution of the treated source to the PCB concentration in the air. Note that, in this case, the concentration at the exposed surface is much less than the average concentration in the barrier layer (Figure 5.6).

Figure 5.8 shows the contribution of the encapsulated source to the PCB concentration in the room air, following the same pattern as the PCB concentration on the surface and in the barrier layer, i.e., the PCB concentration increases first and then slowly decreases.

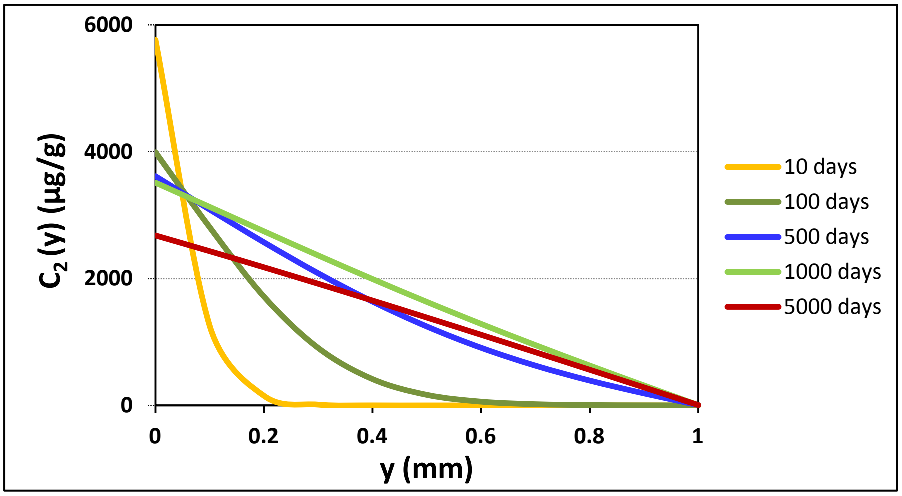

Figure 5.5. Concentration profiles for congener #110 as a function of depth in the top layer of the caulk from which the congener had been removed by the AMTS treatment

(The effective penetration depth is between y = 0 mm and y = 1 mm. The caulk/air interface is at y = 1 mm.)

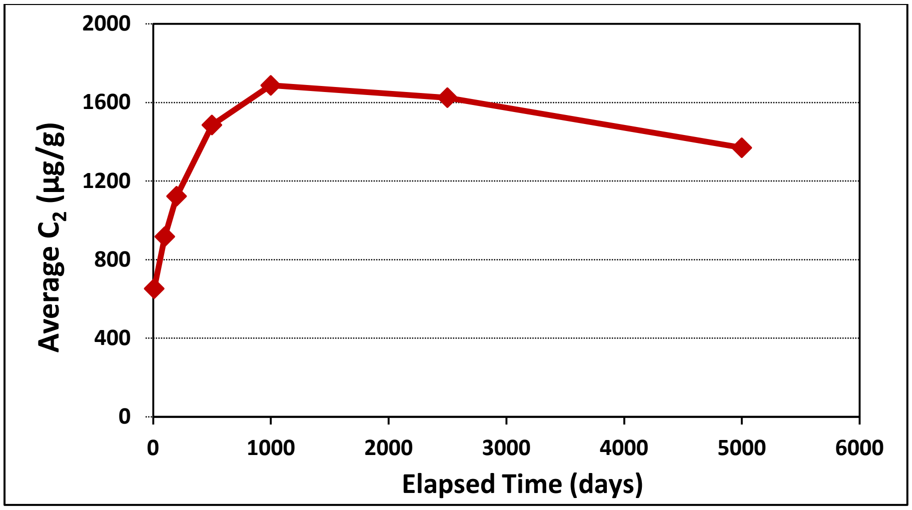

Figure 5.6. Average concentration of congener #110 as a function of time (C_2) in the top layer of the caulk from which the congener had been removed by treatment with AMTS

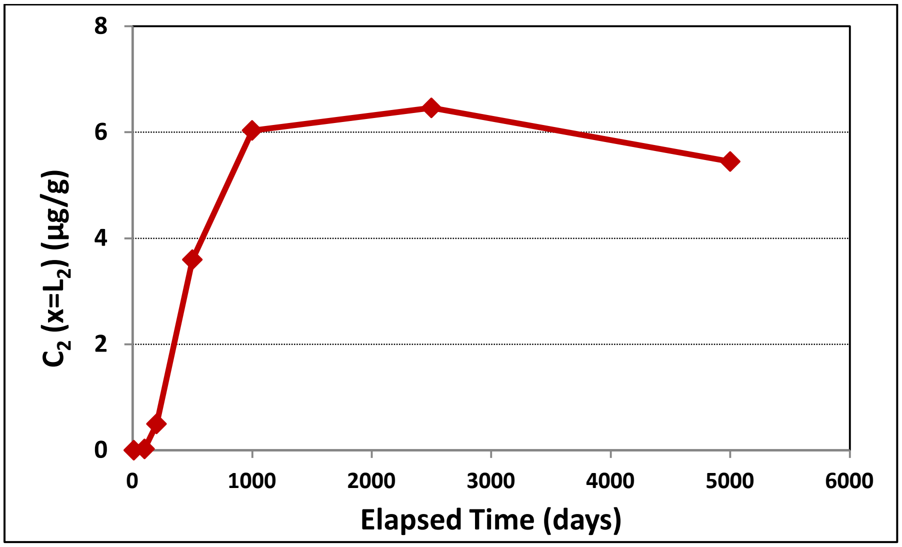

Figure 5.7. Concentrations of congener #110 at the air exposed surface of the caulk [C_2 ($x=L_2$)] as a function of time

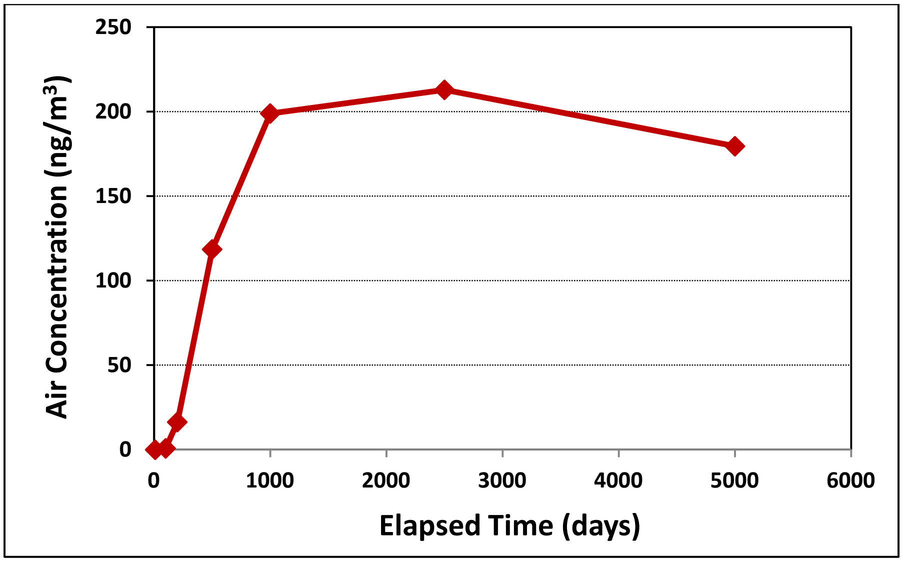

Figure 5.8. Concentrations of congener #110 in bulk room air as a function of time due to emissions from the treated PCB caulk assuming that the air is well mixed

5.3.3 Simulations for Treated Concrete

The parameters used for simulation of the results of treating PCB-containing concrete with the AMTS method are listed in Tables 5.4 and 5.6.

Table 5.6. Input parameters for the fugacity-based simulation model for PCB-containing concrete (PCB-110)

Parameter	Symbol	Value	Units	Notes
Thickness of the source layer (L_{T2}-D_p)	L_1	0.007	m	7 mm [a]
Thickness of the barrier layer (D_p)	L_2	0.003	m	3 mm [b]
Molecular weight of the contaminant (congener #110)	MW	3.27×10^8	µg/mol	= 327 g/mol
Initial concentration in the source layer (C_0)	C_{01}	3.91×10^8	µg/m^3	=196 µg/g [c]
Initial concentration in the barrier layer (AMTS treated)	C_{02}	0	µg/m^3	
Diffusion coefficient	D_1, D_2	4.00×10^{-15}	(m^2/s)	[d]
Partition coefficient between the source layer and air	K_1, K_2	6.95×10^7	dimensionless	[d]

[a] $L_2 = D_p$; $L_{T2} = L_1 + L_2$ is the total thickness of the AMTS-treated concrete sample. [b] Based on experimental data. [c] Assuming the density of the concrete is 2 g/cm^{3,} concentration is based on experimental data. [d] Data from Guo et al. (2012a); in this case, $D_1 = D_2$, $K_1 = K_2$

Figure 5.9 shows the PCB-110 concentration in the concrete as a function of time and depth. The blue dotted line in the figure separates the top layer and the bottom layer.

Profiles of the contaminant in the concrete layer from which the PCBs had been removed by treatment with AMTS are shown in Figure 5.9. Figure 5.10 shows the average concentration of PCBs as function of time in the concrete layer from which the PCBs had been removed by treatment with AMTS. The PCB concentrations in the exposed surface of the concrete from which the PCBs had been removed are shown in Figure 5.11. The PCB-110 concentration in room air from emissions from the concrete surface as a function of time is shown in Figure 5.12. For comparison, Figures 5.10 through 5.12 also show the concentrations for untreated concrete. The figures show that the PCB concentrations in the concrete layer from which PCBs had been removed, on the surface of that layer, and in the room air were still increasing after 5000 days. The difference that exists between caulk and concrete may be due to the combined effects of the partition and diffusion coefficients and the effective penetration depth.

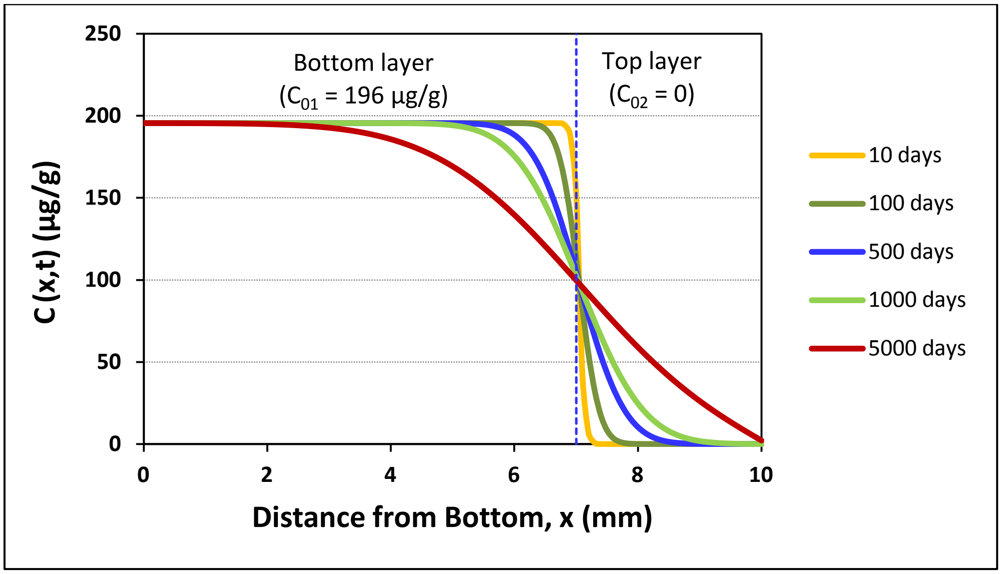

Figure 5.9. Concentration profiles for congener #110 in the PCB-containing concrete as a function of time (t) and depth (x), [C(x,t)], after AMTS treatment

(The concrete/air interface is at y = 10 mm. C_{01} and C_{02} are the initial concentrations after treatment in the bottom and top layers, respectively. Effective penetration depth is between x = 7 mm and x = 10 mm)

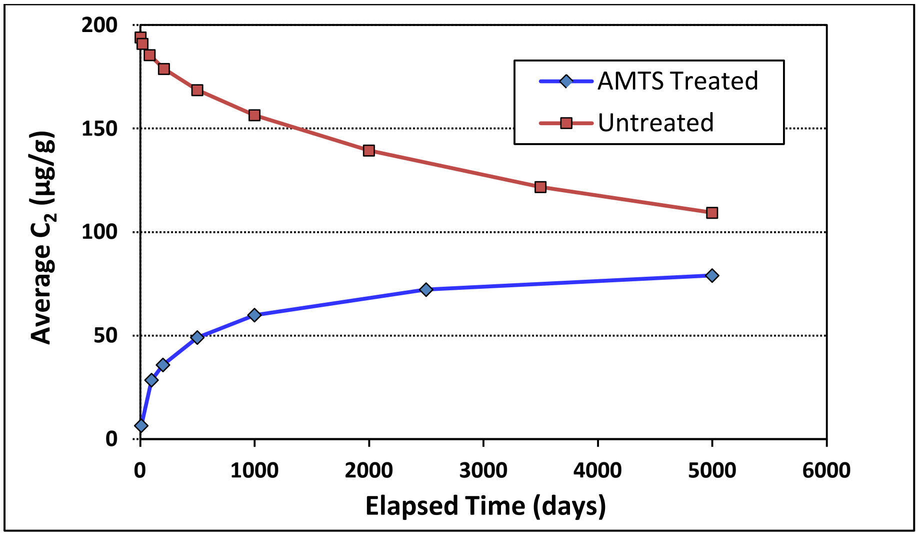

Figure 5.10. Average concentration of congener #110 as a function of time (C_2) in the top layer of the concrete from which the congener had been removed by treatment with AMTS

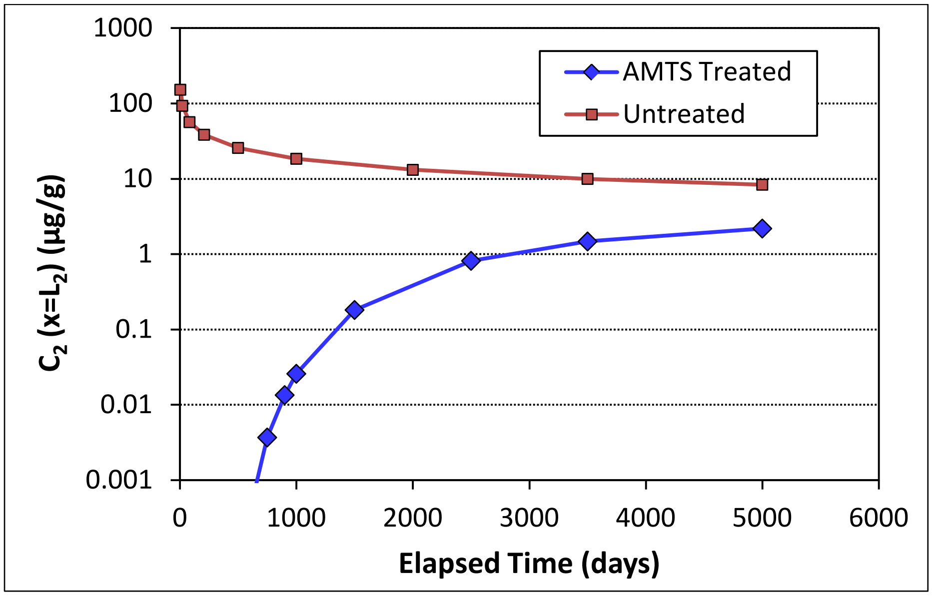

Figure 5.11. Concentrations of congener #110 at the surface of the concrete that is exposed to air [$C_2(x = L_2)$] as a function of time

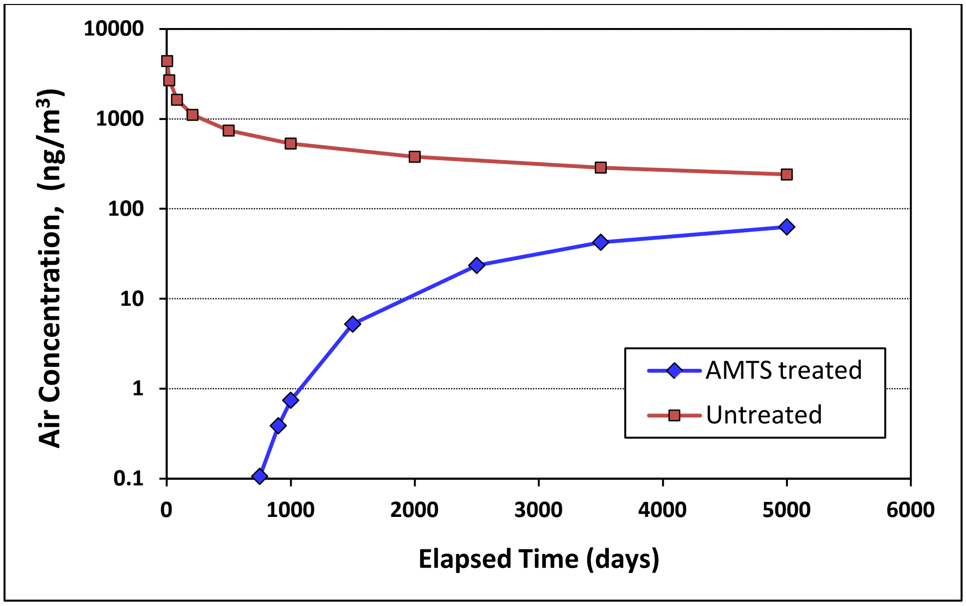

Figure 5.12. Concentration of congener #110 in bulk room air as a function of time due to emissions from the treated concrete assuming that the air is well mixed

5.3.4 Summary of Mathematical Modeling

After treatment by the AMTS method, the top layer of a thick source becomes almost free of PCBs. This layer acts as a barrier that impedes the migration of PCBs from the deep layers to the surface, thereby reducing the concentration at the surface of the source and emissions to the room air. Whether this barrier can provide adequate protection to the occupants is determined by several factors, including the initial concentration of PCBs in the source, the environmental quality criteria that are selected, the effective penetration depth, and the partition and diffusion coefficients for the substrate. For PCB concentrations in room air, the area of the source and the ventilation rate are also important parameters. Because the effective penetration depths for AMTS (See Section 5.2) is greater than the thickness of most encapsulants (Guo et al., 2012b), the barrier created by the AMTS may perform better and lasts longer than the encapsulants for the same source. However, the “bleed-back” of PCBs remains of concern for AMTS treated sources when the initial PCB concentration in the source is high. Therefore, post-treatment environmental monitoring, such as periodical air and wipe sampling, is necessary. The dependence of PCB bleed-back on the initial concentration and the thickness of the encapsulant is described in Guo et al. (2012b).

5.3.5 Limitations of Mathematical Modeling

The simulation results presented above should be considered semi-quantitative because the partition and diffusion coefficients used were rough estimates and because the predictions of the model have not been verified. In addition, it was assumed that the PCB concentration in the source was uniform initially, which is an oversimplification of the real-world situation.

5.4 Reaction Mechanisms

According to the literature (DeVor et al., 2009 and Krug et al., 2010), the mechanisms for PCB destruction by the AMTS method include the generation of hydrogen and dechlorination (Figure 5.13). A key step of these reactions is the replacement of a chlorine atom in the PCB molecule by a hydrogen atom.

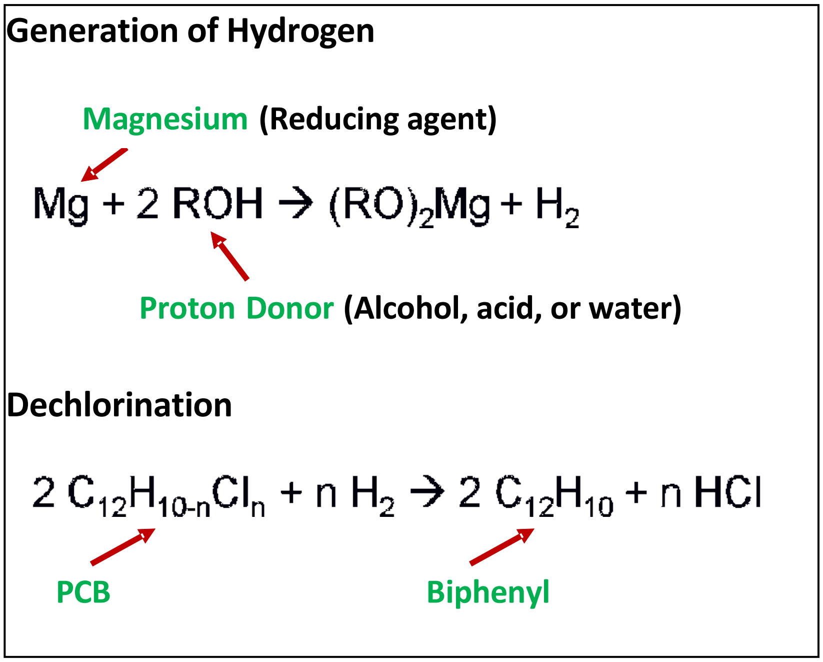

Figure 5.13. PCB destruction mechanisms proposed in the literature

If the reactions in Figure 5.13 represent the major mechanisms for PCB destruction, then there is a chance for incomplete chlorine stripping (i.e., generation of PCB congeners with fewer chlorine atoms). According to the reaction mechanism in Figure 5.13, the disappearance of one mole of PCBs yields one mole of biphenyl if the PCBs are completely dechlorinated. Thus, ascertaining the quantity of biphenyl that is formed and determining whether less-chlorinated PCB congeners are present can shed light on the reaction mechanisms associated with the AMTS process.

In this study, PCB-209 was spiked into a primer and treated with the AMTS paste to test the above hypotheses. Significant reduction of PCB-209 was observed (> 90%) after 24 hours of treatment. However, neither biphenyl nor any other major, less-chlorinated PCB congeners were detected. Further investigation is needed to determine whether other reaction byproducts are formed in the process and whether any of the byproducts are of environmental concerns.

5.5 Organic Solvents

The AMTS pastes contain ethanol, and the Dupli-Color® Undercoat (used as an encapsulant for the paste) contains toluene, mineral spirits, and asphalt. The potential effects of these chemicals on indoor air quality and their potential for posing a fire hazard should be evaluated when the pastes are applied inside buildings in large quantities.

5.6 Study Limitations

This study was conducted in a relatively short period of time, about 3 months, and only 13 tests were conducted with four types of substrates, i.e., coating materials, field caulk, laboratory-made caulk, and laboratory-made concrete.

Among the laboratory-made PCB sources that we tested, the data for the epoxy coating and caulk did not meet the data quality goals and, thus, were not reported.

The PCB-containing concrete samples were made in the laboratory with the PCBs mixed in the concrete during concrete preparation. Mixing PCBs into concrete during initial preparation of the concrete is not representative of the conditions under which PCBs are found in the concrete or masonry of actual buildings.

The parameters used for the barrier models, including solid/air partition coefficients and solid-phase diffusion coefficients, are rough estimates based on previous experiments (Guo et al., 2011; 2012a). The effective penetration depths used in the simulations were the average of experimental data that contained considerable uncertainty (See Tables 5.2 and 5.3). The fact that the precision results did not meet the DQI for the tests does not necessarily mean that the data quality was unacceptable. Rather, it might imply that the concentrations of PCB congeners in the test materials were uneven.

The study of the reaction mechanism for the AMTS was exploratory, and did not provide evidence for the formation of biphenyl or less chlorinated PCBs. Further study is needed to understand the reaction mechanism better.

There are several important areas that this study did not investigate. They include: (1) the effectiveness of multiple treatments; (2) the properties and conditions of the source materials after being treated with AMTS; (3) the PCB residual concentration in the active paste after treatment.

This study evaluated the AMTS technology that was available in early 2011. Since then, the developer of this method has conducted research aimed to improve the performance of the method for contaminated masonry materials by modifying the formulation and application procedure (Quinn, 2012). This study did not evaluate these new methods.

6. Conclusions

Active and inactive AMTS pastes were tested in the laboratory with four types of PCB-containing substrates, i.e., coating materials, field caulk, laboratory-made caulk, and laboratory-made concrete. The performance of the AMTS method was evaluated based on the PCB removal efficiency and the effective penetration depth [Sections 2 and 4].

The test results showed that the AMTS method is highly efficient (> 80 %) for removing PCBs from the coating materials we tested (i.e., the primer and alkyd paint) even for initial PCB concentration levels of several thousand parts per million. The AMTS method was more efficient in removing PCBs from the primer than from the alkyd paint. Our findings were consistent with the results reported from an earlier study (Krug et al., 2010) [Section 4.2].

The high removal efficiencies obtained with the primer and the alkyd paint were associated with relatively thin sources. For thick sources, i.e., the field caulk and the laboratory-made concrete, the AMTS method was less effective in its ability to remove PCBs because of its limited effective penetration depth. The treatment removed 12 to 36% of the target congeners (4 to 36% in term of Aroclor 1254) in the field caulk that contained 15200 and 96200 ppm Aroclor 1254, and it removed 39 to 54% of the target congeners (27 to 52% in term of Aroclor 1254) in the laboratory-made concrete that contained 820 and 2900 ppm Aroclor 1254 [Sections 4.3 and 4.4].

The AMTS method was evaluated for two different PCB concentration levels in the four substrates. The results showed that the PCB removal efficiencies for the two paints, the field caulk, and the laboratory-made concrete were affected very little by the initial PCB concentrations, i.e., whether the concentrations were high or low [Sections 4.2, 4.3, and 4.4].

The effective penetration depth, which is independent of the thicknesses of the AMTS-treated materials, was determined for the field caulk and the laboratory-made concrete. The average effective penetration depth for the field caulk was about 1 mm with 50% RSD, and the average effective penetration depth for the laboratory-made concrete was about 3 mm with 16% RSD [Section 5.2].

After the AMTS treatment, the top layer of a thick source from which the PCBs had been removed acted as a temporary barrier to the migration of the PCBs from the untreated portion of the source substrate to the treated surface. The fugacity-based barrier model is a useful tool for predicting the “bleed-back” of PCBs, which can be a limiting factor for treating thick sources that contain high concentrations of PCBs. Therefore, post-treatment environmental monitoring is necessary [Section 5.3].

While PCB reduction via the substitution of hydrogen for one of the chlorine atoms in the PCB molecule has been depicted as the major mechanism for the AMTS method, the possibility exists that other reactions, as yet unidentified, may also occur. Further work will be required to confirm that no hazardous byproducts (e.g., partially-dechlorinated PCBs) are generated during application of the AMTS method [Sections 4.5 and 5.4].

Overall, the AMTS method has the potential to become a viable method for mitigating PCB contamination in buildings. This method is promising for treating contaminated masonry materials near the expansion joints after the caulking material is removed because the AMTS can treat sources that contain several thousand ppm PCBs, for which the encapsulation method may not be effective. However, more research is needed because the current method has limited effective penetration depths for thick sources, including masonry materials. In addition, the reaction mechanism needs to be verified. The potential effect of the method on indoor air quality during the treatment also needs further evaluation.

7. Recommendations

The authors recommend the following topics for future research.

Evaluate the effectiveness of the latest AMTS method for treating masonry materials. One of the major tasks in mitigating PCB contamination in buildings is to deal with the contaminated masonry materials adjacent to PCB caulking. This report showed that the AMTS method had limited effective penetration depth for concrete after a single treatment. Efforts have been made by the developer of the AMTS method to improve its performance for PCB-contaminated masonry materials. This improved method should be evaluated because, if the effective penetration depth can be significantly increased, the AMTS method may play a role in treating contaminated masonry materials in buildings.

Verify the reaction mechanisms. The reaction mechanisms proposed by the developer of the AMTS method (Figure 5.13) produce one mole of biphenyl for each mole of PCBs destroyed. In this study, exploratory experiments were conducted to determine the formation of biphenyl. However, the results were negative (Section 4.5). The authors recommend verifying the reaction mechanism by a combination of GC/MS analyses and mass balance for chlorine. The GC/MS analyses help identify specific by-products formed. According to Figure 5.13, complete dechlorination of one mole of a PCB congener that contains n chlorine atoms will yield n moles of HCl. Thus, a chlorine mass balance may provide a definitive answer to the question.

Evaluate the advantages and disadvantages of using active and inactive pastes. As described in Sections 1.1 and 2.1, the active AMTS paste extracts and degrades PCBs in a single step while the inactive paste in two separate steps. It has been reported that, unlike the inactive paste in which the extracted PCBs react with the reducing agent in a container after the treatment, the active paste cannot degrade the extracted PCBs to below 50 mg/kg if the PCB concentration in the source is very high (>20,000 mg/kg) (Krug et. al., 2010). The advantages and disadvantages of using the two types of pastes should be further evaluated under realistic application conditions.

Select the cover paint that is suited for indoor uses. The current AMTS method uses a rubberized coating material to encapsulate the paste applied to the substrate to prevent the solvents from evaporation. This coating material is not intended for indoor applications and should be replaced by one that has minimum effects on indoor air quality.

Conduct field measurements. Future evaluation in the laboratory should be complemented by field measurements in PCB-contaminated buildings. Effects on the physical properties of the substrates after multiple treatments should be examined. The evaluation should include analysis of PCB residual concentrations in the active paste after treatment because it is a limiting factor for treating sources with high PCB content.

Evaluate other chemical methods. Chemical methods other than the AMTS for on-site treatment of PCB-contaminated materials need to be identified and evaluated.

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