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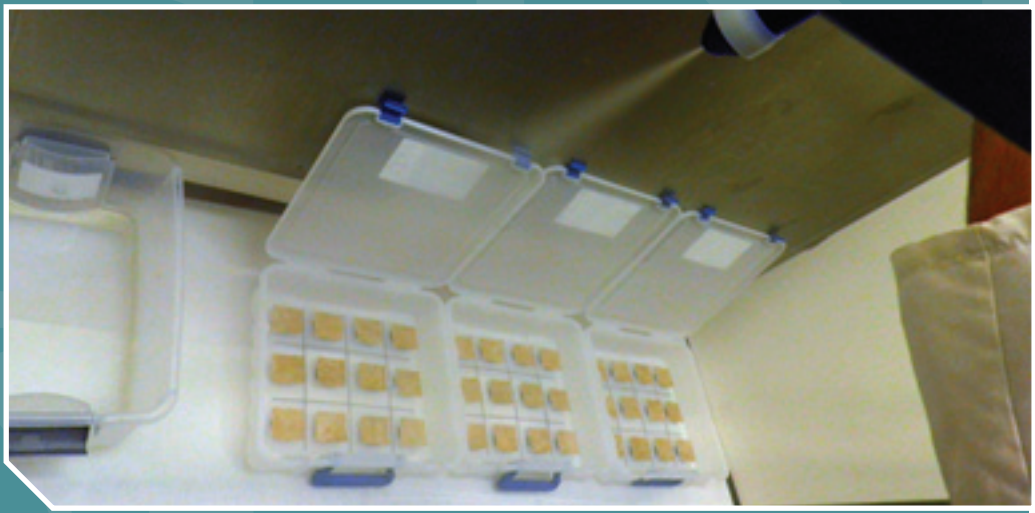
Application of Electrostatic and Backpack Sprayer Systems for Decontamination of Building Materials Contaminated with Malathion

ASSESSMENT AND EVALUATION REPORT



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National Homeland Security Research Center

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Assessment and Evaluation Report

National Homeland Security Research Center
Office of Research and Development
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

Disclaimer

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List of Acronyms and Abbreviations

BPS	backpack sprayer
°C	degrees centigrade (Celsius)
CAS	Chemical Abstracts Service
CBRN	Chemical, Biological, Radiological, and Nuclear
CCV	continuing calibration verification
CMAD	Consequence Management Advisory Division
cm^3	cubic centimeter(s)
CWA	chemical warfare agent
DQO	data quality objective
EPA	U. S. Environmental Protection Agency
ERT	Environmental Response Team
ESS	electrostatic sprayer system
FAC	free available chlorine
g	gram(s)
GC/MS	gas chromatography/mass spectrometry
HSRP	Homeland Security Research Program
ICAL	internal calibration (standard)
IS	internal standard
kHz	kilohertz
L	liter(s)
LB	laboratory blank
MDL	method detection limit
min	minute(s)
mL	milliliter(s)
µg	microgram(s)
ND	non-detect
NHSRC	National Homeland Security Research Center
NIST	National Institute of Standards and Technology
OEM	Office of Emergency Management
ORD	Office of Research and Development
OSRTI	Office of Superfund Remediation and Technology Innovation
OSWER	Office of Solid Waste and Emergency Response
PB	procedural blank
PC	positive control
pg	picogram(s)
P/N	part number

QA	Quality Assurance
QC	Quality Control
QAPP	Quality Assurance Project Plan
RF	response factor
SD	standard deviation
SIM	selected ion monitor
SS	stainless steel
TC	test coupon
VMD	volume median diameter
VX	O-ethyl S-[2-(diisopropylamino)ethyl] methylphosphonothioate

Executive Summary

This project supports the mission of the U.S. Environmental Protection Agency Office of Research and Development's Homeland Security Research Program (HSRP) by providing information relevant to the decontamination of areas contaminated as a result of a chemical contamination incident. The primary objective of this investigation was to evaluate the use of two different types of spray systems to supply a decontaminant to a surface.

The evaluation tests described in this report determined the effectiveness of an electrostatic sprayer system in delivery of a decontaminant to clean a building material contaminated with a toxic chemical. Efficacy results using the electrostatic sprayer for neutralization of the chemical from three building materials were compared to the results obtained using the more traditional backpack sprayer system. Electrostatic sprayers are more efficient than conventional spray systems, and they deliver a more uniform distribution of the liquid on an (uneven) surface. In this study, the targeted decontaminant was full-strength bleach and the chemical was malathion, a pesticide and surrogate for the chemical warfare agent (CWA) O-ethyl S-[2-(diisopropylamino)ethyl] methylphosphonothioate (VX). Full-strength bleach was selected over more traditional diluted bleach (typically 10 fold dilution) based on outcomes from previous bench scale research in which poorer decontamination efficacy was observed against materials contaminated with CWAs HD and VX [1]. Malathion was applied uniformly as a thin film or as more discrete droplets of low or high concentration. The impact on the efficacy of these different malathion distribution patterns was also investigated. Three building material substrates were included, namely, stainless steel, wood, and vinyl.

Summary of results:

The decontamination efficacies as obtained using the electrostatic and backpack sprayer systems are shown in Figure ES-1. For stainless steel surfaces, both spray systems were able to decontaminate the surface by achieving mostly non-detectable malathion amounts after a 30 min dwell time of the full-strength bleach applied to the contaminated material. The results indicate that efficacies were not dependent on the actual mode of malathion distribution.

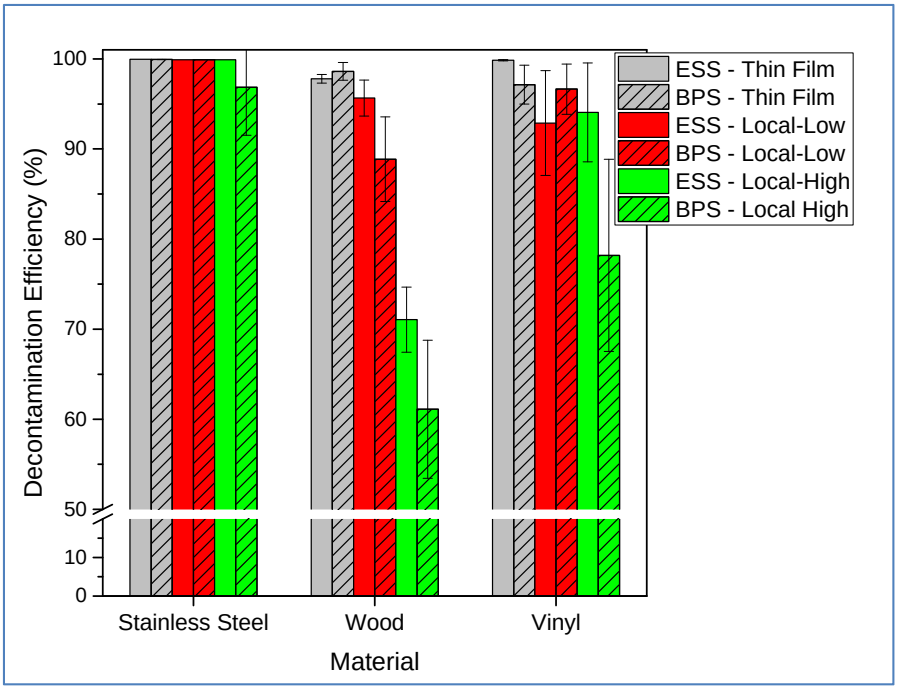

Figure ES-1. Decontamination Efficacies for Malathion-Contaminated Materials Using Bleach Dispensed from an Electrostatic Sprayer (ESS) and Backpack Sprayer (BPS)

The efficacy of bleach for decontamination of malathion on wood is lower for all modes of malathion distribution when compared to the results of decontamination of stainless steel with the lowest efficacy observed when malathion is present as localized, high concentration droplets. For wood, the electrostatic sprayer appeared to perform better than the backpack sprayer, but the difference was not statistically significant (Student's t-test $p>0.05$). Efficacy values for vinyl are between the results observed for stainless steel and wood with a better performance when using the electrostatic sprayer for two of the three modes of malathion distribution. For both wood and vinyl, the malathion distribution has an impact on the observed efficacy. Highly concentrated (malathion) droplets are more difficult to decontaminate than a more evenly distributed film.

Impact of this study

Both sprayer systems performed equally well at decontaminating flat surfaces. The more uniform initial distribution of the decontamination solution on the surface as applied by the electrostatic sprayer does not result in a significantly better efficacy under the tested conditions. Localized high concentrations of chemicals are more difficult to decontaminate and may require repeated application of a given decontamination technology.

1 Introduction

This project supports the mission of the U.S. Environmental Protection Agency's (EPA) Office of Research and Development's (ORD) Homeland Security Research Program (HSRP) by providing information, expertise and products that can be widely used to prevent, prepare for, and recover from public health and environmental emergencies arising from terrorist threats and incidents. EPA researchers initiated bench scale studies to tailor the decontamination practices for surfaces contaminated with chemical warfare agents (CWAs), to the environment and material affected, type of chemical encountered, and to determine the effect on materials using operationally relevant decontamination dwell times. This project is a first effort to scale up decontamination methods from small surfaces (coupon sizes typically less than 10 cm^2) to surface sizes of approximately 1 square foot. This project supports the HSRP mission in determining the effectiveness of undiluted bleach for the removal of malathion, selected as a pesticide and surrogate for the CWA O-ethyl S-[2-(diisopropylamino)ethyl] methylphosphonothioate (VX), from building surfaces using two different spray/fine-mist decontaminant delivery methods.

1.1 Project Objectives

The objective of this study was to determine the decontamination efficacy of bleach as a target decontaminant for the removal of malathion as applied to three building materials (stainless steel (SS), wood, and vinyl). Two decontamination spray methods, an electrostatic sprayer (ESS) system and a backpack sprayer (BPS) system, were evaluated for three building materials and three malathion deposition methods, namely, a thin-film application, an evenly distributed low concentration of droplets, and an evenly distributed high concentration of droplets.

The test matrix for this effort consisted of 18 pairings (three material types, three malathion deposition methods, and two decontamination spray methods). Three (3) replicate test coupons (TCs), three (3) replicate positive control coupons (PCs), one (1) procedural blank (PB), and one (1) laboratory blank (LB) were included in each test - a total of eight (8) coupons for each material/malathion deposition condition/decontamination method combination. A single 30-minute (min) interaction time between the bleach and malathion was used for all 18 tests.

2 Experimental Approach

2.1 Overview of the Experimental Approach

The decontamination efficacy of the bleach solution was assessed by determining the amount of malathion remaining on each test coupon after a 30-min decontamination period using bleach, by comparing this result to the amount recovered from the positive control coupons, which were contaminated but not decontaminated at the same time as the test coupons.

The amount of malathion remaining on the surface was quantified via chemical extraction of the coupon followed by measurement of the concentration of the target chemical through gas chromatographic/mass spectrometric (GC/MS) analysis. The mean decontamination efficacy along with the standard deviation was calculated for each set of replicates and controls. The standard deviation of the efficacy was calculated by propagation of error using the standard deviation of the average mass of agent remaining on the test coupons and on the positive control coupons.

At the end of the 30-min decontamination interaction time for the decontamination product with the malathion, the decontamination process was quenched through extraction of the remaining chemical by neutralizing the decontamination chemical through dilution. The quenching of the decontamination reaction was accomplished by dropping all twelve coupons that constitute one sample into a beaker and adding 150 milliliters (mL) of extraction solvent (hexane) followed by a 10-min sonication. After completion of the sonication and a short (5-10 min) time period to separate solvent phases, an aliquot of the extraction solvent was diluted (10 fold) further prior to analysis. This second dilution step ensures a completely quenched decontamination process. This is critical as the chemical analysis typically occurs hours to days later which is a significantly longer time period than the 30-min decontamination interaction time at the surface. A non-quenched reaction would potentially bias decontamination efficacy measurements.

2.2 Decontamination Efficacy Determination

The decontamination efficacy was determined by measuring the amount of residual malathion on the test coupons and comparing this amount with positive controls (spiked with malathion, not decontaminated and analyzed after the same “contact time” as the test coupons) using Equation 1:

$$E = \left(1 - \frac{M_i \text{ on Test Coupon}}{M_a \text{ on Positive Control Coupon}}\right) \times 100\% \qquad (1)$$

where:

E = % Efficacy

M_i = Measured mass of malathion on the ith test coupon (micrograms [µg]);

M_a = Average measured mass of malathion on the control coupons (µg).

2.3 Statistical Analysis

The standard deviation in the measured mass of malathion is calculated as shown in Equation 2:

$$\sigma = \sqrt{\frac{\sum_{i=1}^{N}(x_i-\mu)^2}{N-1}} \quad (2)$$

where:

σ = standard deviation

μ = mean

x_i = i^{th} value of the variable being evaluated, e.g., control coupon

N = total number of elements in the population.

2.4 Test Setup

Decontamination solution testing occurred in a conventional chemical safety hood. Twelve (12) building material coupons (2” x 2” size) were placed in an individual 14” x 14” transparent test tray to provide representative coverage of an approximately 12” x 12” test area. The use of multiple smaller coupons to represent a larger surface area instead of a single 12” x 12” coupon significantly facilitates the extraction process for the material. The surface of each test tray had a grid to allow for a reproducible coupon arrangement pattern. This coupon arrangement was used for the even distribution of droplets for contamination scenario tests (Figure 2-1). For the thin-film application, an airbrush type of application was used to apply malathion in ethanol solution onto twelve individual 2”x 2” size coupons. In that case, the coupons were placed closely together to reduce variation in the amount of malathion received during the airbrush spraying between individual coupons. (Figure 2-2).

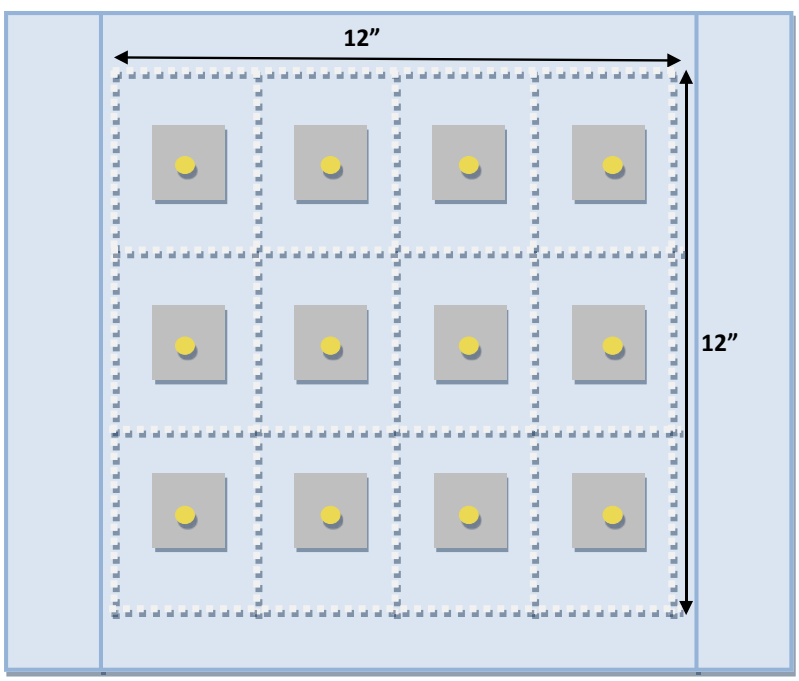

Figure 2-1. Coupon Configuration for the Even-Distribution Droplet Contamination Scenario.

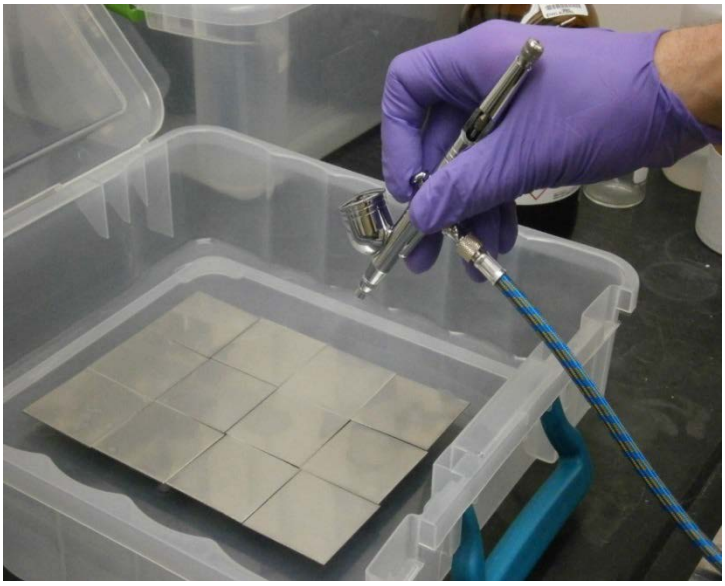

Figure 2-2. Coupon Configuration during the Thin Film Contamination.

2.5 Test Materials and Characteristics

The representativeness and uniformity of test materials are essential in achieving statistically defensible evaluation results. Material representativeness means that these materials are typical of those currently used in buildings in terms of quality, surface characteristics, and structural integrity. In this effort, representativeness was assured by selecting test materials typical of the materials found in residential dwellings that meet industry standards or specifications for indoor use, and by obtaining those materials from appropriate suppliers. Material uniformity means that all these material coupons are equivalent for purposes related to testing. Uniformity was maintained by obtaining and preparing a quantity of material sufficient to allow multiple test samples to be prepared with presumably uniform characteristics (i.e., test coupons were cut from the interior rather than the edge of a large piece of material). A description of the selected building materials for decontamination testing is provided in Table 2-1.

Table 2-1. Description of Building Materials for Decontamination Testing

Material	Description	Manufacturer/ Supplier Name, Location	Coupon Surface Size, L x W (inches)	Material Preparation
Stainless Steel	Multipurpose Stainless Steel (48 x 48 inch), type 304, #2B mill (unpolished), 0.036 inch thick	McMaster-Carr, Elmhurst, IL, USA	2" x 2" organized into 12" x 12" test area	- Remove any lubricant/grease from shearing with acetone and wipe dry - Remove particles and dust by wiping clean with water and wipe dry
Wood	Untreated Pine Plywood, 0.5" thick	Plytanium/ Lowe's, Durham, NC, USA	1.6" x 1" organized into 12" x 12" test area	- Remove particles by wiping clean with water and wipe dry
Vinyl flooring	Traffic MASTER Allure (12 x 24 inch) Ivory Travertine, vinyl tile; residential grade, stain resistant, scratch resistant, 0197 inch thick	Armstrong/ Home Depot, Durham, NC, USA	2" x 2" organized into 12" x 12" test area	- Remove particles by wiping clean with water and wipe dry

SS coupons were prepared using heavy duty power hydraulic shears to cut the metal from larger sheets to the correct length and width. Lubricant from the shears was removed using acetone and wiped dry.

2.6 Coupon Contamination

2.6.1 Chemical Agent

The target chemical agent was malathion (Chemical Abstracts Service (CAS) # 121-75-5), which is considered to be a surrogate for the CWA VX (O-ethyl S-[2-(diisopropylamino)ethyl] methylphosphonothioate). The chemical agent malathion (part number (P/N): N-12346-100MG, Chem Services Inc., West Chester, PA) was purchased in neat liquid form (>98% purity) and initially dissolved in ethanol (Sigma-Aldrich, St. Louis, MO Cat # 459828-1 liter (L) spectrophotometric grade) to 50/50 percent by volume. Malathion was further dissolved in ethanol at 12, 50 or 500 mg/mL concentration, depending on each spatial coupon deposition approach. The stock solutions were made through the transfer of the contents of a malathion ampoule to a pre-weighed GC vial, capped and weighed again. Ethanol was then added to produce a solution of the desired concentration. The vial was then mixed for approximately 30 sec using a vortex mixer.

2.6.2 Coupon Contamination

Three types of malathion contamination were evaluated for all material/decontamination combinations:

1. Application of malathion as a thin film, applied by an airbrush technique
2. Application by microsyringe of evenly distributed low concentration droplets of diluted malathion solution
3. Application by microsyringe of evenly distributed high concentration droplets of diluted malathion solution.

A Master Performance Pro Dual-Action Gravity Feed Airbrush with a 0.2 mm nozzle (Item #: MAS G-233-SET, TCP Global, San Diego, CA), shown in Figure 2-3, was used for the thin film application of malathion solution to the surface of the coupons.

Figure 2-3. Master Performance Pro Dual-Action Gravity Feed Airbrush

As is visible in Figure 2-2, a set of twelve 2” x 2” size coupons of the same material was uniformly contaminated with malathion solution. An approximately 70-second long sweeping spray motion (left to right across the twelve coupons first; then up and down across the same coupons, followed by a spray on the circumference) was used to deliver the stock solution in a uniform manner. Upon completion of the malathion solution application, the weight of the test tray containing the set of twelve coupons was measured and compared against the tray weight with coupons prior to the application of the stock solution. This weight increase was converted to applied volume using the density of ethanol [0.789 grams (g)/cubic centimeter (cm^3)] as the main constituent of the solution. The average volume applied to a set of twelve coupons throughout the test program was 4.0 ± 0.5 mL.

The evenly distributed low concentration and the evenly distributed high concentration droplets of diluted malathion solution were applied using a 25 microliter (μL) syringe (P/N: 702, Hamilton, Reno, NV) and a 5 μL syringe (P/N: 7105, Hamilton, Reno, NV), respectively.

The amount and concentration of solution deposited on a set of twelve coupons is shown in Table 2-2 for each malathion deposition method.

Table 2-2. Contamination Solution Properties

Spatial Distribution of Contamination Scenario	Stock Solution Concentration	Volume of Solution per Test Coupon/Positive Control Coupon	Theoretical Amount of Malathion Applied (mg)
Even distribution-high concentration	500 mg/mL	24 µL (12 x 2 µL)	12
Even distribution-low concentration	50 mg/mL	240 µL (12 x 20 µL)	12
Thin Liquid Film though an airbrush	12 mg/mL	70 seconds spray/4 mL	48

2.7 Decontamination Testing

The overall decontamination effectiveness was determined for the three malathion deposition conditions as a function of the material type and type of decontamination spray apparatus (ESS or BPS) for a 30-min interaction time with the test coupons (contaminated) or procedural blank coupons.

2.7.1 Decontamination Solution

Concentrated bleach solution (hereafter, "bleach") (Clorox® Concentrated Germicidal Bleach, The Clorox® Company, Oakland, CA) was purchased from a local retail store. As per label, the bleach contains 8.25% sodium hypochlorite. The "bleach" definition used in this report refers to this product as received without dilution or pH adjustments. Details on the concentrated Germicidal Bleach decontamination solution are shown in Table 2-3. Germicidal bleach was selected over other concentrated Clorox® bleach products based on its more frequent use in biological remediation efforts.

Table 2-3. Decontamination Solution Properties

Solution	Manufacturer/ Supplier Name, Location	Active ingredients	pH Range
Bleach (8% sodium hypochlorite)	Clorox® Concentrated Germicidal Bleach (The Clorox® Company), Oakland, CA, USA	Hypochlorite ion/ hypochlorous acid /hydroxide ion [bleach stabilizer]	11-12

2.7.2 Decontamination Approach

Two decontamination spray methods (ESS and BPS systems) were evaluated for the three materials/malathion deposition methods. The amount of bleach applied was derived from the gain in weight of the sample setup box (which includes the test coupons) following the application of the bleach.

2.7.2.1 Electrostatic Sprayer

For ESS applications, a self-contained disinfection sprayer system (SC-1 electrostatic sprayer, Electrostatic Spraying Systems ESS, Watkinsville, GA, USA), shown in Figure 2-4, was used in this study.

Figure 2-4. Picture of SC-1 Electrostatic Sprayer.

This air-assisted ESS (22” height [H] x 18” wide [W] x 10.5” length [L]) produces electrically charged spray drops that are carried to the target in a low pressure, gentle, air stream. The SC-1 ESS system is intended for light-duty, quick disinfection and sanitization applications and compatible with most conventional chemicals. The ESS is equipped with a patented MaxCharge™ technology electrostatic spray gun that delivers droplets with a volume median diameter (VMD) of 40 µm at a flow rate of 3.8 liters (L) per hour. The electrostatic charge induced by the MaxCharge™ nozzle is strong enough to allow the droplets to move in any direction to cover surfaces homogeneously. Air-assisted electrostatic spray technology gives more than twice the deposition efficiency of hydraulic sprayers and non-electrostatic types of air-assisted sprayers. Prior to testing, the spray distance was set to cover the whole 12”x12” test area. The spray volume was determined for each decontamination test by collecting the entire sprayed volume into a graduated cylinder of the appropriate size. The ESS was used for the decontamination of three trays at a time containing coupons with an average mass flow rate of 75.4 ± 5.8 g/min over a one (1) min spray time, leading to an average mass loading of bleach in each tray of 25.1 ± 2.5 g. The ESS flow rate was checked at the start and at the end of each set of spray applications and the drift, if any, was recorded.

2.7.2.2 Electric Backpack Sprayer

An SHURFLO® SRS-600 ProPack rechargeable backpack sprayer BPS (approximately 36” H x 24” W x 6” L), SHURflo®, Cypress, CA, (Figure 2-5) was used as an alternative to a handheld sprayer. The mass flow rate of was set to achieve a mass of bleach in each tray comparable to the ESS. The BPS was used for the decontamination of three trays at a time containing coupons with an average mass flow rate of 607 ± 68 g/min over a ten (10) second spray time, leading to an average mass loading of bleach in each tray of 33.8 ± 7.9 g. The backpack sprayer flow rate was checked at the start and at the end of each set of spray applications and the drift, if any, was recorded.

Figure 2-5. Electric Backpack Sprayer

2.8 Extraction Solvent

Hexane (OmniSolv® HX0296, EMD Millipore, Billerica, MA; or Optima™ H303, Fisher Scientific, Pittsburgh, PA) was used as the diluting medium for both the chemical and the decontaminant. The volume of solvent that was used in each test was dispensed via a bottletop dispenser (P/N: BRAND 4730351 US, Sigma-Aldrich, St. Louis, MO).

2.9 Decontamination Testing

2.9.1 Test Matrix

Table 2-4 shows the test matrix containing the malathion distribution method, materials tested, and decontamination spray type.

Table 2-4. Building Material Surface Decontamination Test Matrix

Test ID	Chemical Contamination Scenario	Decontamination Application	Test Material	Total # of Coupons
E-1	Even distribution-high concentration droplets	ESS	Stainless steel	8
E-2	Even distribution-low concentration droplets	ESS	Stainless steel	8
E-3	Thin-film application	ESS	Stainless steel	8
E-4	Even distribution-high concentration droplets	ESS	Wood	8
E-5	Even distribution-low concentration droplets	ESS	Wood	8
E-6	Thin-film application	ESS	Wood	8
E-7	Even distribution-high concentration droplets	ESS	Vinyl flooring	8
E-8	Even distribution-low concentration droplets	ESS	Vinyl flooring	8
E-9	Thin-film application	ESS	Vinyl flooring	8
H-1	Even distribution-high concentration droplets	BPS	Stainless steel	8
H-2	Even distribution-low concentration droplets	BPS	Stainless steel	8
H-3	Thin-film application	BPS	Stainless steel	8
H-4	Even distribution-high concentration droplets	BPS	Wood	8
H-5	Even distribution-low concentration droplets	BPS	Wood	8
H-6	Thin-film application	BPS	Wood	8
H-7	Even distribution-high concentration	BPS	Vinyl flooring	8
H-8	Even distribution-low concentration	BPS	Vinyl flooring	8
H-9	Thin-film application	BPS	Vinyl flooring	8

Each test consisted of three test coupons (contaminated and decontaminated), three positive controls (contaminated but not decontaminated), one procedural blank (not contaminated but decontaminated) and a laboratory blank (not contaminated and not decontaminated). The decontamination solution was applied 30 min following the application of the malathion. Such an amount of time was chosen to allow for some weathering of the chemical into the material (as applicable).

2.9.2 Extraction Method for Coupons

After completion of each test, sets of twelve 2 in x 2 in coupons (test coupons or positive controls) were carefully transferred into a custom-made extraction coupon holder made of SS wire and Teflon (Figure 2-6). Extraction of SS and vinyl coupons occurred in a 250 mL glass beaker with 150 mL hexane.

Figure 2-6. Stainless Steel Coupons during Extraction

The wood coupons were too thick to fit in this custom-made coupon holder. Instead, the wood coupons were placed face up in the bottom (single layer) of a 400 mL beaker to which 150 mL hexane was added. Once all coupons of the set were placed in individual beakers with hexane, the beakers were covered with aluminum foil and sonicated at 40 kilohertz (kHz) for 10 min. The temperature of the water in the sonic bath was monitored at least once every 2 min. If the temperature of the bath increased to more than 40 degrees Celsius (ºC), the water was replaced to prevent overheating of samples/avoid excessive evaporation of extraction solvent during sonication. Losses in extraction solvent following sonication were compensated by adding solvent to the same marked level. Significant losses (more than 10%) were documented.

After extraction, a 100 µL aliquot of the raw extract was transferred to a 1.8 mL GC vial, preloaded with hexane spiked with isotopically-labeled internal standards. This transfer occurred shortly after the end of the sonication process. The additional dilution of samples reduces the chance that residual bleach may continue to interact with the malathion in the extract.

An aliquot of 100 µL of the extract was added to a preloaded sample vial with 880 µL of hexane, 10 µL of a 50 mg/mL d-10 malathion solution in dichloromethane (P/N DLM-76-1.2, CIL Inc, Tewksbury, MA), and 10 µL of a 50 mg/mL of Internal Standards Mixture (P/N ERS-091, Sigma-Aldrich, St. Louis, MO). The deuterated malathion was added as a pre-injection labeled surrogate, to check for variations and irregularities in the instrumental analysis.

All extraction samples were stored at 4 ºC ± 3 ºC until analysis by GC/MS. Vials were marked at the solvent level so that sample integrity could be maintained by verification of the solvent level by the EPA/NRMRL Organic Support Laboratory, who conducted the GC/MS analyses.

2.10 Sample Identification

Each sample was identified by a description of the technology tested and a unique sample number. Table 2-5 specifies the sample identification.

Table 2-5. Sample Identification

Coupon Identification: 67-A#-B-CC(CC)-DD(x min)-SS-N		
ID	Code	Description
A# *Test ID*	E1	Test ID per Table 2-4, Neutralization test would be N1, N2, etc...
B *Material Type*	S	Stainless steel
	W	Wood
	F	Vinyl flooring
CC (CC) *Process type*	EE	Extraction efficiency testing
	BC (NT)	Neutralization testing
	BC (ST)	Solution testing bleach
	BC (ES)	Decontamination bleach via ESS
	BC (HS)	Decontamination bleach via BPS
DD x min	30	Processing Time (30 min)
SS *Sample Type*	PC	Positive control coupon/sample
	TC	Test (decontaminated) coupon/sample
	PB	Procedural Blank
	LB	Laboratory Blank
N *Sample Number*	N	Replicate number

2.11 Chemical Analysis

2.11.1 GC/MS Analysis

Extraction samples were analyzed using a Thermo 1310 Gas Chromatograph (Thermo Scientific, Waltham, MA, USA) equipped with a low resolution Thermo ISQ Mass Spectrometer. The GC/MS was operated in alternating selected ion monitoring (SIM) mode and full scan mode. The 173 *m/z* (SIM target ion) was used to quantify the amount of malathion in the extract. Qualifier ions were 125 *m/z*. and 127 *m/z*. The GC was equipped with a 60 meter DB-5 column (0.25 mm x 0.25 µm) (J&W/Agilent, USA). The GC was programmed from the initial temperature of 110 ºC, followed by a ramp at 25 ºC/min to an intermediate one min hold at 215 °C followed by a ramp at 30 °C/min to a final temperature of 300 ºC with a hold of 7.25 min. The carrier gas was helium at a 1.3 mL/min flow rate. The temperature of the injection

port was 150 ºC, and the transfer line temperature was 250 °C. Thermo Xcalibur software was used for data acquisition and processing.

Nine calibration standards were prepared and injected onto the GC/MS system prior to the malathion extract analysis. The composition and concentrations of calibration standards are given in Table 2-6. The initial calibration for the malathion and phenanthrene d-10 as the internal standard (IS) was completed with phenanthrene d-10 at 1000 picograms (pg)/uL and the malathion ranged from 100 pg/uL to 10000 pg/uL. The calibration curve was analyzed with every batch of samples.

Table 2-6. Calibration Solutions (CS1-CS9) Concentrations

	ICAL-1	ICAL-2	ICAL-3	ICAL-4	ICAL-5	ICAL-6	ICAL-7	ICAL-8	ICAL-9
	pg/ µL								
Malathion	100	500	1000	2000	3000	4000	5000	7500	10000
d-10 phenanthrene (IS)	1000	1000	1000	1000	1000	1000	1000	1000	1000

2.11.2 GC/MS Detection Limit

Method detection limit (MDL) values were determined in accordance with SW 846 [2] guidelines for determination of method detection limits. The lowest internal calibration standard (ICAL-1) was injected seven times. The standard deviation from these data was multiplied by the appropriate one-sided 99% t-statistics value [2.998] for seven samples to determine the MDL. The instrumental MDL was calculated to be 2 pg/ µL. For samples where the malathion signal was less than a signal to noise ratio of three, the concentration was reported as ND (non-detect). The signal to noise ratio of the lowest calibration standard was approximately 50. Therefore the precision of the data was better than the signal to noise ratio, and the MDL should therefore be considered to be 6 pg/uL based on the signal to noise ratio of the lowest calibration point.

2.11.3 GC/MS Quantification

Sample concentrations were assessed by using an internal standard (IS) method as outlined in SW 846 [2]. The internal standard used was d-10 phenanthrene.

A response factor (RF) was calculated for each calibration point with the following equations:

$$RF = \frac{A_{cal}}{A_{is}} \times \frac{C_{is}}{C_{cal}} \tag{3}$$

where A_{cal} = Area of malathion calibration point,
C_{cal} = Concentration of the malathion calibration point,
A_{is} = Area of the d-10 phenanthrene internal standard, and
C_{is} = Concentration of the d-10 phenanthrene internal standard.

The calculated response factors from each malathion calibration point were averaged, and this average response factor ($\overline{RF}$) was used to calculate the concentration of the samples.

The sample concentration equation used is:

$$C_x = \frac{A_x \times C_{is}}{A_{is} \times \overline{RF}} \quad (4)$$

where C_x = Concentration of the sample, and
A_x = Area of malathion in the sample.

A malathion-d10 internal standard was used to verify the injection sequence and to ensure that the correct amount was injected, and that no sample degraded in the injection port.

3 Results and Discussion

The results for the decontamination efficacy of bleach as a target decontaminant for the removal of malathion as applied to three building materials (SS, wood, and vinyl) are presented in the following subsections. The results are presented for the decontamination spray methods (ESS and BPS) for all three materials/malathion deposition methods (thin-film application, even-distributed-low droplets, and evenly distributed high concentration droplets).

The decontamination efficacy for each test coupon was determined by measuring the amount of residual chemical on a specific test coupon and comparing the residual chemical to the average values of the positive controls that were subjected to the same malathion deposition method. Positive control values for each material were evaluated to determine the precision with which deposition and analysis of malathion occurred.

3.1 Shakedown of Airbrush Delivery Method

The initial airbrush application was conducted using a solution of 6 mg/mL malathion in ethanol. The recovered malathion amount (by GC/MS analysis) was 5.0 ± 0.8 mg per set of twelve SS coupons (triplicate sets) after an approximately 50 sec spraying time while the theoretical amount was 16.1 ± 1.2 mg per set of twelve SS coupons. Extraction efficiencies were therefore only 24-35%, which are much lower than the anticipated 70-95% range for extraction of malathion from a SS coupon when deposited as a discrete droplet. This apparent lower extraction efficiency may be attributed to either an overspray of the airbrush into the tray resulting in a bias in the spray volume applied per set of coupons or a lower than anticipated extraction efficiency of the extraction method itself. A detailed investigation to identify this lower recovery was not conducted. To compensate for the losses, the malathion concentration was doubled to 12 mg/mL, and the spray time was extended to approximately 70 sec per set of twelve coupons. Although the theoretical amount applied was now approximately 48 mg per twelve coupons, the amounts extracted were in the 8-12 mg range per twelve coupons, similar to the discrete malathion droplet deposition amounts.

3.2 Electrostatic Sprayer Decontamination Results

3.2.1 Thin Film Deposition Method

The results of the airbrush malathion solution deposition technique for the three building materials (SS, wood, and vinyl coupons) are illustrated in Figures 3-1 through 3-3, respectively. As expected, the malathion solution remains on the surface of the SS coupons and to a certain extent on the vinyl coupons but not on the wood coupons due to the absorption capacity of this material. After the 30 or 60 min “weathering” time, the ethanol had evaporated, and the coupons were dry.

A better than 97.5% efficacy was observed following decontamination of all materials with bleach (30 min interaction time) using the ESS (Table 3-1). Decontamination of SS led to malathion non-detects in the test coupon extracts resulting in a better than 99.95% efficacy based on a method detection limit of 6 pg/µL (equivalent to 9 µg in the coupons extract).

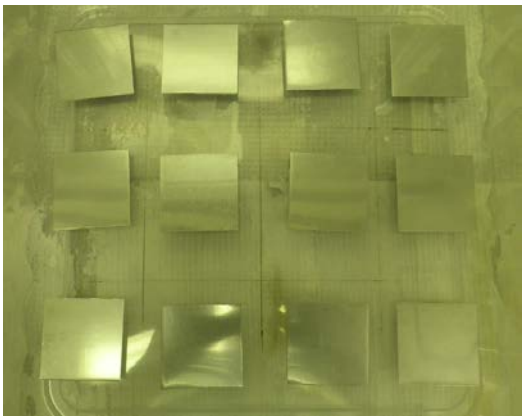

Figure 3-1. Stainless Steel Material Coupons Contaminated using an Airbrush

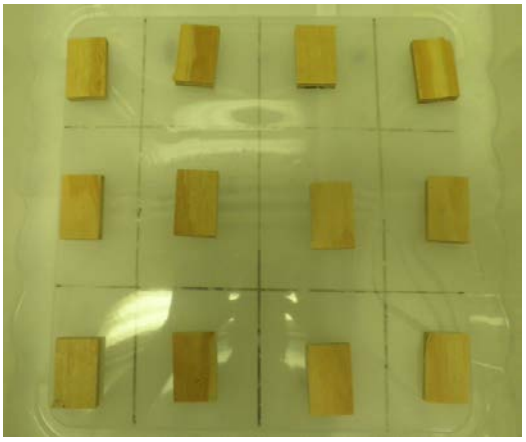

Figure 3-2. Wood Material Coupons Contaminated using an Airbrush

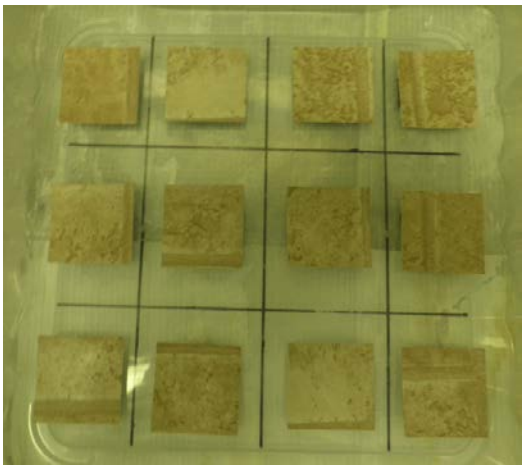

Figure 3-3. Vinyl Material Coupons Contaminated using an Airbrush

Table 3-1. Test Results for Bleach Decontamination using an Electrostatic Sprayer on Material Coupons Contaminated via an Airbrush

Test Results	Amount of Malathion Solution Sprayed		Mass of Malathion Sprayed (mg)	Concentration of Malathion in the Sample Extract (Test/Controls)		Control Sample Malathion Mass Recovery (%)	Decontamination Efficiency (%)	Amount of Bleach Solution Applied (g)
Sample ID	Mass (g)	Volume (mL)		Pre-Decontamination Calculated (pg/mL)	Post-Decontamination Measured (pg/mL)			
Stainless Steel								
67-E3-S-BC(ES)-30-PB-1	--	--	--	--	ND			20.1
67-E3-S-BC(ES)-30-TC-1	2.507	3.178	38.1354	25.4 x10^3	ND		99.95	24.2
67-E3-S-BC(ES)-30-TC-2	3.569	4.523	54.2783	36.2 x10^3	ND		99.95	26.2
67-E3-S-BC(ES)-30-TC-3	3.586	4.545	54.5414	36.4 x10^3	ND		99.95	24.9
67-E3-S-BC(ES)-30-PC-1	3.419	4.334	52.0030	34.7 x10^3	11.9 x10^3	34.3		
67-E3-S-BC(ES)-30-PC-2	3.387	4.292	51.5057	34.3 x10^3	10.6 x10^3	31.0		
67-E3-S-BC(ES)-30-PC-3	3.437	4.357	52.2798	34.9 x10^3	10.8 x10^3	31.0		
67-E3-S-BC(ES)-30-LB-1	--	--	--	--	ND			
Average[1]	*3.32*	*4.20*	*50.46*	*33.6 x10^3*	*11.1 x10^3*	*32.1*	*99.95*	*25.1*
Standard Deviation (SD)[1]	*0.41*	*0.51*	*6.16*	*4.1 x10^3*	*0.7 x10^3*	*1.9*	*0.00*	*1.0*
Wood								
67-E6-W-BC(ES)-30-PB-1	--	--	--		ND			25.0
67-E6-W-BC(ES)-30-TC-1	3.199	4.054	48.6540	32.4 x10^3	251		97.48	21.8
67-E6-W-BC(ES)-30-TC-2	2.736	3.468	41.6137	27.7 x10^3	166		98.34	26.9
67-E6-W-BC(ES)-30-TC-3	3.414	4.326	51.9163	34.6 x10^3	246		97.54	23.2
67-E6-W-BC(ES)-30-PC-1	3.449	4.371	52.4578	35.0 x10^3	10.2 x10^3	29.1		
67-E6-W-BC(ES)-30-PC-2	3.088	3.913	46.9612	31.3 x10^3	10.4 x10^3	33.2		
67-E6-W-BC(ES)-30-PC-3	2.933	3.717	44.6084	29.7 x10^3	9.4 x10^3	31.7		
67-E6-W-BC(ES)-30-LB-1	--	--	--					
Average[1]	*3.14*	*3.98*	*47.70*	*31.8 x10^3*	*10.0 x10^3*	*31.3*	*97.79*	*24.0*
SD[1]	*0.28*	*0.35*	*4.21*	*2.8 x10^3*	*0.5 x10^3*	*2.1*	*0.49*	*2.6*
Vinyl								
67-E9-F-BC(ES)-30-PB-1	--	--	--		ND			22.0
67-E9-F-BC(ES)-30-TC-1	3.330	4.220	50.6449	33.8 x10^3	14		99.81	24.2
67-E9-F-BC(ES)-30-TC-2	2.864	3.630	43.5650	29.0 x10^3	14		99.81	27.1
67-E9-F-BC(ES)-30-TC-3	2.927	3.710	44.5141	29.7 x10^3	2		99.92	25.7
67-E9-F-BC(ES)-30-PC-1	2.841	3.601	43.2061	28.8 x10^3	5.8 x10^3	20.0		
67-E9-F-BC(ES)-30-PC-2	2.868	3.635	43.6228	29.1 x10^3	8.0 x10^3	27.4		
67-E9-F-BC(ES)-30-PC-3	3.238	4.103	49.2395	32.8 x10^3	8.7 x10^3	26.4		
67-E9-F-BC(ES)-30-LB-1	--	--	--					
Average[1]	*3.01*	*3.82*	*45.80*	*30.5 x10^3*	*7.5 x10^3*	*24.6*	*99.85*	*25.7*
SD[1]	*0.21*	*0.27*	*3.27*	*2.2 x10^3*	*1.5 x10^3*	*4.0*	*0.07*	*1.5*

ND: Non-Detect; set to 6 pg/µL (instrument MDL) in calculation of decontamination efficacy.

[1] Average and SD as calculated for shaded values.

3.2.2 Evenly Distributed Low Concentration Droplet Deposition Method

The results of the evenly distributed low concentration malathion solution droplet (nominal 20 µL volume) approach for the three building material (SS, wood, and vinyl) coupons are illustrated in Figures 3-4 through 3-6, respectively. The spiked area with the malathion solution is visibly shown at the center of each coupon of the SS material, but not on the wood and the vinyl coupon materials.

The results for the decontamination efficiency of the evenly distributed low concentration deposition method mirror the results of the decontamination of malathion deposited on the coupons as a thin film. A better than 92.9% efficacy was observed following decontamination of all materials with bleach (30 min interaction time) using the ESS (Table 3-2). Decontamination of SS led to malathion non-detects in the test coupon extracts, resulting in a better than 99.90% efficacy considering a method detection limit of 6 pg/µL.

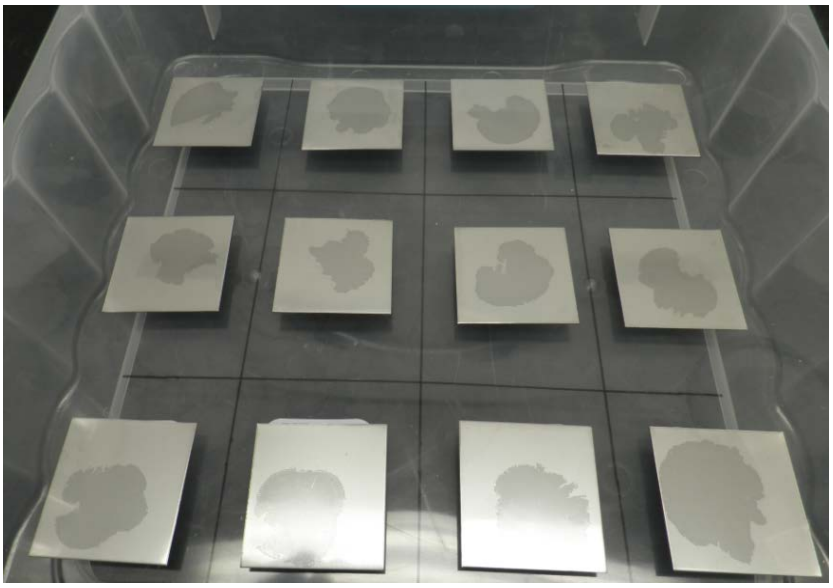

Figure 3-4. Evenly Distributed Low Concentration Droplet Deposition Method on SS Material Coupons

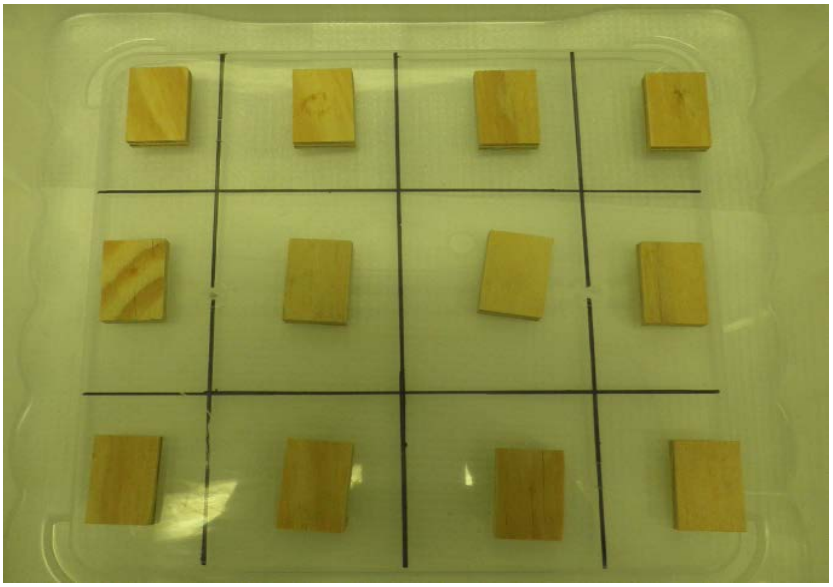

Figure 3-5. Evenly Distributed Low Concentration Droplet Deposition Method on Wood Material Coupons

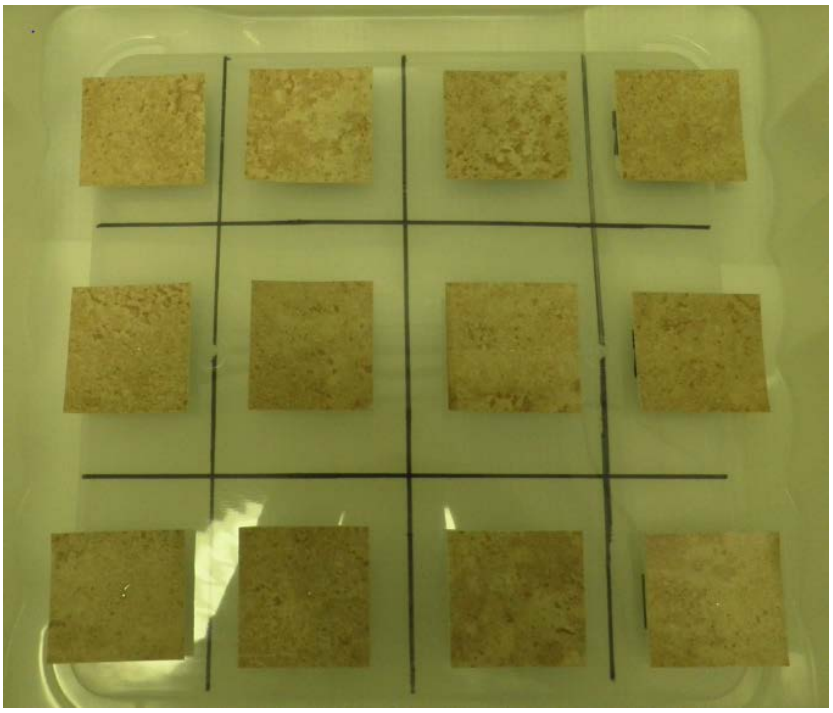

Figure 3-6. Evenly Distributed Low Concentration Droplet Deposition Method on Vinyl Material Coupons

Table 3-2. Test Results for Bleach Decontamination using an Electrostatic Sprayer on Evenly Distributed Low Concentration Droplet Deposition Method on Material Coupons

Stainless Steel							
Test Results	Amount of Solution Spiked (Malathion @ 50 mg/mL)		Concentration of Malathion in the Sample Extract (Test/Controls)		Control Sample Malathion Mass Recovery (%)	Decontamination Efficiency (%)	Amount of Bleach Solution Applied (g)
Sample ID	Volume (mL)	Mass (mg)	Pre-Decontamination Calculated (pg/mL)	Post-Decontamination Measured (pg/mL)			
67-E2-S-BC(ES)-30-PB-1	--	--	--	ND			24.8
67-E2-S-BC(ES)-30-TC-1	240 µL (12 spots x 20 µL)	12	8000	ND		99.90	22.5
67-E2-S-BC(ES)-30-TC-2				ND		99.90	24.3
67-E2-S-BC(ES)-30-TC-3				ND		99.90	20.5
67-E2-S-BC(ES)-30-PC-1				6.4×10^3	80		
67-E2-S-BC(ES)-30-PC-2				6.2×10^3	77		
67-E2-S-BC(ES)-30-PC-3				6.2×10^3	78		
67-E2-S-BC(ES)-30-LB-1	--	--	--	ND			
Average[1]				*6.3×10^3*	*78*	*99.90*	*22.4*
SD[1]				*0.14×10^3*	*2*	*0.00*	*1.9*
Wood							
67-E5-W-BC(ES)-30-PB-1	--	--	--	ND			28.7
67-E5-W-BC(ES)-30-TC-1	240 µL (12 spots x 20 µL)	12	8000	245		93.57	27.5
67-E5-W-BC(ES)-30-TC-2				100		97.37	29.7
67-E5-W-BC(ES)-30-TC-3				151		96.03	25.7
67-E5-W-BC(ES)-30-PC-1				3.3×10^3	41		
67-E5-W-BC(ES)-30-PC-2				4.0×10^3	49		
67-E5-W-BC(ES)-30-PC-3				4.2×10^3	52		
67-E5-W-BC(ES)-30-LB-1	--	--	--	ND			
Average[1]				*3.8×10^3*	*48*	*95.66*	*27.6*
SD[1]				*0.5×10^3*	*6*	*2.00*	*2.0*
Vinyl							
67-E8-F-BC(ES)-30-PB-1	--	--	--	ND			27.2
67-E8-F-BC(ES)-30-TC-1	240 µL (12 spots x 20 µL)	12	8000	98		98.44	25.9
67-E8-F-BC(ES)-30-TC-2				829		86.81	28.6
67-E8-F-BC(ES)-30-TC-3				416		93.39	26.4
67-E8-F-BC(ES)-30-PC-1				6.3×10^3	78		
67-E8-F-BC(ES)-30-PC-2				6.3×10^3	78		
67-E8-F-BC(ES)-30-PC-3				6.4×10^3	79		
67-E8-F-BC(ES)-30-LB-1	--	--	--	ND			
Average[1]				*6.3×10^3*	*79*	*92.88*	*27.0*
SD[1]				*0.05×10^3*	*1*	*5.83*	*1.4*

ND: Non-Detect; set to 6 pg/µL (instrument MDL) in calculation of decontamination efficacy.

[1] Average and SD as calculated for shaded values.

3.2.3 Evenly Distributed High Concentration Droplet Deposition Method

The results of the evenly distributed high concentration malathion solution droplet approach for the three building material (SS, wood, and vinyl) coupons are illustrated in Figures 3-7 through 3-9, respectively. The spiked area with the malathion solution (nominal 2 µL volume) is visibly shown and concentrated at the center of each coupon of the SS material, but not visible on the wood and the vinyl coupon materials.

The results for the decontamination efficiency of bleach using the ESS (Table 3-3) on the wood material with an evenly distributed high concentration deposition is relatively lower (71 ± 4%) than the decontamination efficiencies encountered for wood material coupons contaminated via the thin film deposition method or evenly distributed low concentration deposition methods. Higher decontamination efficacies (better than 94%) for bleach using the ESS were observed for the SS and vinyl materials. Decontamination of the SS led to malathion non-detects in the test coupon extracts, resulting in a better than 99.91% efficacy considering a method detection limit of 6 pg/µL. Further, for the highly concentrated droplet deposition method, corrosion was observed on the SS coupons after the decontamination with bleach as shown in Figure 3-10. This corrosion may be attributed to the formation of various malathion-related degradation acids at a localized high concentration resulting in corrosion of the SS material.

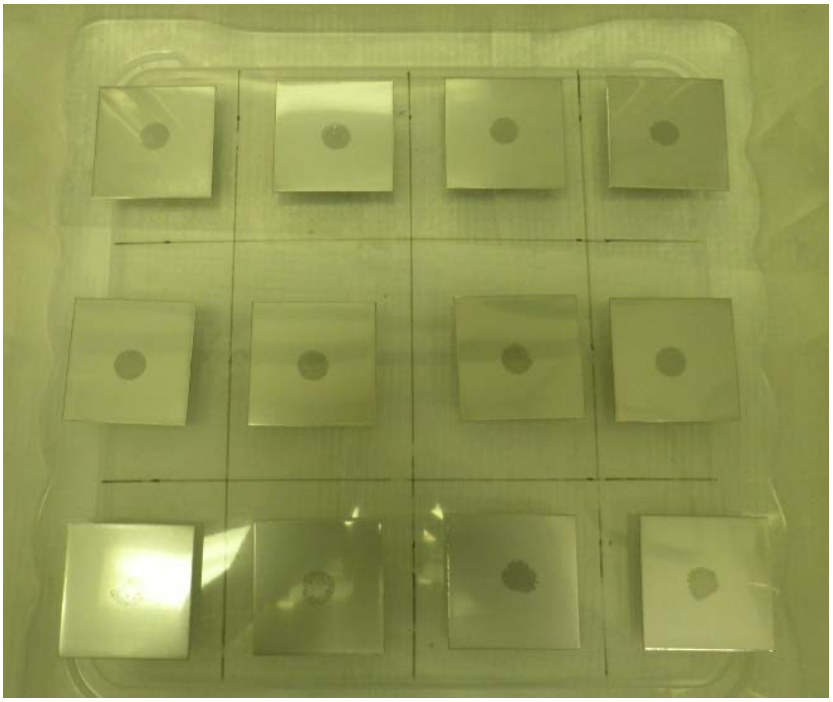

Figure 3-7. Evenly Distributed High Concentration Droplet Deposition Method on SS Material Coupons

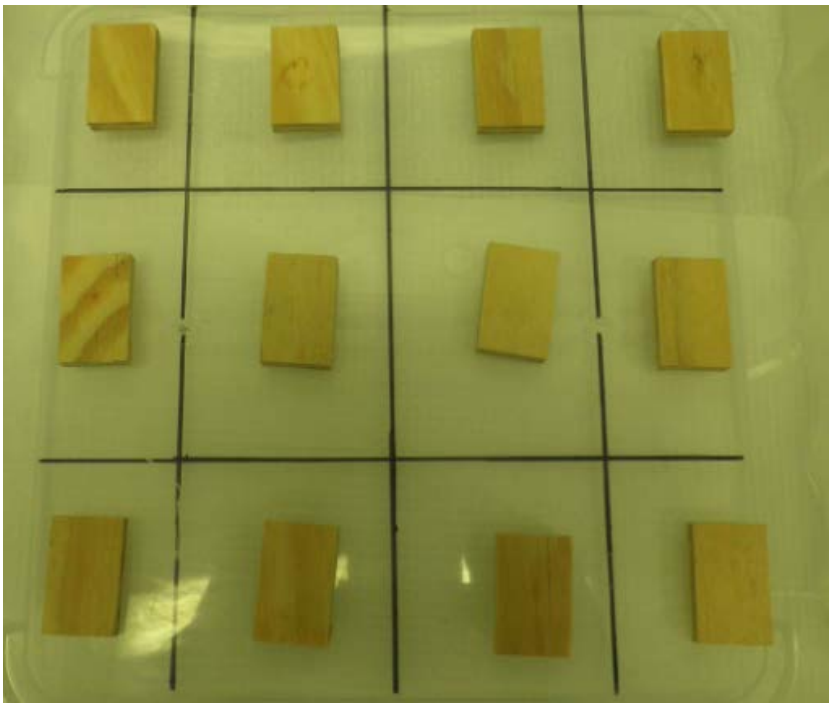

Figure 3-8. Evenly Distributed High Concentration Droplet Deposition Method on Wood Material Coupons

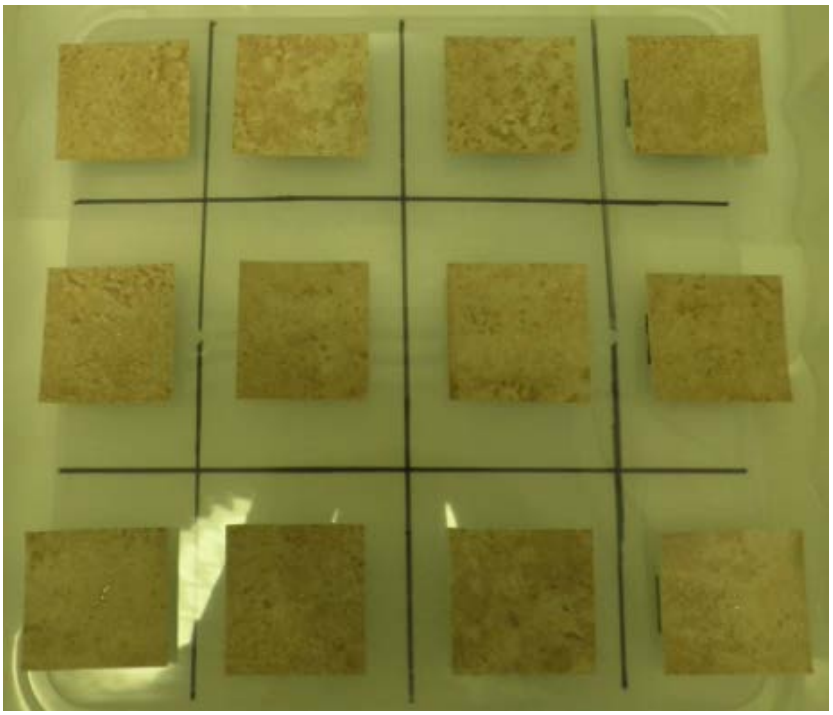

Figure 3-9. Evenly Distributed High Concentration Droplet Deposition Method on Vinyl Material Coupons

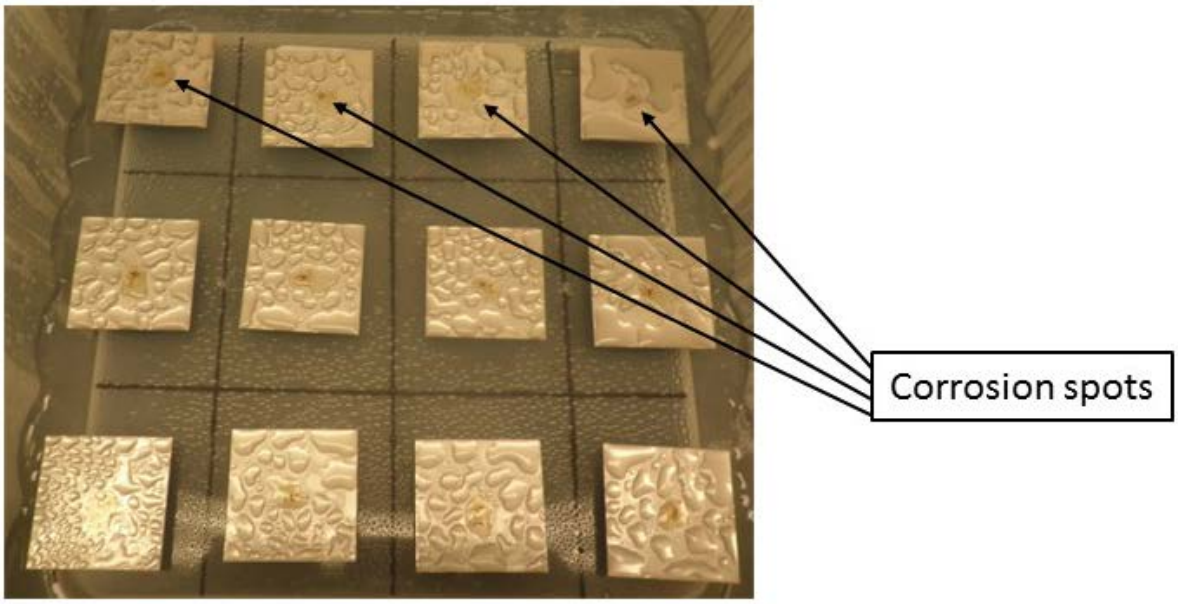

Figure 3-10: Corrosion Spots on SS Material Coupons following Decontamination with Bleach using the Evenly Distributed High Concentration Droplets Deposition Method

Table 3-3. Test Results for Bleach Decontamination using an Electrostatic Sprayer on Evenly Distributed High Concentration Droplet Deposition Method on Material Coupons

Stainless Steel							
Test Results	Amount of Solution Spiked (Malathion @ 500 mg/mL)		Concentration of Malathion in the Sample Extract (Test/Controls)		Control Sample Malathion Mass Recovery (%)	Decontamination Efficiency (%)	Amount of Bleach Solution Applied (g)
Sample ID	Volume (mL)	Mass (mg)	Pre-Decontamination Calculated (pg/mL)	Post-Decontamination Measured (pg/mL)			
67-E1-S-BC(ES)-30-PB-1	--	--	--	ND			22.0
67-E1-S-BC(ES)-30-TC-1	24 µL (12 spots x 2 µL)	12	8000	ND		99.91	21.8
67-E1-S-BC(ES)-30-TC-2				2		99.91	25.6
67-E1-S-BC(ES)-30-TC-3				2		99.91	19.3
67-E1-S-BC(ES)-30-PC-1				6.9×10^3	86		
67-E1-S-BC(ES)-30-PC-2				6.6×10^3	83		
67-E1-S-BC(ES)-30-PC-3				6.8×10^3	85		
67-E1-S-BC(ES)-30-LB-1	--	--	--	ND			
Average[1]				*6.7×10^3*	*84*	*99.91*	*22.2*
SD[1]				*0.1×10^3*	*2*	*0.00*	*3.2*
Wood							
67-E4-W-BC(ES)-30-PB-1	--	--	--	ND			24.4
67-E4-W-BC(ES)-30-TC-1	24 µL (12 spots x 2 µL)	12	8000	1.1×10^3		74.2	26.9
67-E4-W-BC(ES)-30-TC-2				1.4×10^3		67.3	27.9
67-E4-W-BC(ES)-30-TC-3				1.2×10^3		71.7	22.5
67-E4-W-BC(ES)-30-PC-1				4.3×10^3	54		
67-E4-W-BC(ES)-30-PC-2				4.3×10^3	54		
67-E4-W-BC(ES)-30-PC-3				4.1×10^3	51		
67-E4-W-BC(ES)-30-LB-1	--	--	--	ND			
Average[1]				*4.2×10^3*	*53*	*71.1*	*25.8*
SD[1]				*0.1×10^3*	*2*	*3.6*	*2.9*
Vinyl							
Test Results	Amount of Solution Spiked (Malathion @ 500 mg/mL)		Concentration of Malathion in the Sample Extract (Test/Controls)		Control Sample Malathion Mass Recovery (%)	Decontamination Efficiency (%)	Amount of Bleach Solution Applied (g)
Sample ID	Volume (mL)	Mass (mg)	Pre-Decontamination Calculated (pg/mL)	Post-Decontamination Measured (pg/mL)			
67-E7-F-BC(ES)-30-PB-1	--	--	--	ND			21.0
67-E7-F-BC(ES)-30-TC-1	24 µL (12 spots x 2 µL)	12	8000	0.8×10^3		88.0	25.4
67-E7-F-BC(ES)-30-TC-2				0.3×10^3		95.7	27.7
67-E7-F-BC(ES)-30-TC-3				0.1×10^3		98.6	26.6
67-E7-F-BC(ES)-30-PC-1				7.0×10^3	87		
67-E7-F-BC(ES)-30-PC-2				6.4×10^3	80		
67-E7-F-BC(ES)-30-PC-3				6.7×10^3	84		
67-E7-F-BC(ES)-30-LB-1	--	--	--	ND	--		
Average[1]				*6.7×10^3*	*84*	*94.1*	*26.6*
SD[1]				*0.3×10^3*	*4*	*5.5*	*1.2*

ND: Non-Detect; set to 6 pg/µL (instrument MDL) in calculation of decontamination efficacy.

[1] Average and SD as calculated for shaded values.

3.3 Backpack Sprayer Decontamination Results

3.3.1 Thin Film Deposition Method

The decontamination efficacy of the bleach, dispensed via an electrical backpack sprayer, mirrors the results of the ESS for all three building materials (SS, wood, and vinyl) contaminated via the airbrush technique. A greater than 97% efficacy was observed following decontamination of all three materials with bleach (30 min interaction time) using the BPS as shown in Table 3-4. Decontamination of SS led to non-detects for malathion in all test coupon extracts. Considering a method detection limit of 6 pg/µL (equivalent to 9 µg in the coupon extract), the efficacy (for SS) was better than 99.93%.

3.3.2 Evenly Distributed Low Concentration Droplets Deposition Method

The decontamination efficiency of bleach to decontaminate the evenly distributed low concentration malathion deposition for the wood material coupons (88.9 ± 4.7%) is lower than the decontamination efficiency when contamination occurred via the thin film deposition method using the backpack sprayer (98.6 ± 1.0%). For similar material/deposition, the ESS showed relatively higher decontamination efficiency (95.7 ± 2%) than with the BPS. For the vinyl and SS material, the difference in the effects of the decontamination method (BPS versus ESS) is statistically insignificant. Near complete decontamination for vinyl (96.7 ± 2.8%) to full decontamination for SS material (better than 99.90%) was observed for the bleach using the backpack sprayer (Table 3-5).

3.3.3 Evenly Distributed High Concentration Droplet Deposition Method

The results for the decontamination efficiency of the evenly distributed high concentration malathion deposition is relatively lower for the wood material coupons (61.1 ± 7.7%) and for the vinyl coupons (78 ± 11%) than the decontamination efficiencies encountered for the respective materials contaminated via the thin film deposition method using the BPS decontamination approach. For the similar material/deposition, the ESS was shown to be more efficient than the BPS with efficiencies of 71 ± 4% for the wood material and 94 ± 5% for the vinyl material, respectively. For the SS material, the ESS seems to be slightly more effective with full decontamination (non-detects) than the backpack sprayer, but full decontamination was not reached for one of the tests (90.7%). The results for this series of tests are shown in Table 3-6.

Table 3-4. Test Results for Bleach Decontamination using a Backpack Sprayer on Material Coupons Contaminated via an Airbrush

Test Results	Amount of Malathion Solution Sprayed		Mass of Malathion Sprayed (mg)	Concentration of Malathion in the Sample Extract (Test/Controls)		Control Sample Malathion Mass Recovery (%)	Decontamination Efficiency (%)	Amount of Bleach Solution Applied (g)
Sample ID	Mass (g)	Volume (mL)		Pre-Decon Calculated (pg/mL)	Post Decon Measured (pg/mL)			
Stainless Steel								
67-H3-S-BC(HS)-30-PB-1	--	--	--	--	ND			46.7
67-H3-S-BC(HS)-30-TC-1	2.421	3.068	36.817	24. x10^3	ND		99.93	38.4
67-H3-S-BC(HS)-30-TC-2	2.691	3.411	40.934	27.3 x10^3	ND		99.93	46.1
67-H3-S-BC(HS)-30-TC-3	2.506	3.176	38.117	25.4 x10^3	ND		99.93	29.8
67-H3-S-BC(HS)-30-PC-1	2.565	3.251	39.014	26.0 x10^3	7.8 x10^3	34.3		
67-H3-S-BC(HS)-30-PC-2	2.546	3.226	38.715	25.8 x10^3	7.5 x10^3	31.0		
67-H3-S-BC(HS)-30-PC-3	2.634	3.338	40.061	26.7 x10^3	8.8 x10^3	31.0		
67-H3-S-BC(HS)-30-LB-1	--	--	--	--	ND			
Average[1]	*2.56*	*3.25*	*38.94*	*26.0 x10^3*	*8.0 x10^3*	*31*	*99.93*	*38.1*
SD[1]	*0.10*	*0.12*	*1.45*	*1.0 x10^3*	*0.7 x10^3*	*2*	*0.01*	*8.2*
Wood								
67-H6-W-BC(HS)-30-PB-1	--	--	--		ND			36.2
67-H6-W-BC(HS)-30-TC-1	2.508	3.178	38.141	25.4 x10^3	210		97.65	29.0
67-H6-W-BC(HS)-30-TC-2	2.245	2.845	34.143	22.8 x10^3	45		99.50	41.2
67-H6-W-BC(HS)-30-TC-3	2.514	3.186	38.237	25.5 x10^3	118		98.68	27.2
67-H6-W-BC(HS)-30-PC-1	2.904	3.681	44.173	29.4 x10^3	10.8 x10^3	36.8		
67-H6-W-BC(HS)-30-PC-2	3.312	4.198	50.373	33.6 x10^3	6.5 x10^3	19.4		
67-H6-W-BC(HS)-30-PC-3	3.029	3.839	46.070	30.7 x10^3	9.5 x10^3	31.0		
67-H6-W-BC(HS)-30-LB-1	--	--	--	--	ND			
Average[1]	*2.75*	*3.49*	*41.86*	*27.9 x10^3*	*9.0 x10^3*	*29*	*98.6*	*32.5*
SD[1]	*0.40*	*0.50*	*6.03*	*4.0 x10^3*	*2.2 x10^3*	*9*	*1.0*	*7.6*
Vinyl								
67-H9-F-BC(HS)-30-PB-1	--	--	--		ND			32.9
67-H9-F-BC(HS)-30-TC-1	2.196	2.783	33.396	22.2 x10^3	288		95.67	26.7
67-H9-F-BC(HS)-30-TC-2	2.444	3.097	37.170	24.8 x10^3	248		96.26	37.2
67-H9-F-BC(HS)-30-TC-3	2.026	2.568	30.820	20.5 x10^3	35		99.48	23.1
67-H9-F-BC(HS)-30-PC-1	2.188	2.773	33.281	22.2 x10^3	7.5 x10^3	33.7		
67-H9-F-BC(HS)-30-PC-2	2.126	2.694	32.333	21.6 x10^3	4.9 x10^3	22.5		
67-H9-F-BC(HS)-30-PC-3	2.269	2.876	34.517	23.0 x10^3	7.6 x10^3	33.0		
67-H9-F-BC(HS)-30-LB-1	--	--	--					
Average[1]	*2.21*	*2.80*	*33.59*	*22.4 x10^3*	*6.6 x10^3*	*29.7*	*97.1*	*29.0*
SD[1]	*0.14*	*0.18*	*2.15*	*1.4 x10^3*	*1.5 x10^3*	*6.2*	*2.2*	*7.3*

ND: Non-Detect; set to 6 pg/µL (instrument MDL) in calculation of decontamination efficacy.

[1] Average and SD as calculated for shaded values.

Table 3-5. Test Results for Bleach Decontamination using a Backpack Sprayer on Evenly Distributed Low Concentration Droplet Deposition Method on Material Coupons

Stainless Steel							
Test Results	Amount of Solution Spiked (Malathion @ 50 mg/mL)		Concentration of Malathion in the Sample Extract (Test/Controls)		Control Sample Malathion Mass Recovery (%)	Decontamination Efficiency (%)	Amount of Bleach Solution Applied (g)
Sample ID	Volume (mL)	Mass (mg)	Pre-Decontamination Calculated (pg/mL)	Post-Decontamination Measured (pg/mL)			
67-H2-S-BC(ES)-30-PB-1	--	--	--	--			26.2
67-H2-S-BC(ES)-30-TC-1	240 µL (12 spots x 20 µL)	12	8000	1		99.90	27.0
67-H2-S-BC(ES)-30-TC-2				ND		99.90	39.1
67-H2-S-BC(ES)-30-TC-3				ND		99.90	33.5
67-H2-S-BC(ES)-30-PC-1				6.1×10^3	76.3		
67-H2-S-BC(ES)-30-PC-2				6.1×10^3	75.7		
67-H2-S-BC(ES)-30-PC-3				6.1×10^3	75.7		
67-H2-S-BC(ES)-30-LB-1	--	--	--	ND			
Average[1]				*6.1×10^3*	*76*	*99.90*	*33.2*
SD[1]				*0.03×10^3*	*0*	*0.00*	*6.1*
Wood							
67-H5-W-BC(ES)-30-PB-1	--	--	--	ND			46.2
67-H5-W-BC(ES)-30-TC-1	240 µL (12 spots x 20 µL)	12	8000	433		88.2	26.3
67-H5-W-BC(ES)-30-TC-2				230		93.7	38.5
67-H5-W-BC(ES)-30-TC-3				563		85.7	23.7
67-H5-W-BC(ES)-30-PC-1				3.3×10^3	41		
67-H5-W-BC(ES)-30-PC-2				3.8×10^3	48		
67-H5-W-BC(ES)-30-PC-3				3.9×10^3	49		
67-H5-W-BC(ES)-30-LB-1	--	--	--	ND			
Average[1]				*3.7×10^3*	*45.9*	*88.9*	*29.5*
SD[1]				*0.4×10^3*	*4.4*	*4.7*	*7.9*
Vinyl							
67-H8-F-BC(ES)-30-PB-1	--	--	--	ND			27.3
67-H8-F-BC(ES)-30-TC-1	240 µL (12 spots x 20 µL)	12	8000	263		95.7	36.6
67-H8-F-BC(ES)-30-TC-2				13		99.8	55.4
67-H8-F-BC(ES)-30-TC-3				339		94.5	31.0
67-H8-F-BC(ES)-30-PC-1				6.1×10^3	76		
67-H8-F-BC(ES)-30-PC-2				6.1×10^3	77		
67-H8-F-BC(ES)-30-PC-3				6.1×10^3	76		
67-H8-F-BC(ES)-30-LB-1	--	--	--	ND			
Average[1]				*6.1×10^3*	*76.5*	*96.7*	*41.0*
SD[1]				*0.01×10^3*	*0.1*	*2.8*	*12.8*

ND: Non Detect; set to 6 pg/µL (instrument MDL) in calculation of decontamination efficacy.

[1] Average and SD as calculated for shaded values.

Table 3-6. Test Results for Bleach Decontamination using a Backpack Sprayer on Evenly Distributed High Concentration Droplet Deposition Method on Material Coupons

Stainless Steel							
Test Results	Amount of Solution Spiked (Malathion @ 500 mg/mL)		Concentration of Malathion in the Sample Extract (Test/Controls)		Control Sample Malathion Mass Recovery (%)	Decontamination Efficiency (%)	Amount of Bleach Solution Applied (g)
Sample ID	Volume (mL)	Mass (mg)	Pre-Decontamination Calculated (pg/mL)	Post-Decontamination Measured (pg/mL)			
67-H1-S-BC(ES)-30-PB-1	--	--	--	ND			21.8
67-H1-S-BC(ES)-30-TC-1	24 µL (12 spots x 2 µL)	12	8000	658		90.7	23.8
67-H1-S-BC(ES)-30-TC-2				ND		99.9	41.2
67-H1-S-BC(ES)-30-TC-3				ND		99.9	35.1
67-H1-S-BC(ES)-30-PC-1				7.1 x10^3	89		
67-H1-S-BC(ES)-30-PC-2				7.0 x10^3	87		
67-H1-S-BC(ES)-30-PC-3				7.1 x10^3	89		
67-H1-S-BC(ES)-30-LB-1	--	--	--	ND			
Average[1]				*7.1* x10^3	*89*	*96.9*	*33.4*
SD[1]				*0.08* x10^3	*1*	*5.3*	*8.8*
Wood							
67-H4-W-BC(ES)-30-PB-1	--	--	--	ND			41.1
67-H4-W-BC(ES)-30-TC-1	24 µL (12 spots x 2 µL)	12	8000	2.4 x10^3		53.4	28.4
67-H4-W-BC(ES)-30-TC-2				1.9 x10^3		63.2	40.9
67-H4-W-BC(ES)-30-TC-3				1.7 x10^3		66.8	28.8
67-H4-W-BC(ES)-30-PC-1				5.0 x10^3	63		
67-H4-W-BC(ES)-30-PC-2				4.8 x10^3	60		
67-H4-W-BC(ES)-30-PC-3				5.7 x10^3	71		
67-H4-W-BC(ES)-30-LB-1	--	--	--	1.4			
Average[1]				*5.2 x10^3*	*65*	*61.1*	*32.7*
SD[1]				*0.4 x10^3*	*5*	*7.7*	*7.1*
Vinyl							
67-H7-F-BC(ES)-30-PB-1	--	--	--	ND			35.3
67-H7-F-BC(ES)-30-TC-1	24 µL (12 spots x 2 µL)	12	8000	2.3 x10^3		69.0	24.1
67-H7-F-BC(ES)-30-TC-2				0.8 x10^3		89.8	41.7
67-H7-F-BC(ES)-30-TC-3				1.8 x10^3		75.8	35.9
67-H7-F-BC(ES)-30-PC-1				7.7 x10^3	96		
67-H7-F-BC(ES)-30-PC-2				7.8 x10^3	98		
67-H7-F-BC(ES)-30-PC-3				7.1 x10^3	88		
67-H7-F-BC(ES)-30-LB-1	--	--	--	ND			
Average[1]				*7.5 x10^3*	*94*	*78.2*	*33.9*
SD[1]				*0.4 x10^3*	*5*	*10.7*	*9.0*

ND: Non-Detect; set to 6 pg/µL (instrument MDL) in calculation of decontamination efficacy.

[1] Average and SD as calculated for shaded values.

4 Quality Assurance/Quality Control

This project was performed under an approved Category III Quality Assurance Project Plan (QAPP): *Decontamination Solution Methods for Toxic Industrial Chemicals, Part A: Assessment of Decontamination Application and Sampling Method, Task A, approved by the U.S. EPA NHSRC Quality Assurance (QA) Representative (July 2014),* and related amendments.

4.1 Quality Assurance and Quality Control Checks

Uniformity of the test materials was a critical attribute to assuring reliable test results. Samples and test chemicals were maintained to ensure their integrity. Samples were stored away from standards or other samples that could cause cross-contamination.

Supplies and consumables were acquired from reputable sources and were National Institute of Standards and Technology (NIST)-traceable when available. Supplies and consumables were examined for evidence of tampering or damage upon receipt and prior to use, as appropriate. Supplies and consumables showing evidence of tampering or damage were not used. All examinations were documented and supplies were appropriately labeled. Project personnel checked supplies and consumables prior to use to verify that they met specified task quality objectives and did not exceed expiration dates.

4.2 Data Quality Acceptance Criteria Verification

The Data Quality Objectives (DQOs) define the critical measurements needed to address the stated objectives and specify tolerable levels of potential errors associated with simulating the prescribed decontamination environments. The following measurements were deemed to be critical to accomplish part or all of the project objectives:

- ✓ Volume of malathion solution applied to the coupon surface
- ✓ Decontamination exposure time
- ✓ Volume of decontamination solution
- ✓ Volume of extraction solvent
- ✓ Residual chemical quantification of the coupon extractions.

Non-critical measurements were:

- ✓ Temperature measurements of bleach prior to each test.
- ✓ Free Available Chlorine (FAC) and pH of the bleach decontamination solution prior to each test
- ✓ Sonicator temperature.

4.2.1 Control of Monitoring and Measuring Devices

Standard operating procedures for the maintenance and calibration of all laboratory equipment were in place. All equipment (e.g., pipettes, pH meter, microbalances, FAC titrator) and monitoring devices (e.g., thermometer, hygrometer, stopwatches) used at the time of evaluation was verified as being certified

calibrated or having the calibration validated by EPA's on-site (Research Triangle Park, NC) Metrology Laboratory at the time of use. The data quality results for the critical parameters are listed in Table 4-1

Table 4-1. Data Quality Objectives and Results for Test Measurements.

Parameter	Equipment	QC Acceptance Criteria	Results
Extraction Solvent Volume	BRAND® Dispensette ®Organic bottletop dispenser	± 0.5% precision	All these instruments were verified as being certified by the respective manufacturer
Volume of Malathion Applied	Hamilton Microsyringe for Evenly Distributed High Concentration Droplet Deposition Method	± 0.04 µL[1] precision	
	Hamilton Microsyringe for Evenly Distributed Low Concentration Droplet Deposition Method	± 0.20 µL[1] precision	
Mass of Malathion Applied	Airbrush: Balance with daily calibration check using standard weights	Balance precision at least 0.1 x lowest measured value	Balance calibration checks passed Quality Control (QC) requirement
Decontamination Solution Mass	Determined by mass balance with daily calibration check using standard weights	Balance precision at least 0.1 x lowest measured value	Balance calibration checks passed QC requirement
(Decontamination) Time	NIST stopwatch	1 second of NIST time after a 1-min time check	Accuracy of stopwatch clock was acceptable
Residual Malathion Quantification of the Coupon Extractions	GC/MS	Continuing Calibration Verification (CCV) Drift	See Section 4.3
Malathion on Positive Control	Extraction, GC/MS analysis	The mean percent recovery for malathion added to a coupon used to determine recovery must fall within the range of 70%-110% and have a coefficient of variation of <30% between replicates (for same material)	Extraction for Evenly Distributed High and Low Concentration Droplet Deposition distributions passed QC criteria; airbrush/thin film application failed criteria. See Section 4.4.1
Malathion on Laboratory Blank	Extraction, GC/MS analysis	Laboratory blanks should have less than 1% of the amount of malathion compared to that found on positive controls	All laboratory blanks (except for one) were blank (no malathion detected). One exception was within the acceptable range (0.04%)
Malathion on Procedural Blank	Extraction, GC/MS analysis	Procedural blanks should have less than 5% of the amount of malathion compared to that found on positive controls	All were within the acceptable range

[1] Based on trueness and precision Statement of Conformance from the Hamilton Company Website.

4.3 GC/MS Calibrations

Malathion analyses were performed by the EPA Organic Support Laboratory using a GC/MS instrument. The analytical equipment used to determine the amount of malathion on the coupons was calibrated at the time of use and at the frequency specified in Table 4-2.

Table 4-2. Equipment Calibration Schedule

Equipment	Frequency and Approach
GC/MS	At the beginning of each batch of test samples (nine-point calibration curve) and a calibration verification standard (at least every 10 samples and at the end of a batch of samples)

For GC/MS, one (mid-range, ICAL-4) calibration curve standard was analyzed following the calibration curve standards and following at least every ten samples and at the end of each batch of samples. The response to ICAL-4 within 15% of nominal concentration was acceptable. Samples analyzed prior to or following this mid-range calibration standard that were outside of acceptance limits were re-analyzed.

A nine-point calibration was used for each batch of samples for analysis of malathion with a lower level of approximately 100 pg/µL and an upper range of approximately 10000 pg/µL. The GC/MS calibration curves met the following performance requirements:

- R^2 greater than 0.999
- % bias for the lowest standard less than 25%
- % bias for the remaining standards less than 15%.

4.4 Other QC Chemical Analyses

Quality control efforts conducted during decontaminant testing included positive control coupons (contaminated, not decontaminated), procedural blanks (not contaminated, decontaminated), laboratory blanks (not contaminated, not decontaminated), and spike control samples (laboratory quality indicators).

4.4.1 Positive Control Recoveries

Positive control values for each material were evaluated to determine the precision with which deposition and analysis of malathion occurred. The standard deviation of this mean was used in the calculation of error in efficacy values (mean positive control value was used in the calculation of the decontamination efficacy). The recovered amounts of malathion (in triplicate) for each decontamination test are shown in Tables 3-1 through 3-6 and summarized in Table 4-3 for all the malathion deposition methods/types of material tested combinations. The mean recovery of the amount of malathion as applied to a set of twelve coupons using the described extraction procedure and solvents was between 25 and 31% for the airbrush deposition method tests for all the testing materials. This value was well outside the set target recovery of better than 70% (but not more than 110%). The low recovery was attributed to losses during the application procedure and not necessarily due to the analytical extraction approach that was used. The impact of not meeting the extraction criteria is minimal as this low extraction efficiency impacts both the positive control coupons and test coupons equally. Hence, they do not significantly impact the calculated efficacy values.

For the evenly distributed concentration droplet deposition methods, where the application losses are minimal or non-existent, the recoveries for the SS and vinyl sets of coupons were between 76 and 94%, well within the target set recoveries. For the evenly distributed concentration droplet deposition

method/wood combination, the recoveries were between 46 and 65% due to the absorptive nature of the material. The impact of this lower extraction efficiency for wood should be minimal as explained above.

Precision, listed in Table 4-3, was calculated for each data set to describe agreement among the recoveries of the positive controls. Precision is calculated as the percent deviation of the standard deviation from the average mean of the recovery. The results indicate better reproducibility of the data for the evenly distributed concentration droplet deposition methods (0.2 – 12% range) compared to the airbrush deposition method (6 -31% range) due to the variability of the malathion losses when applying the airbrush method to contaminate a set of coupons.

Table 4-3. Quality Assurance Measurements

Deposition Method / Decontamination Approach	Material Type	Extraction Efficiencies Positive Controls (%,± SD)	Precision of Extraction Efficiency Positive Controls (%)	Procedural Blanks	Laboratory Blanks (% of Positive Control Amount)
Airbrush/ESS	Stainless Steel	32 ± 2	6	ND	ND
	Wood	31 ± 2	7	ND	ND
	Vinyl	25 ± 4	16	ND	ND
Airbrush/BPS	Stainless Steel	31 ± 2	7	ND	ND
	Wood	29 ± 9	31	ND	ND
	Vinyl	30 ± 6	21	ND	ND
Evenly Distributed Low Concentration Droplet/ESS	Stainless Steel	78 ± 2	2	ND	ND
	Wood	48 ± 6	12	ND	ND
	Vinyl	79 ± 1	1	ND	ND
Evenly Distributed Low Concentration Droplet//BPS	Stainless Steel	76 ± 0.3	0.4	ND	ND
	Wood	46 ± 4	10	ND	ND
	Vinyl	77 ± 0.1	0.2	ND	ND
Evenly Distributed High Concentration Droplet/ESS	Stainless Steel	84 ± 2	2	ND	ND
	Wood	53 ± 2	3	ND	ND
	Vinyl	84 ± 4	4	ND	ND
Evenly Distributed High Concentration Droplets//BPS	Stainless Steel	89 ± 1	1	ND	ND
	Wood	65 ± 5	8	ND	0.04
	Vinyl	94 ± 5	5	ND	ND

4.4.2 Procedural and Laboratory Blanks

For each chemical-material type, a procedural blank was collected. The results of the procedural blanks are listed in Table 4-3. All the procedural blank results were non-detects (< 6 pg/µL). Recovered chemical amounts from laboratory blanks were all non-detects except for one (1.4 pg/µL or 0.04% of the chemical amount recovered from the positive control for that material).

5 Summary

The objective of this evaluation was to develop/demonstrate two decontamination solution delivery systems and apply them to determine the decontamination efficacies of bleach for decontamination of building materials that were contaminated with malathion. Three different types of malathion distributions on the surfaces were investigated, namely, the presence of a thin uniform film, a localized low concentration droplet; and a localized high concentration distribution.

The decontamination efficacy was evaluated utilizing an electrostatic sprayer and backpack sprayer system. Table 5-1 summarizes the observed decontamination efficacy values.

Table 5-1. Summary of Decontamination Efficacies using ESS and BPS for Three Malathion Deposition Distributions

	ESS					
	Thin Film		Local Low		Local High	
	Mean (%)	SD (%)	Mean (%)	SD (%)	Mean (%)	SD (%)
SS	>99.95	0.00	>99.90	0.00	99.91	0.00
Wood	97.79	0.49	95.66	2.00	71.06	3.61
Vinyl	99.85	0.07	92.88	5.83	94.07	5.49
	BPS					
	Thin Film		Local Low		Local High	
	Mean (%)	SD (%)	Mean (%)	SD (%)	Mean (%)	SD (%)
SS	>99.93	0.01	99.90	0.00	96.85	5.32
Wood	98.61	0.99	88.87	4.70	61.12	7.66
Vinyl	97.14	2.16	96.65	2.79	78.20	10.67

>: Non-detects on all test coupons; calculated efficacy based on instrument MDL of 6 pg/µL.

Very high decontamination efficiency (better than 99.9%) was observed using sprayed bleach on malathion-contaminated SS material. Efficacy was independent of the type of spraying method (ESS or BPS) and independent of the malathion deposition method (thin film, evenly distributed low concentration, or evenly distributed high concentration). With the exception of one SS test coupon (using BPS to decontaminate the localized high concentration malathion), all SS coupon extracts had non-detectable or detectable amounts of malathion below the MDL following the decontamination with bleach (30 min contact time). No difference in performance of the ESS vs. BPS can be derived from the SS efficacy data.

For the wood material, decontamination efficacies were in general significantly lower (Student's t-test p value <0.05) than for the SS material. While decontamination with either spray system resulted in high efficacy (98-99%) for a thin film malathion-contamination distribution, efficacies were lower for the localized low distribution and even lower for the localized high concentration distribution pattern. For each of these distributions, the ESS performed better than the BPS. However, differences were not statistically different (p >0.05).

Efficacy values for the decontamination of vinyl are between those for the SS and wood material. For thin film and localized low concentration distribution, both sprayer systems work equally well with non-statistically significant differences in efficacy. Although the localized high concentration distribution yielded different efficacy values when comparing the ESS against the BPS, the differences were not statistically significant.

A material comparison of efficacy values indicates that the decontamination of wood is in general more difficult than the decontamination of vinyl and SS. Only on two occasions (ESS to decontaminate a thin film or to decontaminate localized high concentration droplet distribution) were these differences statistically significantly different ($p<0.05$).

In general, the ESS is shown to be equally effective as the BPS in decontaminating the three building materials that were tested. The difference in decontamination solution spray pattern on the surface by these two types of sprayers is apparently not a critical aspect in obtaining high efficacy. This result is not too surprising considering that the contact time of 30 min allows for a spreading of decontamination solution on the (horizontal) surfaces. The initial distribution is less relevant as long as the whole surface gets “wetted”.

The thin film, or evenly distributed low concentration droplets of the malathion solution application were found to be more easily decontaminated than the evenly distributed high concentration droplets application for wood and vinyl materials with either decontamination sprayer approach. One explanation for this behavior is that not all applied bleach is in efficient contact with a highly localized contaminant. Therefore, the overall decontamination reaction rate may be limited by the mass transfer rate.

As stated, the difference in decontamination solution spray pattern on the surface by the ESS or BPS does not appear to make a significant difference in these experiments. Further research would be required to determine whether this is true under conditions where, e.g., the amount of decontamination liquid that is applied is (much) lower than what was applied here. This factor would be relevant when spray systems are considered for decontamination of, e.g., personal protective equipment of first responders when there could be a limitation in the amount of decontaminant that is available. A difference in decontamination droplet distribution may also impact the efficacy at shorter contact times between chemical and decontaminant, such as those seen in a personnel decontamination line.

6 References

[1] U.S. Environmental Protection Agency Report, Evaluation of Household or Industrial Cleaning Products for Remediation of Chemical Agents, EPA report 600-R-11-055, 2011.

[2] U.S. EPA Method SW-846. Chapter One. Quality Control. http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/chap1.pdf; last accessed September 14, 2015

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