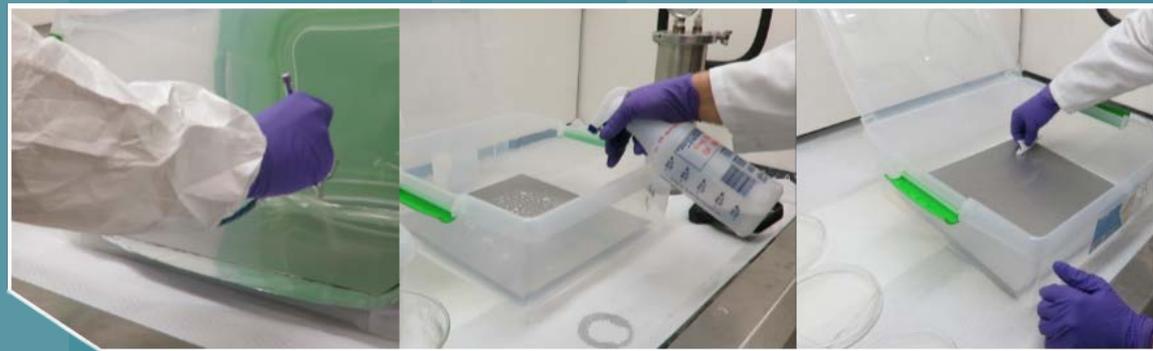


## Assessment of Solution Application Methods for Decontamination of Surfaces Contaminated with Pesticides



**Assessment of Solution Application Methods  
for Decontamination of Surfaces Contaminated  
with Pesticides**

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## Disclaimer

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This research effort is part of the U.S. Environmental Protection Agency's (EPA's) Homeland Security Research Program (HSRP) to evaluate decontamination cleanup procedures in the context of remediation of building material surfaces contaminated with pesticides. Understanding the multifactorial-way interactions between surface, pesticide, and decontaminant is critical for optimization of time- and cost-effective decontamination approaches. The results of this work would inform responders, governments, and health departments in their guidance development for remediation recommendations to the general public.

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## Executive Summary

The U.S. Environmental Protection Agency (EPA) Homeland Security Research Program's (HSRP's) purpose is to protect human health and the environment from adverse impacts of environmental contamination (including terrorist incidents) by investigating the effectiveness and applicability of remediation technologies for environmental response. Within the HSRP, EPA's National Homeland Security Research Center (NHSRC) conducts research needed to identify methods and equipment that can be used for decontamination of building surfaces contaminated with chemicals, including toxic industrial chemicals or materials and chemical warfare agents (CWAs). This study focused on the laboratory-scale evaluation of several decontamination cleanup procedures (DCPs) in the context of a remediation of building surfaces contaminated with pesticides.

Pesticide misuse incidents for controlling bed bugs and other insects in indoor environments continue to occur. These incidents include pesticide products not registered by the U.S. Environmental Protection Agency (EPA) for indoor use or approved pesticide products that are improperly applied and/or applied at concentrations that exceed the approved labeled rates. The bed bug epidemic is expected to result in a growing number of pesticide misuse incidents. State and local agencies and EPA regional offices are often called on to assist local communities in remediating homes and businesses following indoor misapplications where pesticide levels might be unsafe.

Decontamination of surfaces contaminated with chemicals/pesticides is a complex process that involves a combination of chemical and physical interactions between target contaminants, decontaminated materials/surfaces, decontaminant, and, if applicable, also the cleaning media used to deliver the decontaminant [1,2]. Understanding these multifactorial interactions is critical for optimizing time- and cost-effective decontamination approaches. Currently, there are no standard cleaning procedures to reduce pesticide levels in affected structures. Field decontamination and cleaning practices vary widely, and there is no agreement on cleanup and remediation procedures for the wide range of pesticides and surfaces encountered, especially for indoor misuse or overuse situations.

In this study, decontamination testing was performed on a nonporous reference building material (stainless steel coupon, 12 x 12 inch surface area) coated with chemical films of malathion (organophosphorus pesticide) and carbaryl (carbamate pesticide) at a level of hundreds of milligrams per square meter ( $\text{mg}/\text{m}^2$ ). Contaminated surfaces were cleaned using a commercially available solution (EasyDECON® DF200; active ingredient activated hydrogen peroxide,  $\text{H}_2\text{O}_2$ ) or a concentrated germicidal bleach solution. Solutions were applied using various application techniques (spraying, wiping/scrubbing and rolling-on). DCPs were tested in single- and multistep configurations; the single-step method had only one application of contaminant followed by an one-hour dwell time of the decontaminant with the contaminated surface. The multistep method used the same decontaminant applied twice with a cumulative dwell time between decontaminant and contaminated surface of 28 hours (second application four hours after first application). After the desired processing times were reached, test surfaces were rinsed with deionized water and dried overnight. This process is similar to field remediation approaches where residual decontaminant is rinsed off a surface.

Post-decontamination sampling of surfaces was performed using wipes dampened (semi-saturated) with isopropyl alcohol. Following sampling, wipes were then sonicated in hexane at ambient temperature for 15 minutes. Resulting extracts were analyzed by gas chromatography/mass spectrometry (GC/MS). Decontamination efficacy (DE) for each chemical-DCP combination was calculated using the means of

chemical mass recovered from the surfaces of replicate test (decontaminated) coupons and the associated set of positive control (not decontaminated) coupons. No characterization of liquid runoff was performed for spray-on DCPs. Additionally, evaluations of the chemical transfer from the contaminated nonporous material to cleaning media and chemical dissipation from selected surfaces were performed to provide an initial estimate of how mechanical removal and natural indoor environmental attenuation processes only may have contributed to the overall decontamination effectiveness.

The results of the various decontamination processes were variable, depending upon the chemical, decontamination agent, and cleaning technique that was utilized. The decontamination solution with the greatest reduction of pesticide surface concentration was the activated hydrogen peroxide formulation, EasyDECON® DF200. This formulation resulted in nondetectable surface levels of malathion (or  $< 0.54 \text{ mg/m}^2$ ) and a corresponding decontamination efficiency or DE rate of  $> 99.7\%$ ; this DE rate was calculated by comparison with pre-decontamination surface loading of hundreds of  $\text{mg/m}^2$ . EasyDECON® DF200 offered a high decontamination rate for carbaryl as well, with DE ranging from 94.8% to  $> 97.2\%$  (initial surface loading of hundreds of  $\text{mg/m}^2$ ). Only 13% of the test samples treated with various EasyDECON® DF200-based application procedures showed detectable levels ( $> 5.4 \text{ mg/m}^2$ ) of carbaryl with a highest average residual surface concentration of  $11 \text{ mg/m}^2$ . EasyDECON® DF200 did not damage stainless steel and caused no visible material incompatibilities, with post-drying residue easily removable with a final post-decontamination water rinse.

All concentrated germicidal bleach-based DCPs reduced the malathion contamination from hundreds of  $\text{mg/m}^2$  to nondetectable levels ( $< 0.54 \text{ mg/m}^2$ ), with  $> 99.7\%$  DE for malathion. For similar surface concentrations of carbaryl, a maximum average decontamination efficacy of 95% was observed for one multistep decontamination process, namely, the procedure that used a large industrial-grade synthetic cleaning sponge that allowed the highest surface loading of decontaminant among tested procedures. Other bleach-based decontamination procedures had DEs ranging from 63% to 83%, rendering the carbaryl surface level to an average surface concentration of 30 to  $66 \text{ mg/m}^2$ . In addition to a somewhat poorer decontamination performance compared to EasyDECON® DF200, concentrated germicidal bleach caused corrosion and discoloration on the stainless steel surface.

One of the most important considerations when selecting a decontaminant and associated application method is the ability of such DCP to decrease the chemical burden to levels that are considered safe for re-entry without specialized protective equipment, and ultimately for re-occupation of a building. The laboratory determined DEs provide an assessment of the potential of these decontaminants and associated application methods to reduce the chemical loading on the surface under controlled conditions of the test (material, decontaminant, decontamination method, starting surface concentration of the chemical contaminant, etc.). A DE does not provide information whether a safe level of residual chemical is left on the surface. Here, human-health based screening levels were used for the calculation of risk-based cleanup thresholds for malathion and carbaryl, similarly to how these would be established during an actual response. Since there are no EPA regulatory values for surface cleanup goals, risk-based cleanup goals are determined on a site- and situation-specific basis. The method referenced here is based upon the information presented in "World Trade Center Indoor Environment Assessment: Selecting Contaminants of Potential Concern and Setting Health-Based Benchmarks" [3]. It is important to note that these recommendations are not legally binding on any U.S. EPA program and should be interpreted as suggestions that program offices or individual exposure assessors can consider and modify as needed.

For malathion, the estimated child and adult, noncancer, nonporous material surface cleanup thresholds using a conservative target hazard quotient (THQ) [4] of 0.1 are 0.3 and 1.7 mg/m<sup>2</sup>, respectively. These threshold values are lower (for child) and higher (for adult) than the non detectable (< 0.54 mg/m<sup>2</sup>) malathion surface concentrations. Decontamination with bleach or EasyDECON® DF200 using any of the DCPs tested here would have led to acceptable residual pesticide levels for adults. The quantification limit for malathion in this study is too high to determine whether the surface cleanup threshold for a child would have been reached with either decontaminant.

For carbaryl, the noncancer child human health risk-based cleanup threshold for nonporous surfaces and using a conservative THQ=0.1 was estimated to be 1.5 mg/m<sup>2</sup> (8.5 mg/m<sup>2</sup> for a human adult). When using EasyDECON® DF200, one of the thirty test samples across all DCPs resulted in approximately two times higher residual surface concentrations than the derived noncancer child human health risk-based cleanup threshold for nonporous surfaces (11 mg/m<sup>2</sup>). For bleach, only the multistep DCP4 (use of a cleaning sponge on the surface) reduced carbaryl levels to an average level of <10 mg/m<sup>2</sup>, which is still above the human-health (child and adult) risk-based cleanup thresholds for nonporous surfaces. In this laboratory study, the use of germicidal bleach resulted in residual carbaryl surface concentrations that always exceed the estimated health risk-based cleanup threshold values.

The supplemental tests on the natural indoor attenuation of selected pesticides showed slow dissipation (e.g., high persistence) of carbaryl and malathion from nonporous surfaces, with a less than twenty percent reduction of surface chemical loading observed after 46 hours of post-contamination contact time. A more permeable, semi-porous substrate initially evaluated in this study, painted drywall, showed a high permeation-based uptake of malathion and carbaryl, with surface concentration of target pesticides decreased by approximately 90% (as compared to analogous surface-bound levels reported for nonporous materials). Therefore, no further surface decontamination studies were conducted with the drywall coupons due to the lack of effective sampling methods for such semi-porous contaminated materials. Extraction of the pesticide from this material into an organic solvent was considered but not deemed practical considering the size (12 x 12 inches) of the coupons.

The results from the “chemical uptake by cleaning media”-tests suggest that effectiveness of evaluated DCPs should be attributed mostly to chemical reactivity of the decontaminants. However, selected cleaning processes, especially the processes with high liquid decontaminant volume, can remove contamination solely by water via mechanical scrubbing and/or wiping steps. The transfer of contaminant to liquid waste was minimal for DCPs with mechanical removal steps, with a maximum of 10% of the total amount of chemical transferred to runoff.

## **IMPACT OF STUDY**

This study demonstrated that chemical neutralization-based processes that employ a more technologically advanced formula of oxidizers (e.g., EasyDECON® DF200), including addition of activators and/or surfactants, was more advantageous than bleach for surface decontamination of stable and water-insoluble pesticides. In addition, EasyDECON® DF200 was less damaging to sensitive surfaces than bleach and resulted in less post-decontamination waste, thereby offering an economic advantage. The addition of a mechanical cleaning step, especially one that delivers a high surface loading of solution, is desirable for remediation of challenging chemicals, as it showed reduction of contamination even while using non-chemically active cleaning solutions like water. The natural attenuation of high concentration pesticides under ambient indoor environmental conditions does not provide an expedient reduction of chemical films

from nonporous substrates. The low post-contamination surface concentration observed for semi-porous materials after a 30 min contact time was attributed to significant (more than 90%) permeation of pesticides into subsurface layers of the semi-porous test materials, potentially causing reduced susceptibility of absorbed chemicals to non-invasive/non-destructive decontamination technologies.

## **LIMITATIONS OF STUDY**

This study involved the determination of the efficacy of various decontamination applications at a bench scale level and should lead to further research that would address the following elements that were excluded in this effort:

- With the emphasis on the determination of the efficacy towards neutralization of the targeted pesticide, this effort did not address the possible formation of toxic decontamination byproducts, which is important considering the use of malathion as one of the targeted pesticides that may degrade to malaaxon, an oxidation byproduct of equal or higher toxicity than malathion.
- All materials were clean and prepared specifically for this study. Hence, the study did not address the impact of dirt and grime or imperfections on the decontamination efficacy when cleaning these surfaces.
- Pesticides are typically applied using their commercially available technical formulation. The presence of, e.g., water and co-solvents in such formulations may alter the fate and transport of these pesticides, especially into a semi-porous material. However, it is likely that the decontamination efficacy against the neat pesticide applied here as a thin film can be extrapolated to the decontamination efficacy of the same pesticide in a technical solution, especially when such a solution has dried after application of the pesticide product.
- Calculation of the decontamination efficacy assumes equal sampling efficiencies of the chemical/pesticide prior to decontamination (positive control) and post-decontamination (test coupon). A lower sampling efficiency at low surface concentrations may bias the calculated decontamination efficacies high.

# Table of Contents

Disclaimer.....	ii
Acknowledgments .....	iii
Executive Summary .....	iv
Figures .....	x
Tables .....	xii
Acronyms and Abbreviations .....	xiv
1.0. Introduction .....	1
1.1. Project Objectives .....	1
2.0. Experimental Approach.....	3
2.1. Test Facility .....	3
2.2. Experimental Design .....	3
3.0. Materials and Methods.....	4
3.1. Preparation of Test Coupons .....	4
3.2. Target Chemicals .....	4
3.3. Contamination of Coupons .....	5
3.4. Test Setup.....	7
3.5. Method Development Tests .....	8
3.5.1. Sampling and Extraction for Surface Samples .....	8
3.5.2. Optimization of Chemical Delivery to Surface Samples .....	9
3.5.3. Persistence and Uptake of Chemicals by Test Coupon Materials.....	11
3.5.4. Chemical Uptake by Cleaning Media and Transfer to Liquid Effluents .....	12
3.6. Decontamination Tests .....	13
3.6.1. Preparation of Decontamination Solutions .....	13
3.6.2. Decontamination Procedures.....	14
3.6.2.1. Cleaning Media .....	14
3.6.2.2. Test Matrix .....	19
4.0 Sampling and Analysis Methods.....	22
4.1. Sample Process Design for Single- and Multistep Testing.....	22
4.2. Surface Sampling and Extraction Methods.....	26
4.3. Liquid and Solid Waste Sampling and Extraction Procedures .....	28
4.4. Preparation of Samples for Analysis.....	29
4.5. Instrumental Analysis .....	29
4.6. Data Reduction Procedures .....	34
4.6.1. Chemical Concentration in Extract Calculations.....	34
4.6.2. Decontamination Cleanup Efficacy Calculations .....	34
5.0. Results .....	36
5.1. Persistence and Uptake of Chemicals by Nonporous and Semi-Porous Materials.....	36
5.2. Surface Decontamination Efficacy .....	39

5.3. Residual Pesticides and Cleanup Thresholds .....	46
5.4. Transfer of Pesticide to Cleaning Media and Liquid Waste .....	48
6.0. Quality Assurance/Quality Control .....	51
6.1. Test Equipment Calibration.....	51
6.2. Data Quality Results for Critical Measurements .....	51
7.0. Summary.....	53
References.....	55
Appendix A: Supporting Information .....	57
Appendix B: Wipe Sampling Procedure.....	71
Appendix C: Method Development for Liquid Waste Extraction .....	75

## Figures

Figure 2-1. General experimental scheme and timeline. ....	3
Figure 3-1. Master Performance Pro dual-action gravity-feed airbrush. ....	6
Figure 3-2. Contamination spray pattern. ....	6
Figure 3-3. Airbrush application of a malathion solution on stainless steel and chemical film post-application. ....	7
Figure 3-4. Stainless steel and painted drywall coupon assembly readied for testing. ....	7
Figure 3-5. Assembled test setup with nebulization shield placed around test box. ....	9
Figure 3-6. Hand-held pressurized sprayer. ....	14
Figure 3-7. Cleaning-grade hand-held spray bottle. ....	15
Figure 3-8. Absorbent cleaning cloth. ....	15
Figure 3-9. Perforated synthetic wash sponge. ....	16
Figure 3-10. Paint roller cover. ....	16
Figure 3-11. DCP1 through DCP5 application of decontaminant using various cleaning media and resulting appearance of the TC surface. ....	18
Figure 4-1. Day 0 experimental design and sample flow for single- and multistep DCPs: contamination of coupons and surface sampling of PCs. ....	22
Figure 4-2. Day 1 experimental design and sample flow for single-step DCPs: application of decontaminant and water rinse. ....	23
Figure 4-3. Day 1 experimental design and sample flow for multistep DCPs: application and re-application of decontaminant. ....	24
Figure 4-4. Day 2 experimental design and sample flow for single-step DCPs: surface sampling of TCs. ....	25
Figure 4-5. Day 2 experimental design and sample flow for multistep DCPs: application of water rinse. ....	26
Figure 4-6. Day 3 experimental design and sample flow for multistep DCPs: surface sampling and extractions of TCs and PB. ....	26
Figure 4-7. Example of surface wipe sampling of stainless steel in horizontal orientation. ....	27
Figure 5-1. Malathion surface concentration over time on nonporous material, stainless steel. ....	36
Figure 5-2. Carbaryl surface concentration over time on nonporous material, stainless steel. ....	37
Figure 5-3. Malathion surface concentration over time on semi-porous material, painted drywall. ....	38
Figure 5-4. Carbaryl surface concentration over time on semi-porous material, painted drywall. ....	38
Figure 5-5. Malathion decontamination efficacy (%±SD) for single- vs multistep DCPs. ....	44
Figure 5-6. Carbaryl decontamination efficacy (%±SD) for single- vs multistep DCPs. ....	44
Figure 5-7. Appearance of the material surface after multistep treatment with bleach (A) and EasyDecon® DF200 (B). ....	45
Figure 5-8. Post cleanup surface concentrations of malathion versus calculated human health risk-based cleanup thresholds – <i>THQ: target hazard quotient</i> . ....	47
Figure 5-9. Post cleanup surface concentrations of carbaryl versus calculated human health risk-based cleanup thresholds – <i>THI: target hazard index</i> . ....	48

Figure 5-10. Expended cleaning materials generated during decontamination of an approximate area of 12 ft<sup>2</sup> using three types of cleaning media ..... 49

Figure 5-11. Malathion post-cleanup surface, liquid and solid waste amounts for all mechanical cleaning media; % contribution calculated based on ½ LOQ..... 50

Figure 5-12. Carbaryl post-cleanup surface, liquid and solid waste amounts for all mechanical cleaning media; dashed bars – no quantification, % contribution calculated based on ½ LOQ ..... 50

Figure B-1. Folding wipe for sampling the first wiping pathway (horizontal)..... 71

Figure B-2. Horizontal wiping pathway. .... 72

Figure B-3. Folding wipe for sampling the second wiping pathway (vertical). .... 72

Figure B-4. Vertical wiping pathway..... 72

Figure B-5. Folding wipe for sampling the third wiping pathway (diagonal). .... 73

Figure B-6. Diagonal wiping pathway. .... 73

Figure B-7. Folding wipe for sampling the fourth pathway (perimeter)..... 74

Figure B-8. Perimeter wiping pathway. .... 74

## Tables

Table 3-1. Specifications of building materials.....	4
Table 3-2. Physical and chemical properties of malathion and carbaryl .....	5
Table 3-3. Experimental parameters for surface contamination, wipe sampling and extraction optimization tests.....	8
Table 3-4. Chemical surface loading of PCs (decontamination tests).....	10
Table 3-5. Samples for persistence and uptake of chemical by test material .....	12
Table 3-6. Uptake of chemical by cleaning media and transfer to liquid effluent test .....	13
Table 3-7. Decontamination solutions.....	13
Table 3-8. Average surface loadings of decontaminant solutions and water rinses for different cleaning media.....	17
Table 3-9. Test Matrix for Malathion Decontamination Testing .....	20
Table 3-10. Test Matrix for Carbaryl Decontamination Testing .....	21
Table 4-1. Wiping media, wetting solvent, and wetting solvent volume for surface sampling.....	27
Table 4-2. Instrumental parameters and conditions for GC/MS analyses of malathion (EMSL Analytical, Inc.) .....	30
Table 4-3. Instrumental parameters and conditions for GC/MS analyses of carbaryl (EMSL Analytical, Inc.) .....	31
Table 4-4. Instrumental parameters and conditions for GC/MS analyses of carbaryl (EPA OSL) .....	31
Table 4-5. QC checks for instrumental analyses performed by subcontracting laboratory. ....	32
Table 4-6. The initial and continuing laboratory proficiency results .....	33
Table 5-1. Malathion and carbaryl surface concentrations on stainless steel and painted drywall over three contact times tested .....	36
Table 5-2. Single- and -multistep bleach decontamination test results for malathion on stainless steel....	40
Table 5-3. Single- and -multistep EasyDECON® DF200 decontamination test results for malathion on stainless steel.....	41
Table 5-4. Single- and -multistep bleach decontamination test results for carbaryl on stainless steel .....	42
Table 5-5. Single- and -multistep EasyDECON® DF200 decontamination test results for carbaryl on stainless steel.....	43
Table 6-1. Instrument Calibration Frequency.....	51
Table 6-2. Acceptance Criteria for Critical Measurements and Corresponding Test Results .....	52
Table A-1. Experimental parameters for malathion decontamination with bleach (single-step procedure ....	58
Table A-2. Experimental parameters for malathion decontamination with bleach (multistep procedure) ..	59
Table A-3. Experimental parameters for carbaryl decontamination with bleach (single-step procedure .....	60
Table A-4. Experimental parameters for carbaryl decontamination with bleach (multistep procedure) .....	61
Table A-5. Experimental parameters for malathion decontamination with EasyDECON® DF200 (single-step procedure .....	62
Table A-6. Experimental parameters for malathion decontamination with EasyDECON® DF200 (multistep procedure).....	63

Table A-7. Experimental parameters for carbaryl decontamination with EasyDECON® DF200 (single-step procedure) ..... 64

Table A-8. Experimental parameters for carbaryl decontamination with EasyDECON® DF200 (multistep procedure) ..... 65

Table A-9. Calculated risk-based surface cleanup thresholds for malathion and carbaryl..... 66

Table A-10. Concentration of malathion in cleaning media (solid waste), liquid waste, and post- cleanup surface concentration - all mechanical cleaning media (DCP deployed using water only) ..... 69

Table A-11. Concentration of carbaryl in cleaning media (solid waste), liquid waste, and post-cleanup surface concentration - all mechanical cleaning media (DCP deployed using water-only) ..... 70

## Acronyms and Abbreviations

AChE	acetylcholinesterase
BL	bleach, blank
°C	degrees centigrade
CC	continuous calibration
cm	centimeter(s)
CMAD	Consequence Management and Advisory Division
COC	chain of custody
COTS	commercial-off-the-shelf
CS	control spike
CT	contact time
DCP	decontamination cleanup procedure
DE	decontamination efficacy
df	film thickness
DI	deionized
DQI	data quality indicator
DT	dwel time
DUP	duplicate injection
EC	end check
EPA	U.S. Environmental Protection Agency
ft	foot / feet
GC	gas chromatography or gas chromatographic
GC/MS	gas chromatography/mass spectrometry
h	hour(s)
HPLC	high performance liquid chromatography
HSRP	Homeland Security Research Program (EPA)
ICAL	initial calibration
ICV	Initial Calibration Verification
in	inch
IPA	isopropyl alcohol
IS	internal standard
ISO	International Organization for Standardization
LCS	laboratory control spike
LCSD	laboratory control spike duplicate
LOQ	limit of quantitation
m	meter(s)
m <sup>2</sup>	square meter(s)
mg	milligram(s)
min	minute(s)
mL	milliliter(s)
mm	millimeter(s)
NCP	National Contingency Plan
NHSRC	National Homeland Security Research Center
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
OLEM	Office of Land and Emergency Management

OP	organophosphorus/organophosphate
ORD	Office of Research and Development (EPA)
OSL	Organic Support Laboratory (EPA)
oz	ounce(s)
Pa	Pascal
PB	procedural blank (coupon)
PC	positive control (coupon)
psi	pounds per square inch (pressure)
PTFE	polytetrafluoroethylene
QAPP	quality assurance project plan
QC	quality control
RH	relative humidity
RLV	reporting limit verification
RPD	relative percent difference
RSD	relative standard deviation
RTP	Research Triangle Park
SB	solvent blank
SD	standard deviation
TAT	turnaround time
TC	test coupon (contaminated and decontaminated)
THI	target hazard index
µg	microgram(s)
WA	work assignment

## 1.0. Introduction

Pesticide misuse incidents for controlling bed bugs and other insects in indoor environments have increased. These incidents include pesticide products not registered by the U.S. Environmental Protection Agency (EPA) for indoor use or approved pesticide products that are improperly applied and/or applied at concentrations that exceed the labeled rates. The bed bug epidemic is expected to result in a growing number of pesticide misuse incidents. State and local agencies and EPA regional offices are often called on to assist local communities in remediating homes and businesses following indoor misapplications where pesticide levels might be unsafe. Currently, there are no standard cleaning procedures to reduce pesticide levels in affected structures. Field decontamination and cleaning practices vary widely, and there is no agreement on cleanup and remediation procedures for the wide range of pesticides and surfaces encountered, especially for indoor misuse or overuse situations.

This report discusses the deployment of one-step and multistep cleaning procedures for decontamination of a standard reference material (stainless steel) contaminated with two common pesticides (malathion and carbaryl). Decontamination Cleanup Procedures (DCPs) tested used low-tech or specialized oxidizing decontaminants (concentrated household germicidal bleach or hydrogen peroxide-based EasyDECON® DF200), applied in various ways. DSPs ranged from spray-on application of the decontaminant to application in combination with mechanical/physical removal (scrubbing). The goal of this project is to provide field remediation specialists with more information on the effectiveness of various decontamination solutions and related application methods for cleaning indoor surfaces contaminated with pesticides.

### 1.1. Project Objectives

The purpose of this project was to provide responding agencies with information on the effectiveness of several decontamination and cleanup methods for high levels of pesticides on several building materials. This research built on previous efforts under U.S. Environmental Protection Agency's (EPA's) Homeland Security Research Program (HSRP) that determined conditions that led to effective cleanup of building materials using specialized and commercial-off-the-shelf (COTS) cleaning products. This work evaluated selected decontamination solution application methods that are being used in the field in response to the misuse or overuse of pesticides.

This work had three primary objectives:

1. Develop DCPs that are easily deployable in the field and can be used for remediation of indoor building material surfaces contaminated with pesticides.
2. Determine the feasibility and effectiveness of proposed approaches by measuring post-decontamination surface pesticide residue concentrations in comparison to the mass of pesticide on the positive control (non-decontaminated) coupons. The post-decontamination surface pesticide concentrations would then be compared to derived health-based threshold values.
3. Compare remedial effectiveness of single step vs multistep protocols using specialized and COTS cleaning media (e.g., wipes, sponges, cloths).

The secondary objectives were as follows:

1. Provide information on uptake of chemicals by various sampling media used for cleaning as well as on the transfer of residual chemical to liquid effluents (decontamination solutions or water rinses).
2. Provide initial information on the amount of solid and liquid secondary waste generated from various cleaning protocols.
3. Assess (qualitatively) the impact of the decontaminant and application method on the material
4. Compare residual pesticide levels post-decontamination against estimated cleanup threshold levels

Results from this study characterize the performance versus practicality of different cleanup techniques and remediation methods. This information will assist in creating guidelines for selection of the best standardized approaches for remediation of pesticides in indoor environments.

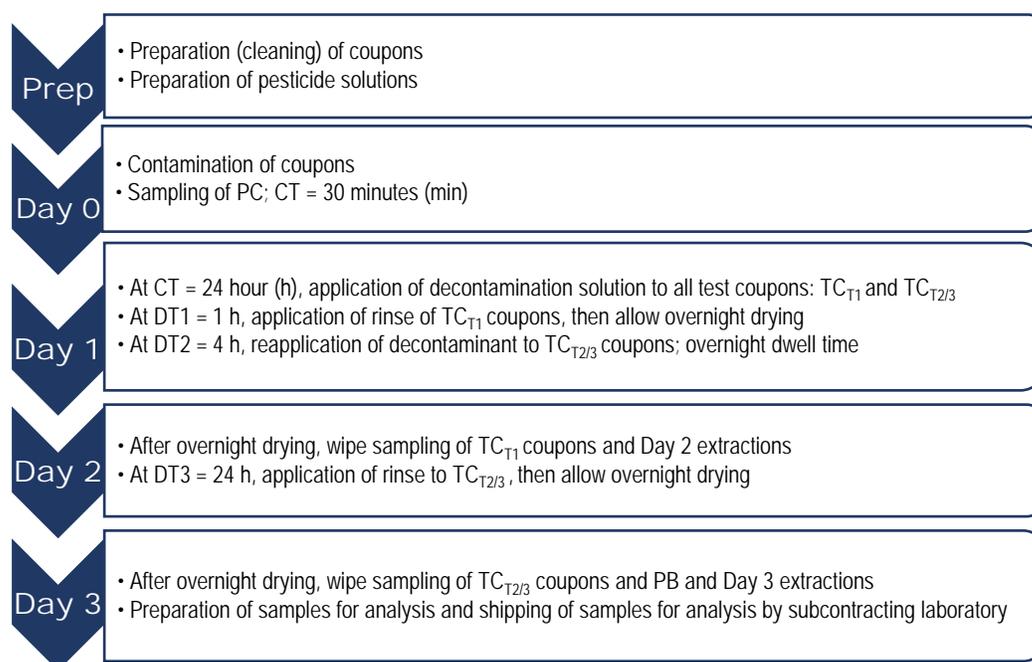
## 2.0. Experimental Approach

### 2.1. Test Facility

The experimental work was performed at the EPA's facilities in Research Triangle Park (RTP), NC. Instrumental analyses of target chemicals in extracts and control samples were performed by an external accredited chemical analysis laboratory (EMSL Analytical, Inc., Cinnaminson, NJ, USA) and by the in-house EPA Organic Support Laboratory (OSL), located in RTP, NC.

### 2.2. Experimental Design

This study evaluated five different approaches to deliver two types of decontaminants (concentrated germicidal bleach and EasyDECON® DF200) to indoor nonporous and semi-porous surfaces (stainless steel and painted drywall) that were contaminated with malathion or carbaryl. The decontaminant delivery approaches consisted of spraying, scrubbing, rolling on, or wiping of the decontamination product, and combinations thereof. For each procedure, a rinse step was incorporated (at various dwell times) after the decontamination was completed as to remove residual decontaminant from a surface and to define the decontamination time for the surface. A reapplication of decontaminant was tested as well for multiday DCPs. The surface sampling of post-decontamination residual pesticides was performed after completion of an overnight (24 h) drying step for each procedure. The schemes and general timelines for the single and multistep test approaches are shown in Figure 2-1. Details of each cleanup procedure are described in Sections 3.6.2 and 4.1. The sampling of a single set of positive controls (PCs) at a 30 min contact time (CT) was based on the minimal dissipation of target chemicals observed during method development for CTs up to 46 h (see Section 5.1)



PC -- Positive control; CT - Contact time (time the chemical is in contact with a material surface); DT - Dwell time (time the decontaminant is in contact with the material surface contaminated with pesticide; TC<sub>T1</sub> - material test (decontaminated) coupon (TC) for one application of decontaminant at DT1 = 1 h; TC<sub>T2/3</sub> - material test coupon for two applications of decontaminant at dwell times DT2 = 4 h plus DT3 = 24 h (equals 28 h); PB - Procedural blank

**Figure 2-1. General experimental scheme and timeline.**

## 3.0. Materials and Methods

### 3.1. Preparation of Test Coupons

Two building materials (stainless steel and painted drywall) were selected for evaluation of cleanup procedures (Table 3-1). Stainless steel was a reference material, selected for inertness and minimal porosity and considered a good model for optimization of sampling approaches. Stainless steel was also considered a surrogate for nonporous building surfaces. Painted drywall was selected as representative of a semi-porous, permeable (painted/sealed) building material. The building material specifications are given in Table 3-1. Due to substantial difficulties with recovery of target chemicals from the painted drywall surface (results are described in Section 3.5.2.), this material was not subsequently used in decontamination tests. Research that addresses phenomena specific to the (significant) chemical transfer into porous/semi-porous materials and the efficiency of various decontamination methods for neutralization of chemicals that are partially absorbed into a semi-porous building material (surrogate) is in progress.

**Table 3-1. Specifications of building materials**

Material	Description	Manufacturer/ Supplier Name/Location	Coupon Size, L x W (in)	Material Preparation
Stainless steel	Multipurpose stainless steel (48 x 48 in), type 304, #2B mill (unpolished), 0.036 in thick	McMaster-Carr, Douglasville, GA, USA	14 x 14*	Cut into coupons and remove any lubricant/grease from shearing with acetone. Wipe dry. Immediately before use, remove particles and dust by wiping clean with acetone and then water. Wipe dry.
Painted drywall	½ in x 4 foot (ft) x 8 ft drywall panel primed with KILZ® latex primer and painted with premium 100% acrylic latex interior flat paint in white	National Gypsum Company, Charlotte, NC, USA/ Lowe's, Mooresville, NC, USA; KILZ® Latex Primer, Masterchem Industries, Santa Ana, CA, USA; Behr, Santa Ana, CA, USA /Home Depot, Atlanta, GA, USA	14 x 14*	Cut into coupons. Remove particles by wiping clean with water and wipe dry.

\* Actual test area was center 12 x 12 inches (in) or 929 cm<sup>2</sup>

Stainless steel coupons were cut to the correct length and width from larger sheets using heavy-duty power hydraulic shears. Painted drywall panels were pre-cut to desired dimensions using a table saw. The edges were finished using two-inch joint tape and joint compound applied over the tape using a putty knife. After the joint compound cured, any rough spots were removed using a sanding block. Coupons were then primed, sanded and painted using latex-based primer and 100% acrylic latex interior flat paint (Table 3-1). All coupons were cleaned prior to testing using procedures described in Table 3-1.

### 3.2. Target Chemicals

The pesticides used to evaluate DCPs were malathion and carbaryl. Malathion is an organophosphorus (OP) insecticide widely used in agriculture, outdoor pest control, and residential landscaping. Carbaryl is a carbamate insecticide that is commonly used in gardens, commercial agriculture, and forestry. Both insecticides have been misused indoors in improper responses related to the current bed bug epidemic.

A malathion analytical standard was purchased from Chem Service (Chem Service, Inc., West Chester, PA, USA; product # N-12346-500MG; purity: 99.5%). A carbaryl analytical standard was purchased from Sigma-Aldrich (Sigma-Aldrich Co. LLC, St. Louis, MO, USA; product # 32055-250MG;

purity 99.9%). The relevant physical and chemical properties of malathion and carbaryl are summarized in Table 3-2.

**Table 3-2. Physical and chemical properties of malathion and carbaryl**

Property*	Malathion	Carbaryl
CAS Registry Number	121-75-5	63-25-2
Molecular weight	330.4	201.2
Formula	C <sub>10</sub> H <sub>19</sub> O <sub>6</sub> PS <sub>2</sub>	C <sub>12</sub> H <sub>11</sub> NO <sub>2</sub>
Density (g/cm <sup>3</sup> ) at 20 °C	1.23	1.20
Physical form at 20 °C	Liquid	Solid
Vapor pressure	4.0 x 10 <sup>-5</sup> mm Hg at 25 °C	1.5 x 10 <sup>-6</sup> mm Hg at 25 °C
Solubility in water	0.143 g/L	0.04 g/L
Log K <sub>ow</sub>	2.36-2.89	2.36

\*Data from <https://pubchem.ncbi.nlm.nih.gov> (last accessed September 2017)

The target surface chemical concentrations in this study were based on field studies on wipe sampling of indoor household surfaces after the misuse of malathion and carbaryl in residences, which have shown a maximum concentration of 4.46 and 24.2 micrograms per square centimeter (µg/cm<sup>2</sup>) for malathion and carbaryl, respectively; the concentration targets per 12 in x 12 in test area on each coupon were then approximately 4 and 24 milligrams (mg) per surface area of 929 cm<sup>2</sup>, yielding theoretical contamination levels in tens to hundreds of milligrams per square meter (mg/m<sup>2</sup>) range. Chemicals were applied onto the test coupon (TC) as a chemical film using an airbrush-based application method described in Section 3-3.

### 3.3. Contamination of Coupons

Pesticide solutions were applied to coupon materials using an airbrush tool to form a thin film of chemical on the coupon surface. Solutions were prepared using procedures developed in previous research efforts [5] by dissolution of neat chemicals in organic solvents. Briefly, neat chemicals were dissolved in high performance liquid chromatography (HPLC)-grade solvent to produce a mg/mL concentration nebulization solution, then mixed using a vortex mixer and finally mixed via sonication for approximately 30 seconds. Malathion was dissolved in ethanol at 6 mg/mL, and carbaryl was dissolved in acetone at 8 mg/mL. The concentrations and volumes of nebulization solutions were delivered experimentally to allow deposition of the chemical amount at the target surface concentration levels (Section 3.5.2). The optimized nebulization methods accounted for losses related to overspray and settling of the chemical cloud on the walls of the spraying shield, as well as losses of airborne chemical solutions due to high air flow in the chemical hood; the latter effect was mitigated but not completely removed by the use of a nebulization (or spraying) shield (Figure 3-5 in Section 3.5.2).

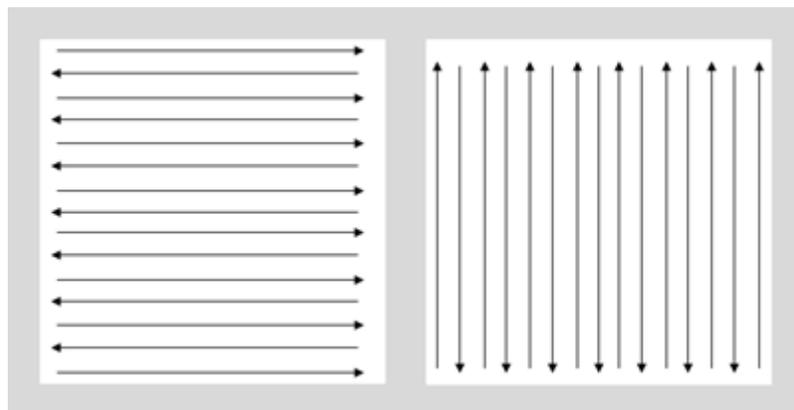
The accuracy and precision of preparation of spiking solutions was assessed for each experimental batch by analysis of control spike (CS) samples (see Section 4.5 for results of analyses of control spikes). TCs were cleaned using acetone and water and wiped dry, then contaminated with target chemicals using the procedure described below.



**Figure 3-1. Master Performance Pro dual-action gravity-feed airbrush.**

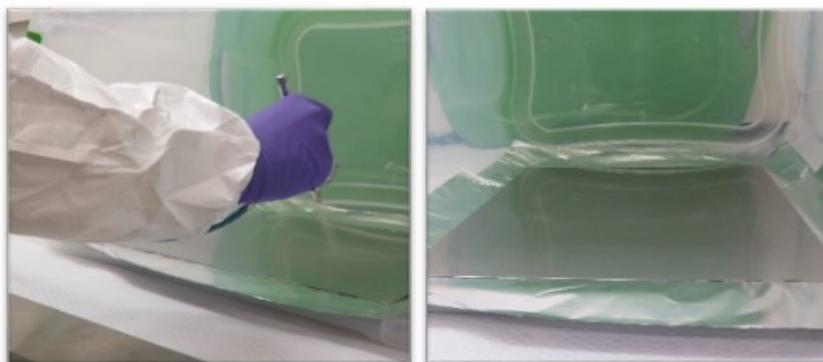
A Master Performance Pro dual-action gravity-feed airbrush with a 0.2-millimeter (mm) nozzle (Item No. MAS G-233-SET, TCP Global, San Diego, CA), shown in Figure 3-1, was used for the thin-film application of malathion and carbaryl solutions onto the surface of the coupons. This airbrush tool is equipped with solvent-resistant needle packing and can be used with organic solvents.

The air supply was regulated using the airbrush compressor air pressure regulator gauge equipped with a water trap moisture filter, with the pressure regulator set to 25 pounds per square inch (psi). Prior to each use, the airbrush tool was purged with 5 milliliters (mL) of the target solvent (ethanol for malathion and acetone for carbaryl). Then, the airbrush barrel was filled with 5 mL of pesticide solution (malathion in ethanol at 6 mg/mL or carbaryl in acetone at 8 mg/mL). A clean coupon was placed inside a plastic spraying shield, and pesticide solution was sprayed onto the coupon surface using slow sweeping motions; spraying started at the top left corner of the coupon and continued from left to right/top to bottom in a swiping motion, as shown in Figure 3-2.



**Figure 3-2. Contamination spray pattern.**

The spraying pattern was replicated until the reservoir was empty. Start and stop time of spraying was recorded. Figure 3-3 shows the application of the chemical solution onto the stainless steel surface (left) and the chemical film visible on the coupon surface post-application (right - the example shown is the malathion chemical film).

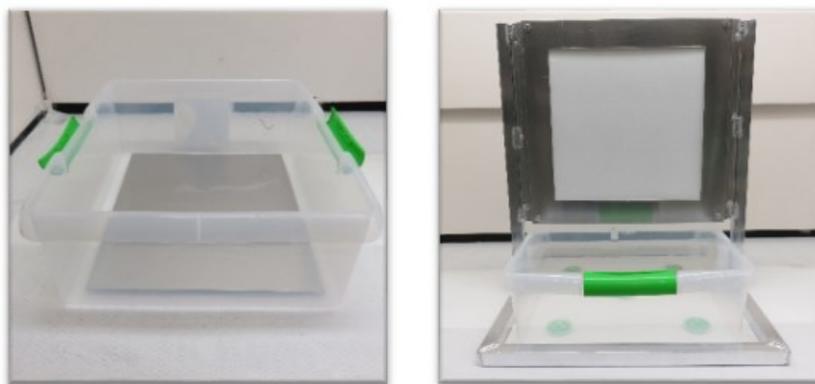


**Figure 3-3. Airbrush application of a malathion solution on stainless steel and chemical film post-application.**

After nebulization of the chemical was completed, each contaminated coupon was gently transferred horizontally by holding onto the edges into an individual pre-cleaned test box (dimensions: 18 in × 18.75 in × 6.25 in; Stor-N-Slide square box with lid, product # 491530; IRIS USA, Inc., Pleasant Prairie, WI, USA) to allow for a simulated (indoor) weathering period/contact time; weathering was performed under normal ambient laboratory conditions of approximately 22 °C and 25% relative humidity (RH) (averages typical for early winter/spring months when testing was performed). Test boxes were cleaned using laboratory-grade detergent solution, then wiped with acetone and water and wiped dry.

### 3.4. Test Setup

Method development (Section 3.5) and decontamination testing (Section 3.6) were performed in a chemical safety hood. Each contaminated coupon was placed in an individual pre-cleaned test box (specifications and cleaning procedures in Section 3.3). Coupons were stored in closed test boxes during the simulated weathering pre-decontamination phase to reduce evaporation due to the high air flow conditions inside the chemical safety hood. Immediately prior to decontamination, contaminated stainless steel coupons were placed horizontally on pre-cleaned latex spacers to allow collection of liquid waste. Contaminated painted drywall coupons for vertical orientation (most common orientation in indoor setting) testing were secured in pre-cleaned custom-made coupon holders. Figure 3-4 shows examples of stainless steel and painted drywall coupon assemblies readied for horizontal or vertical testing.



**Figure 3-4. Stainless steel and painted drywall coupon assembly readied for testing.**

Details of each decontamination procedure that was tested are given in Sections 3.6.2. and 4.1. During the post-decontamination phase (dwell time of 1 h to 28 h, followed by a rinse step and overnight drying [24 h]), coupons were stored in the chemical hood in open test boxes (Section 4.1 discusses experimental details of single- and multistep experimental procedures, including procedure-specific processing and drying times). After drying, coupons were sampled using methods described in Section 4.2.

### 3.5. Method Development Tests

#### 3.5.1. Sampling and Extraction for Surface Samples

For surface sampling and wipe extraction efficacy tests, all test surfaces were coated with solutions of target chemicals using the procedure described in Section 3-3 and placed in the same type of pre-cleaned test box that was used during the decontamination testing. After a short contact time of the chemical (CT=30 minutes (min) with the surface, wipe samples were collected and extracted using the procedures described in Section 4.2.

The wetting solvent used for sampling optimization was isopropyl alcohol (IPA) (certified ACS, Fisher Chemical, Waltham, MA; product # Ah16-4 UN1219), and this solvent was selected based on previous research efforts [6] over dichloromethane as IPA is less prone to evaporation and is less destructive to the surface. The wetting solvent volume was 3 mL per wipe (Cotton Twill wipes, MG Chemicals Ontario, Canada; Part No. 829-4x4), resulting in semi-saturation of the wipe. Post-sample collection, wipes were extracted and prepared for analysis as described in Section 4.4. Each test set consisted of three TCs and one procedural blank; there was one solvent blank and one control spike sample per test day per chemical. Initial optimization of surface sampling was performed in the horizontal orientation only. The test matrix is shown in Table 3-3.

**Table 3-3. Experimental parameters for surface contamination, wipe sampling and extraction optimization tests**

Chemical/Test Material	Target Chemical Concentration (mg) <sup>a</sup>	Wiping Medium Type <sup>b</sup>	Number of Wipes per Coupon <sup>c</sup>	Wetting Solvent		Extraction Solvent	
				Type	Volume (mL)	Type	Volume (mL)
Malathion/stainless steel	4	Cotton twill wipe	3	Isopropyl alcohol	3	Hexane	50
Malathion/painted drywall	4	Cotton twill wipe	3	Isopropyl alcohol	3	Hexane	50
Carbaryl/stainless steel	24	Cotton twill wipe	3	Isopropyl alcohol	3	Hexane:Acetone <sup>d</sup>	50
Carbaryl/painted drywall	24	Cotton twill wipe	3	Isopropyl alcohol	3	Hexane:Acetone <sup>d</sup>	50

<sup>a</sup>Per test area of 12 in x 12 in (929 cm<sup>2</sup>), <sup>b</sup>See Section 4.1 for detailed product information, <sup>c</sup> Three wipes per coupon collected and extracted as a composite sample, <sup>d</sup>10:1 v/v

### 3.5.2. Optimization of Chemical Delivery to Surface Samples

Three rounds of airbrush delivery method optimization for malathion and carbaryl were performed. Optimization to reach target surface concentrations involved changing the concentration of the nebulization solution and airbrush fill volume (3 to 36 mg/mL at 4 to 8 mL fill volume), as well as introduction of a nebulization shield that prevented disruption of the nebulization cloud during airbrush application, as shown in Figure 3-5.



**Figure 3-5. Assembled test setup with nebulization shield placed around test box.**

After three rounds of method optimization (data not shown), malathion results for the nonporous reference material (stainless steel) were within project-specific acceptance criteria of 60-140% of the target surface concentration target with an average recovery of  $4.7 \pm 0.30$  mg per coupon (target concentration =  $4 \text{ mg} \pm 1.6 \text{ mg}$  per coupon). Meanwhile, the average recovery of carbaryl (at  $95 \pm 15.2$  mg) was approximately four times higher than the surface concentration target of  $24 \pm 9.6$  mg per coupon. The elevated surface delivery rate/surface concentrations for carbaryl could have been due to higher settling rates (compared to malathion). Nebulized carbaryl solutions were observed to have formed prominent (visible) clouds with airborne-particulate-like characteristics that, after settling, were producing powder-like surface chemical films. In comparison, the malathion chemical clouds seemed to have the finer translucent chemical droplet characteristics and ultimately formed an oily sheen film on the surface. Additional method optimization for the carbaryl delivery amount was not performed due to the project time constraints. The concentration of carbaryl in the nebulization solution was subsequently reduced from 36 mg/mL to 8 mg/mL to account for the higher recovery of carbaryl, and the solution with the lower concentration was then used in follow-on decontamination testing without further optimization of the target surface concentration. Importantly, optimized methods resulted in chemical surface concentrations at the desired milligrams per square meter ( $\text{mg}/\text{m}^2$ ) level (Section 3-2) for both pesticides (results are provided in Table 3-4).

Results for chemical surface loading of stainless steel positive control coupons (from test-day-specific batches of decontamination test samples) are given in Table 3-4, below. These results indicate that the optimized airbrush nebulization method provided a reproducible delivery of high-surface-concentration (pesticide) chemical films on a nonporous material, characterized by low intra- and inter-test variability (relative standard deviation [RSD] < 30%) for the reference nonporous material (stainless steel).

**Table 3-4. Chemical surface loading of PCs (decontamination tests)**

Chemical	Malathion		Carbaryl	
Material	Stainless steel			
ID <sup>a</sup>	mg/coupon	mg/m <sup>2</sup>	mg/coupon	mg/m <sup>2</sup>
<b>Bleach Decontamination Tests DCP1 and DCP2 [Batch #1]</b>				
PC-1	14.0	151	18.6	201
PC-2	14.5	156	22.6	243
PC-3	16.0	172	12.8	138
Average (n=3)	14.8	160	18.0	194
SD	1.0	11.2	4.9	53.0
%RSD	7%		27%	
<b>Bleach Decontamination Tests DCP3, DCP4 and DCP5 [Batch #2]</b>				
PC-1	15.5	167	19.3	208
PC-2	20.0	215	19.1	205
PC-3	18.0	194	20.4	220
Average (n=3)	17.8	192	19.6	211
SD	2.3	24.3	0.74	7.9
%RSD	13%		4%	
<b>EasyDECON® DF200 Decontamination Tests DCP1 and DCP2 [Batch #3]</b>				
PC-1	13.0	140	19.0	204
PC-2	15.5	167	14.9	160
PC-3	15.0	161	19.8	213
Average (n=3)	14.5	156	17.9	193
SD	1.3	14.2	2.6	28.2
%RSD	9%		15%	
<b>EasyDECON® DF-200 Decontamination Tests DCP3, DCP4 and DCP5 [Batch #4]</b>				
PC-1	10.0	108	18.2	196
PC-2	12.0	129	21.6	232
PC-3	17.5	188	18.7	201
Average (n=3)	13.2	142	19.5	210
SD	3.9	41.8	1.8	19.4
%RSD	29%		9%	
<sup>a</sup> DCP 1 through 5-Decontamination Cleanup Procedure; see Tables 3-2 and 3-3 for experimental details. PC-positive control coupon; (1-3)- replicate sample number; SD = Standard deviation; RSD = Relative standard deviation.				

The analysis of airbrush application results performed by different personnel suggests that there is an operator-related variation between tests. Here, the method development tests discussed in the first paragraph of this section were performed by a different cross-trained analyst than the decontamination test-

related PC contamination results in Table 3-4. This change in analyst resulted in unexpected significant changes in amounts delivered to the surface via the airbrush. However, this change was not consequential in terms of reaching the overall surface target threshold of milligrams per square meter ( $\text{mg}/\text{m}^2$ ). The differences in surface loadings could be due to varying the distance of the airbrush tip from the coupon surface, the ergonomic work-space arrangements (analyst sitting versus standing during application), or the analyst-preferred (allowing best dexterity while maintaining safety) position of the chemical hood sash (theoretically affecting the air flow around the nebulization shield). The position of the sash in this study was at an operationally safe level to protect the breathing zone of the analysts, but the sash was not set to a specific level or controlled throughout the entire testing. Other factors not yet identified could have contributed to variation in surface loading. The results suggest that the nebulization procedure for inter-related test subsets/batches should be performed by the same analyst and under the same operational conditions in the chemical hood (e.g., using a fixed position of the safety sash, frequent calibration of the flow/face velocity, etc.). Additionally, the intra-personnel cross-comparison checks should be completed prior to experiments that are to be performed by multiple analysts.

The recovery of malathion from painted drywall during the method development tests was  $0.42 \pm 0.12$  mg per coupon (or 9% of the chemical amount recovered from stainless steel). The recovery of carbaryl from painted drywall was 11% of the recovery from stainless steel ( $10.9 \pm 1.96$  mg per coupon). These lower recoveries from painted drywall may have been due to migration of the pesticide solution into the semi-porous painted drywall material, with the consequent inaccessibility of the target chemical for surface wipe sampling and potentially the reduced susceptibility of permeated chemicals to decontamination. Similar low recoveries from semi-porous materials have been reported for OP chemical warfare agents [6]. Therefore, decontamination testing was not performed on painted drywall material. There is a continued need for the development of (wipe) sampling methods that can sample and/or extract (residual) chemical agent from a semi-porous material. As mentioned earlier, the phenomena of the chemical permeation into semi-porous materials and optimization of decontamination strategies for permeated persistent chemicals are currently being studied under a separate research effort.

### 3.5.3. Persistence and Uptake of Chemicals by Test Coupon Materials

As described in the previous section, the CT for the pesticides ranges from 25 h (single step DCP; CT = 24 h plus DT1 = 1 h) to 52 h (multistep DCP; CT = 24 h plus DT2 = 4 h plus DT3 = 24 h). The evaporation and/or other losses (e.g., indoor UV light-related degradation) were not expected to be significant as previous research [7] demonstrated that both carbaryl and malathion are highly persistent on stainless steel surfaces. However, some amount of pesticide was expected to transfer into the semi-porous test materials during the chemical-surface CT.

Independent tests for the persistence and uptake of the chemicals by the test materials were executed prior to the decontamination testing to verify whether a single time point could function as a single set of PCs. PCs would be spiked at the same time as the test coupons. However, extraction by wipe sampling would occur at a different CT that would consider the workload on Day 1 and Day 2 of each test. The persistence and uptake tests consisted of several measurements of the surface concentration of the pesticides taken after 30 min, 24 h, and 48 h (Table 3-5). The latter time was reduced to 46 h due to constraints in the work schedule. Each test consisted of three sets of three TCs and one PB. All PBs were sampled only at total contact time corresponding to the maximum contact time for this study (46 h).

Coupons were contaminated in the horizontal position, and tests were performed in the horizontal and vertical orientations for stainless steel and painted drywall, respectively.

**Table 3-5. Samples for persistence and uptake of chemical by test material**

Chemical/Surface	Contact Time		
	30 min	24 h	46 h
Malathion/stainless steel	TC = 3	TC = 3	TC = 3, PB = 1
Malathion/painted drywall	TC = 3	TC = 3	TC = 3, PB = 1
Carbaryl/stainless steel	TC = 3	TC = 3	TC = 3, PB = 1
Carbaryl/painted drywall	TC = 3	TC = 3	TC = 3, PB = 1
<i>TC- test coupon, PB- procedural blank.</i>			

Each set of TCs was contaminated with the chemical using the procedure described in Section 3.3. The coupons were then placed in the same type of pre-cleaned transparent test box that was used during the decontamination testing; boxes remained closed in the same manner as during the pre-decontamination phase of testing to mitigate the effect of high air flow and air exchange rates in the chemical safety hood, as the ventilation rates in the chemical hood were not considered representative of normal air exchange/ventilation rates in indoor settings. After the prescribed CT (Table 3-5) was completed for each set of coupons, sampling and extraction of the coupons took place following the procedures described in Sections 4.1 and 4.2. Samples were prepared for analysis as described in Section 4.2. Results of the tests for uptake of chemical by test material are given in Section 5.1.

#### 3.5.4. Chemical Uptake by Cleaning Media and Transfer to Liquid Effluents

Physical removal of chemicals from the test material surfaces was expected for scrubbing-, wiping- and to a lesser extent, roll-on-based approaches using cloths, sponges, and paint rollers (DCPs 3, 4, and 5 described in Section 3.6.2). For these DCPs, the uptake of chemical by the cleaning media (from the reference material, stainless steel) was tested using a single-step method for a contact time of 30 minutes (Table 3-5). The chemical uptake by cleaning media was not studied for painted drywall due to overall low surface recovery observed for the semi-porous material as discussed in Section 3.5.2. Each test consisted of three TCs complemented by one material- and chemical-specific PB. Tests were performed in the horizontal orientation only. All cleaning media were pre-wetted with deionized (DI) water to decouple physical removal of chemicals by various types of cleaning media from the neutralizing action of the decontaminants. The amount of water needed to saturate each sampling medium corresponded to the wetting volume of decontaminant that had been determined prior to testing and varied from 50 to 150 mL of water, depending on type of cleaning media (media-specific wetting volumes are given in Section 3.6.2.1). There was no water rinse in this test.

Each set of stainless steel TCs was contaminated with pesticide using the procedure described in Section 3.3 and then placed in the same transparent test boxes used during the decontamination testing. Surfaces were decontaminated with water only, using procedures described in Section 3.6.2.1. The liquid

run-offs were collected and extracted using procedures described in Section 4.3. After the conclusion of the coupon cleaning, the expended cleaning media were collected (as solid waste) for extraction, and surface-wipe samples were collected to allow determination of chemical remaining on each coupon surface after deployment of the mechanical cleaning step. Surface samples were collected and extracted using methods described in Section 4.2.

The test matrix for the uptake of pesticide by cleaning media and transfer to liquid effluent is given in Table 3-6, below. Results are summarized in Section 5.3.

**Table 3-6. Uptake of chemical by cleaning media and transfer to liquid effluent test**

Cleaning Medium Type	Cotton Cloth	Sponge	Paint Roller
Type of Sample	Malathion/Stainless Steel		
	Number of Samples		
Solid waste <sup>a</sup>	TC = 3, PB = 1	TC = 3, PB = 1	TC = 3, PB = 1
Liquid waste <sup>b</sup>	TC = 1(C), PB = 1	TC = 1(C), PB = 1	TC = 1(C), PB = 1
Surface wipe <sup>c</sup>	TC = 3, PB = 1	TC = 3, PB = 1	TC = 3, PB = 1
Type of Sample	Carbaryl/Stainless Steel		
	Number of Samples		
Solid waste <sup>a</sup>	TC = 3, PB = 1	TC = 3, PB = 1	TC = 3, PB = 1
Liquid waste <sup>b</sup>	TC = 1(C), PB = 1	TC = 1(C), PB = 1	TC = 1(C), PB = 1
Surface wipe <sup>c</sup>	TC = 3, PB = 1	TC = 3, PB = 1	TC = 3, PB = 1

<sup>a</sup> Expended cleaning media; <sup>b</sup> Composite sample of run-offs collected during cleaning; <sup>c</sup> Wipe samples collected post-cleaning; three wipes per coupon collected and extracted as a composite sample (C); TC- test coupon, PB- procedural blank

### 3.6. Decontamination Tests

#### 3.6.1. Preparation of Decontamination Solutions

Decontaminants used in this study have proven efficacious for both malathion and carbaryl in previous research efforts [7]. General information and properties of decontamination solutions are given in Table 3-7.

**Table 3-7. Decontamination solutions**

Solution	Manufacturer/Supplier Name/Location	Active Ingredient	pH Range
EasyDECON® DF200	Envirofoam Technologies, Pooler, GA, USA/Intelgard, Lafayette, CO, USA	Hydrogen peroxide	9.6–9.7
Clorox® concentrated germicidal bleach (8.25% sodium hypochlorite)	The Clorox® Company, Oakland, CA	Hypochlorite ion/hypochlorous acid	11–12

Fresh batches of EasyDECON® DF200 solution were prepared daily through proportional mixing as per the manufacturer's instructions in amounts sufficient for testing (e.g., to make 2 L of decontamination solution, 950 mL of EasyDECON® DF200 Part 1 was mixed with 1010 mL of EasyDECON® DF200 Part 2, and then 40 mL of EasyDECON® DF200 Part 3 was added). After mixing, the manufacturer recommends

the use of the EasyDECON® DF200 Fortifier Test Kit to test the stability of the EasyDECON® DF200 final blend. This test (a “Go/No Go” test) measures the percentage of active ingredient and instills confidence that the decontamination solution is effective and ready to use. The ongoing evaluations that occurred prior to use also included pH measurements of the finished blend (target pH range: 9.6-9.9). Results are discussed in Section 6.0 (Table 6-2). Concentrated Clorox® germicidal bleach was used as is (no preparation was needed); no evaluation of the concentrated germicidal bleach solution was performed. DI water was used for post-decontamination rinses.

### 3.6.2. Decontamination Procedures

Various decontamination cleanup procedures were evaluated for their ability to decontaminate pesticides deposited onto test surfaces. Tested DCPs involved several cleaning techniques, including spray-on-only methods (with no mechanical removal step) and methods that allowed a potential physical removal of contamination by the cleaning medium (wiping/scrubbing and roll-on applications). Transfer of contamination to the cleaning media was tested for the latter category (Section 3.5.3). For each application method, two general decontamination approaches were tested:

1. **A simplified/expedient (single-step) approach:** The chosen decontamination solution was applied once and allowed to interact with the material for one hour. Then, test surfaces were rinsed, allowed to dry overnight, and sampled for residual pesticides.

2. **A multi-application/multi-day (multistep) approach:** The chosen decontamination solution was applied twice within the first four hours of the decontamination process and allowed to interact with the material for a total of 24 hours. Then, test surfaces were rinsed, allowed to dry overnight, and sampled for residual pesticides.

#### 3.6.2.1. Cleaning Media

Decontamination solutions were applied using commercially available equipment (hand-held sprayer) and basic household-use supplies (cleaning-grade spray bottle, general-purpose cleaning cloth, general-purpose cleaning sponge, and paint roller). The product information and specifications of cleaning media that were used in this study are given below:

1. **Hand-Held Pressurized Sprayer.** The spray gun (MeterJet™ Gunjet Spray Gun Kit, Forestry Suppliers, Inc., Jackson, MS, USA; Figure 3-6) used in this study can deliver a metered volume of spray ranging from 1 to 16 mL with  $\pm 2\%$  accuracy. This spray gun was used successfully for applications of numerous cleaning agents in previous research [8].



Figure 3-6. Hand-held pressurized sprayer

2. **Cleaning-Grade Hand-Held Spray Bottle.** A durable industrial sprayer bottle (Figure 3-7) was purchased from a national supplier (Lowe's Companies, Inc., Mooresville, NC, USA; 32-ounce (oz)

plastic spray bottle; Lowe's Item No. 366843, Model No. LOAPS30). This type of bottle is equipped with a trigger sprayer with no adjustable spray pattern and is recommended by the manufacturer for general household cleaning purposes, including application of concentrated formulas.



**Figure 3-7. Cleaning-grade hand-held spray bottle.**

- Absorbent Cleaning-Grade Cloth.** Cleaning-grade 100% cotton cloths (Figure 3-8), approximately 14 in x 17 in each, were purchased from a national supplier (ProLine 48-count terry towels; Lowe's Companies, Inc., Mooresville, NC, USA; Lowe's Item No. 503439, Model No. T-99765). These cloths are recommended by the manufacturer for multipurpose cleaning tasks. Each towel was folded twice prior to wetting to allow better manageability during application of the decontamination solution onto the 14 in x 14 in coupon.



**Figure 3-8. Absorbent cleaning cloth.**

- Perforated Synthetic Wash Sponge.** Cleaning-grade polyurethane sponges (Figure 3-9), approximately 4.5 in x 7 in each, were purchased from a national supplier (ProLine polyurethane sponge; Lowe's Companies, Inc., Mooresville, NC, USA; Lowe's Item No. 469322, Model No. K-56P). This type of sponge is recommended by the manufacturer for multipurpose cleaning tasks. The sponge product specifications were inspected for any added ingredients/additives prior to use; no additional additives were noted. Sponges were pre-cut in half using a precision blade pre-cleaned with ethanol to allow easy folding for the subsequent large-volume extraction step (described in Section 4.3).



**Figure 3-9. Perforated synthetic wash sponge.**

- Paint Roller.** Polyester regular paint roller covers and rollers (Figure 3-10), approximately 9 in each, were purchased from a national supplier (Blue Hawk, Lowe's Companies, Inc., Mooresville, NC, USA; Lowe's Item No. 299909, Model No. 1838181). This type of paint roller (with a 3/8 in nap) is recommended by the manufacturer for use on smooth surfaces. Compatible roller handles were purchased separately. Paint rollers were pre-cut in half using a precision blade pre-cleaned with ethanol to allow easy folding for the subsequent large-volume extraction step (described in Section 4.3).



**Figure 3-10. Paint roller cover.**

Decontaminant and water rinses were applied using step-specific cleaning media (Table 3-9 and 3-10). The surface of the coupon was always cleaned using horizontal (left to right) overlapping strokes that were applied from top to bottom of each coupon. Twenty mL of decontaminant or water was pre-loaded for pressurized sprayer application, based on a 20 x 1 mL spray pattern. Following daily calibrations with DI water, spray bottles were preloaded with 100 mL, and 20 mL of solution was delivered onto each coupon. For other cleaning media, wetting volumes were delivered experimentally to allow a uniform saturated (but non-dripping) wetting. The media wetting volumes were determined gravimetrically by weighing each dry cleaning medium and then weighing the wetted medium again, before application onto the coupon surface. A 100 mL of solution was used per sponge, 150 mL of solution was used per cotton cloth, and 50 mL of solution per paint roller (pre-cut in half). All cleaning media were pre-loaded prior to testing and placed in individual plastic bags, and bags were closed to avoid non-specific decontaminant losses. The EasyDECON® DF200 pre-loaded cotton cloths heated significantly (~38 °C), and there was unidentified gas build-up prompting venting of plastic bags prior to deployment. There were no other incompatibilities observed between cleaning media and decontaminants.

Decontamination testing (spiking, decontamination application, and wipe sampling) was conducted by one laboratory support person to limit inter-personal variance. Decontamination technique vigor (i.e., media application pressure and velocity) mimicked typical household cleaning.

The consequent surface loadings (amount of decontaminant or rinse applied onto each coupon) were determined gravimetrically by weighing each test box before and after application. Accuracy of the scale was sufficient to measure an absolute mass change of 1 g, an equivalent of approximately 1 mL of

liquid decontaminant or water rinse. The average decontaminant and water (rinse) surface loading volumes for each cleaning medium are given below in Table 3-8. Sample-specific results are given in Appendix A, Tables A-1 through A-8.

**Table 3-8. Average surface loadings of decontaminant solutions and water rinses for different cleaning media.**

Type of Decontaminant	Pressurized Sprayer		Spray Bottle		Cotton Cloth		Cleaning Sponge		Paint Roller	
	Surface Loading per Test Coupon*									
	mL	±SD	mL	±SD	mL	±SD	mL	±SD	mL	±SD
Concentrated Germicidal Bleach	18.2	NA	18.7	0.83	3.41	0.43	24.7	6.7	4.2	1.4
EasyDECON® DF200	16.9	NA	17.3	0.73	6.02	1.58	22.3	8.7	6.3	3.8
Water rinse	20.0	NA	20.4	0.6	3.82	2.50	17.1	8.2	7.1	5.2

*\*Calculated from gravimetric measurements of pre- and post-applications and specific gravities of decontaminants; for pressurized sprayer, application was based on a 20 x 1 mL spray volume following daily calibrations with DI water.*

*SD: Standard Deviation; NA: Not Applicable*

Other test-specific experimental parameters for the single- and multistep procedures (contact times for chemical weathering, dwell times for decontaminant processing) are given in Appendix A in Tables A-1 through A-8.

Figure 3-11 shows examples of decontamination solution application using different cleaning procedures and post-application appearance of test surfaces (immediately post-application of decontaminant). Examples shown are for EasyDECON® DF200 applications.



Figure 3-11. DCP1 through DCP5 application of decontaminant using various cleaning media and resulting appearance of the TC surface

### *3.6.2.2. Test Matrix*

The decontamination test matrix is shown in Tables 3-9 and 3-10. Three decontaminated TCs were used for each chemical for single- and multistep methods, three PC coupons per test per cleaning medium (coupons contaminated with chemical that did not undergo decontamination), and one PB (coupon not spiked with chemical that will undergo decontamination in the horizontal orientation). Additionally, one control spike sample was prepared per test day to check for nominal concentration of spiking solution as well as for ongoing laboratory proficiency testing; this sample was prepared as a direct spike of chemical solution to hexane, at a level corresponding to 100% of the target surface concentration of chemical expected in the final extract. Test matrices for malathion and carbaryl decontamination testing are summarized in Tables 3-9 and 3-10, respectively; and sample process design for single- and multistep testing is given in Section 4.1. Decontamination test results are given in Section 5.2.

**Table 3-9. Test Matrix for Malathion Decontamination Testing**

Test ID	Procedure	Surface	Orientation	Decontaminant	Application of Decontaminant	Water Rinse #1	Reapplication of Decontaminant	Water Rinse #2
DCP1S-MA-SS-BL-PS-1	S	Stainless steel	Horizontal	Concentrated germicidal bleach	Pressurized sprayer 1	Pressurized sprayer 2	No	No
DCP1S-MA-SS-BL-PS-4/24	M	Stainless steel	Horizontal	Concentrated germicidal bleach	Pressurized sprayer 1	No	Pressurized sprayer 1	Pressurized sprayer 2
DCP1S-MA-SS-ED-PS-1	S	Stainless steel	Horizontal	EasyDECON® DF200	Pressurized sprayer 1	Pressurized sprayer 2	No	No
DCP1S-MA-SS-ED-PS-4/24	M	Stainless steel	Horizontal	EasyDECON® DF200	Pressurized sprayer 1	No	Pressurized sprayer 1	Pressurized sprayer 2
DCP2S-MA-SS-BL-SB-1	S	Stainless steel	Horizontal	Concentrated germicidal bleach	Spray bottle 1	Spray bottle 2	No	No
DCP2S-MA-SS-BL-SB-4/24	M	Stainless steel	Horizontal	Concentrated germicidal bleach	Spray bottle 1	No	Spray bottle 1	Spray bottle 2
DCP2S-MA-SS-ED-SB-1	S	Stainless steel	Horizontal	EasyDECON® DF200	Spray bottle 1	Spray bottle 2	No	No
DCP2S-MA-SS-ED-SB-4/24	M	Stainless steel	Horizontal	EasyDECON® DF200	Spray bottle 1	No	Spray bottle 1	Spray bottle 2
DCP3S-MA-SS-BL-RG-1	S	Stainless steel	Horizontal	Concentrated germicidal bleach	Wetted cloth 1	Wetted cloth 2	No	No
DCP3S-MA-SS-BL-RG-4/24	M	Stainless steel	Horizontal	Concentrated germicidal bleach	Wetted cloth 1	No	Wetted cloth 1	Wetted cloth 2
DCP3S-MA-SS-ED-RG-1	S	Stainless steel	Horizontal	EasyDECON® DF200	Wetted cloth 1	Wetted cloth 2	No	No
DCP3S-MA-SS-ED-RG-4/24	M	Stainless steel	Horizontal	EasyDECON® DF200	Wetted cloth 1	No	Wetted cloth 1	Wetted cloth 2
DCP4S-MA-SS-BL-SP-1	S	Stainless steel	Horizontal	Concentrated germicidal bleach	Wetted sponge 1	Wetted sponge 2	No	No
DCP4S-MA-SS-BL-SP-4/24	M	Stainless steel	Horizontal	Concentrated germicidal bleach	Wetted sponge 1	No	Wetted sponge 1	Wetted sponge 2
DCP4S-MA-SS-ED-SP-1	S	Stainless steel	Horizontal	EasyDECON® DF200	Wetted sponge 1	Wetted sponge 2	No	No
DCP4S-MA-SS-ED-SP-4/24	M	Stainless steel	Horizontal	EasyDECON® DF200	Wetted sponge 1	No	Wetted sponge 1	Wetted sponge 2
DCP5S-MA-SS-BL-PR-1	S	Stainless steel	Horizontal	Concentrated germicidal bleach	Wetted paint roller 1	Wetted paint roller 2	No	No
DCP5S-MA-SS-BL-PR-4/24	M	Stainless steel	Horizontal	Concentrated germicidal bleach	Wetted paint roller 1	No	Wetted paint roller 1	Wetted paint roller 2
DCP5S-MA-SS-ED-PR-1	S	Stainless steel	Horizontal	EasyDECON® DF200	Wetted paint roller 1	Wetted paint roller 2	No	No
DCP5S-MA-SS-ED-PR-4/24	M	Stainless steel	Horizontal	EasyDECON® DF200	Wetted paint roller 1	No	Wetted paint roller 1	Wetted paint roller 2

*MA-malathion; BL-bleach; ED-EasyDECON® DF200; DCP 1 to 5-Decontamination Cleanup Procedure 1 to 5; PS-pressurized sprayer; SB-spray bottle; RG-cotton cloth; SP- sponge; PR-paint roller; S/ M-single or -multistep procedure.*

**Table 3-10. Test Matrix for Carbaryl Decontamination Testing**

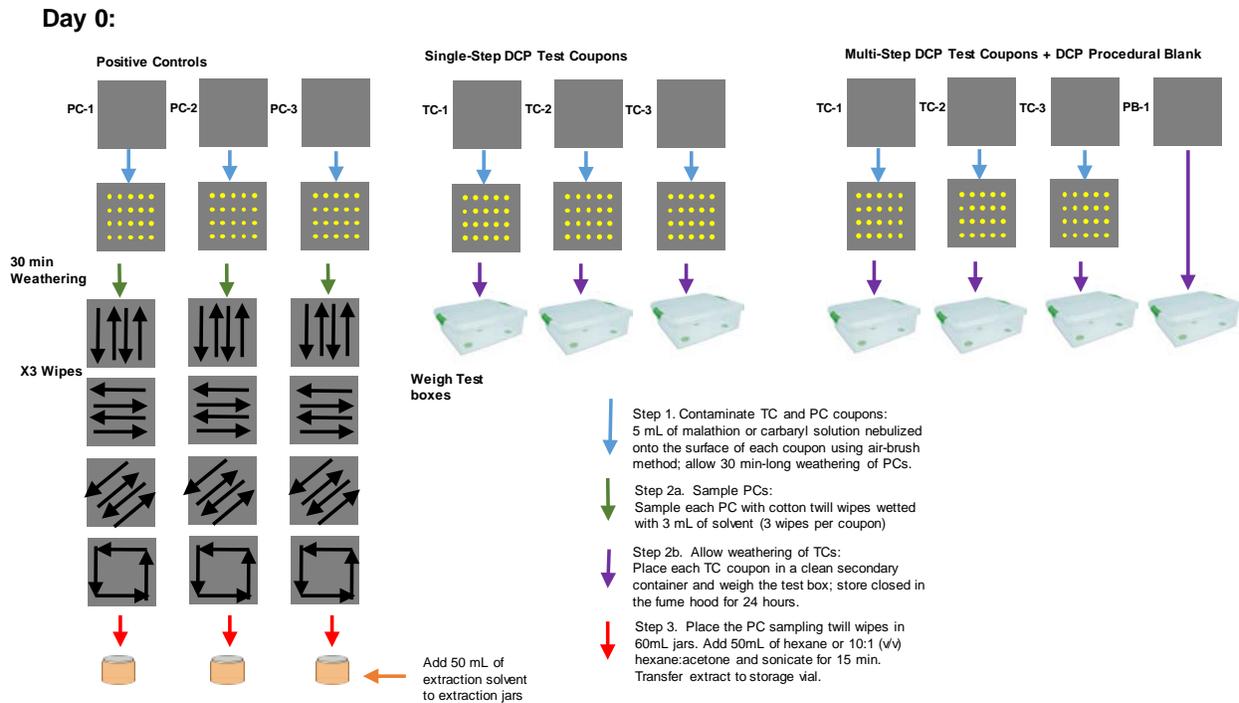
Test ID	Procedure	Surface	Orientation	Decontaminant	Application of Decontaminant	Water Rinse #1	Reapplication of Decontaminant	Water Rinse #2
DCP1S-CA-SS-BL-PS-1	S	Stainless steel	Horizontal	Concentrated germicidal bleach	Pressurized sprayer 1	Pressurized sprayer 2	No	No
DCP1S-CA-SS-BL-PS-4/24	M	Stainless steel	Horizontal	Concentrated germicidal bleach	Pressurized sprayer 1	No	Pressurized sprayer 1	Pressurized sprayer 2
DCP1S-CA-SS-ED-PS-1	S	Stainless steel	Horizontal	EasyDECON® DF200	Pressurized sprayer 1	Pressurized sprayer 2	No	No
DCP1S-CA-SS-ED-PS-4/24	M	Stainless steel	Horizontal	EasyDECON® DF200	Pressurized sprayer 1	No	Pressurized sprayer 1	Pressurized sprayer 2
DCP2S-CA-SS-BL-SB-1	S	Stainless steel	Horizontal	Concentrated germicidal bleach	Spray bottle 1	Spray bottle 2	No	No
DCP2S-CA-SS-BL-SB-4/24	M	Stainless steel	Horizontal	Concentrated germicidal bleach	Spray bottle 1	No	Spray bottle 1	Spray bottle 2
DCP2S-CA-SS-ED-SB-1	S	Stainless steel	Horizontal	EasyDECON® DF200	Spray bottle 1	Spray bottle 2	No	No
DCP2S-CA-SS-ED-SB-4/24	M	Stainless steel	Horizontal	EasyDECON® DF200	Spray bottle 1	No	Spray bottle 1	Spray bottle 2
DCP3S-CA-SS-BL-RG-1	S	Stainless steel	Horizontal	Concentrated germicidal bleach	Wetted cloth 1	Wetted cloth 2	No	No
DCP3S-CA-SS-BL-RG-4/24	M	Stainless steel	Horizontal	Concentrated germicidal bleach	Wetted cloth 1	No	Wetted cloth 1	Wetted cloth 2
DCP3S-CA-SS-ED-RG-1	S	Stainless steel	Horizontal	EasyDECON® DF200	Wetted cloth 1	Wetted cloth 2	No	No
DCP3S-CA-SS-ED-RG-4/24	M	Stainless steel	Horizontal	EasyDECON® DF200	Wetted cloth 1	No	Wetted cloth 1	Wetted cloth 2
DCP4S-CA-SS-BL-SP-1	S	Stainless steel	Horizontal	Concentrated germicidal bleach	Wetted sponge 1	Wetted sponge 2	No	No
DCP4S-CA-SS-BL-SP-4/24	M	Stainless steel	Horizontal	Concentrated germicidal bleach	Wetted sponge 1	No	Wetted sponge 1	Wetted sponge 2
DCP4S-CA-SS-ED-SP-1	S	Stainless steel	Horizontal	EasyDECON® DF200	Wetted sponge 1	Wetted sponge 2	No	No
DCP4S-CA-SS-ED-SP-4/24	M	Stainless steel	Horizontal	EasyDECON® DF200	Wetted sponge 1	No	Wetted sponge 1	Wetted sponge 2
DCP5S-CA-SS-BL-PR-1	S	Stainless steel	Horizontal	Concentrated germicidal bleach	Wetted paint roller 1	Wetted paint roller 2	No	No
DCP5S-CA-SS-BL-PR-4/24	M	Stainless steel	Horizontal	Concentrated germicidal bleach	Wetted paint roller 1	No	Wetted paint roller 1	Wetted paint roller 2
DCP5S-CA-SS-ED-PR-1	S	Stainless steel	Horizontal	EasyDECON® DF200	Wetted paint roller 1	Wetted paint roller 2	No	No
DCP5S-CA-SS-ED-PR-4/24	M	Stainless steel	Horizontal	EasyDECON® DF200	Wetted paint roller 1	No	Wetted paint roller 1	Wetted paint roller 2

*CA--carbaryl; BL-bleach; ED-EasyDECON® DF200; DCP 1 to 5-Decontamination Cleanup Procedure 1 to 5; PS-pressurized sprayer; SB-spray bottle; RG-cotton cloth; SP- sponge; PR-paint roller; S/ M-single or -multistep procedure.*

## 4.0 Sampling and Analysis Methods

### 4.1. Sample Process Design for Single- and Multistep Testing

A multiday sample process/experimental design was used for each DCP test. The sample process for single- and multistep decontamination testing is shown in Figures 4-1 through 4-6, respectively. Each decontamination test was accompanied by collection of reference (non-decontaminated) PC coupons. A PB was tested for each DCP deployed using the multistep method. The wipe sampling and extraction methods are summarized in detail in Section 4.2.



**Figure 4-1. Day 0 experimental design and sample flow for single- and multistep DCPs: contamination of coupons and surface sampling of PCs.**

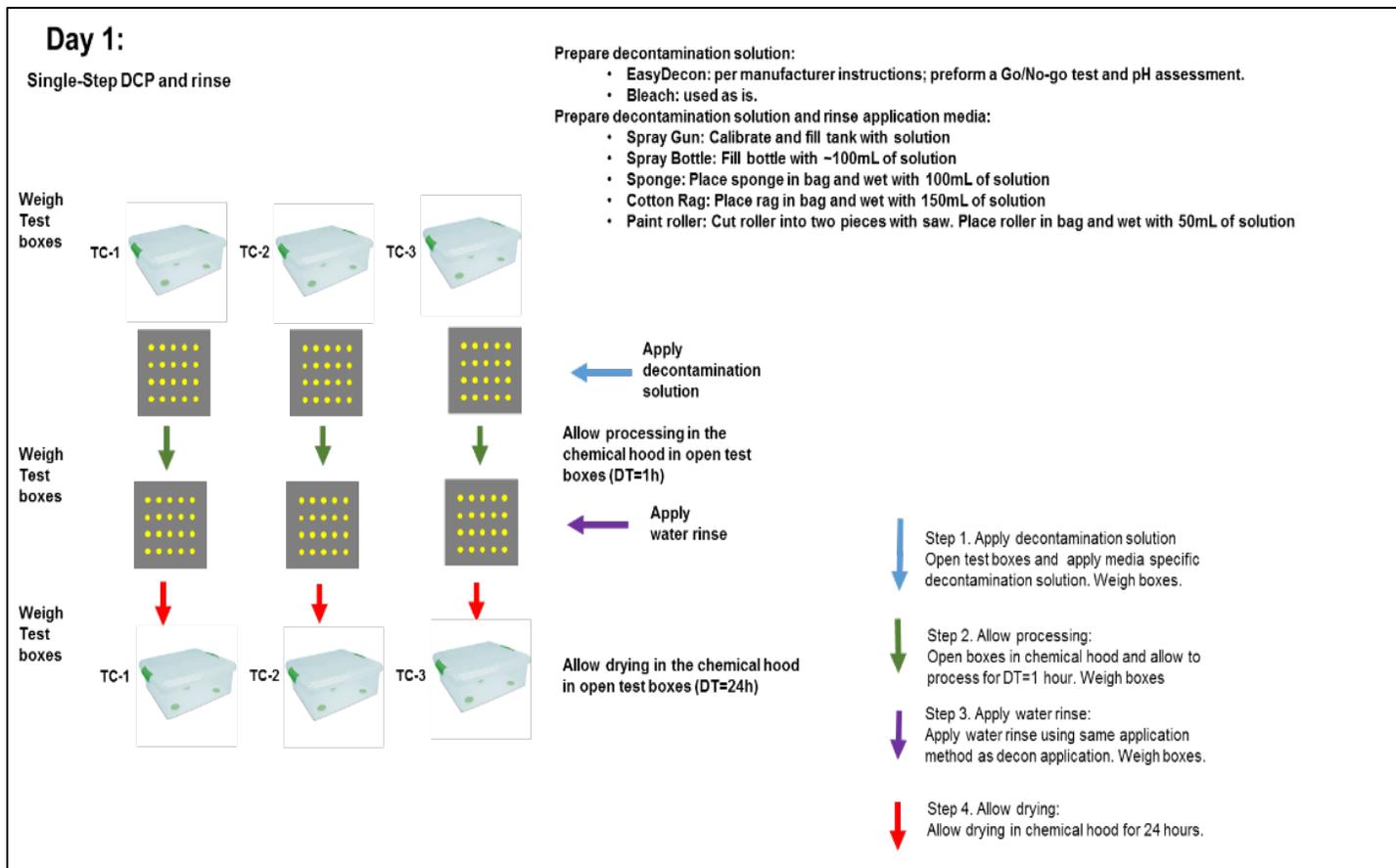


Figure 4-2. Day 1 experimental design and sample flow for single-step DCPs: application of decontaminant and water rinse.

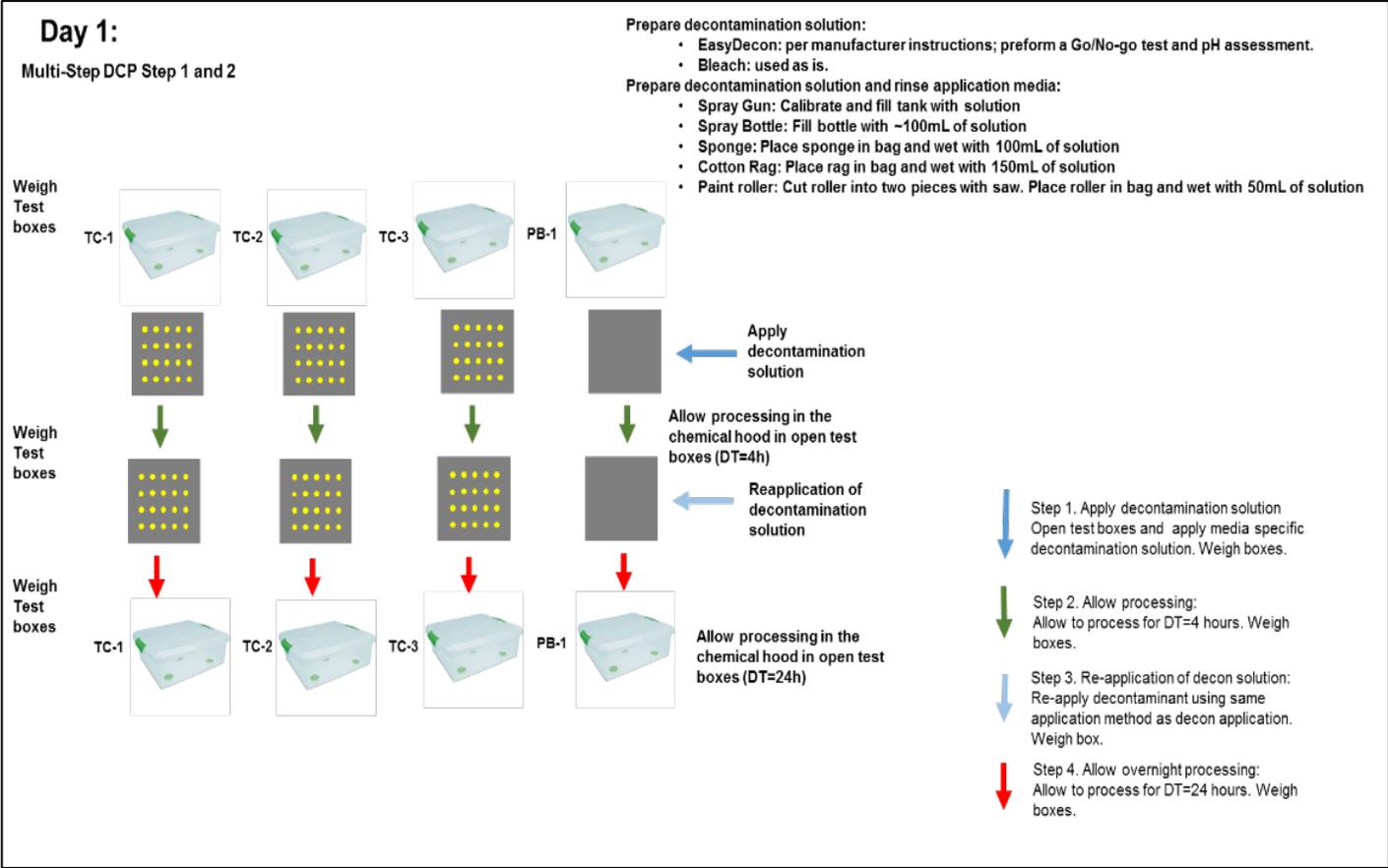


Figure 4-3. Day 1 experimental design and sample flow for multistep DCPs: application and re-application of decontaminant.

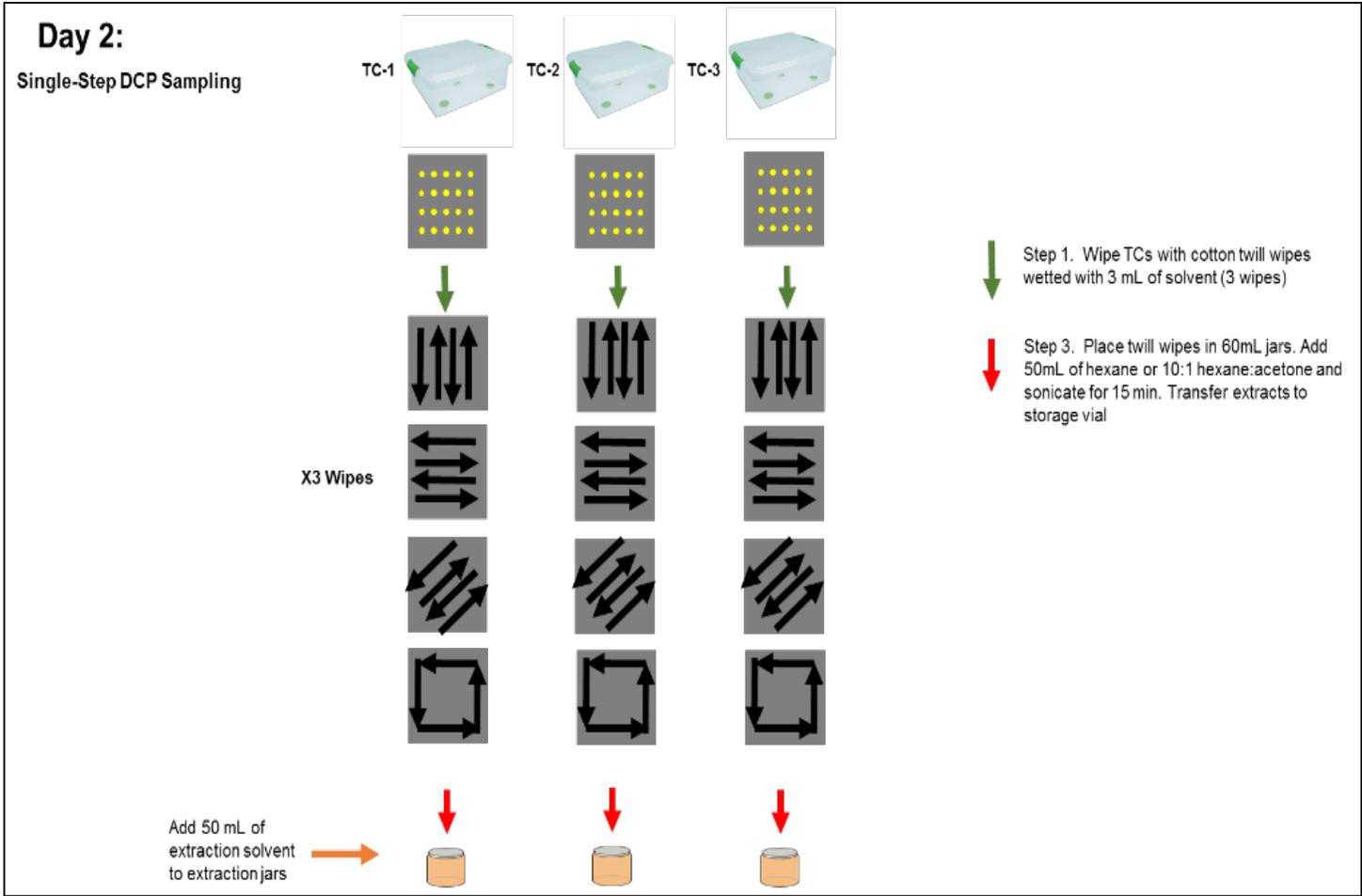


Figure 4-4. Day 2 experimental design and sample flow for single-step DCPs: surface sampling of TCs.

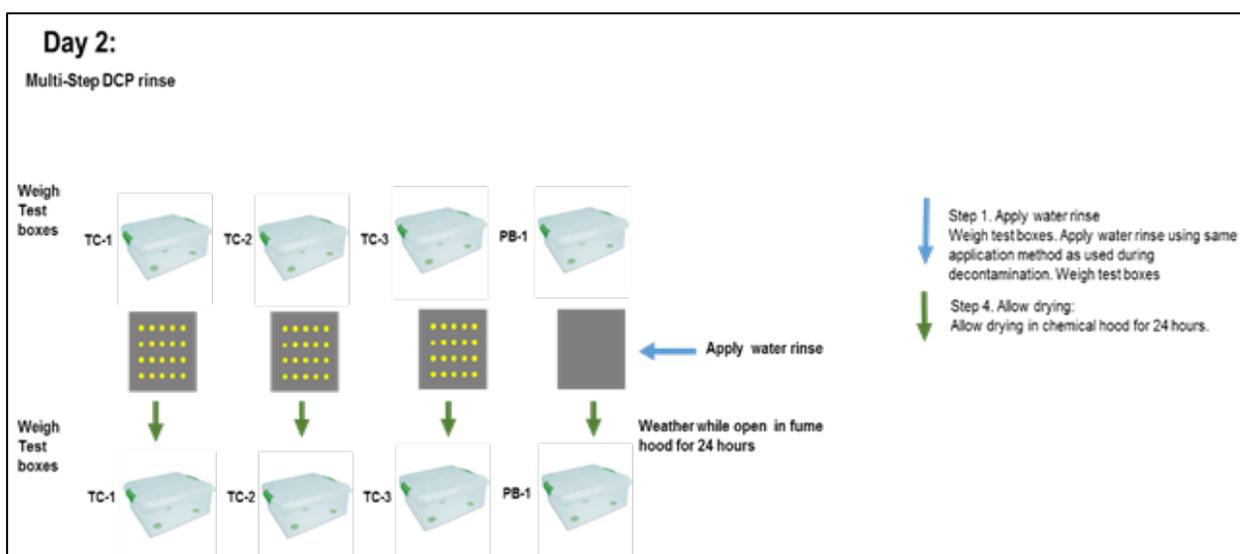


Figure 4-5. Day 2 experimental design and sample flow for multistep DCPs: application of water rinse.

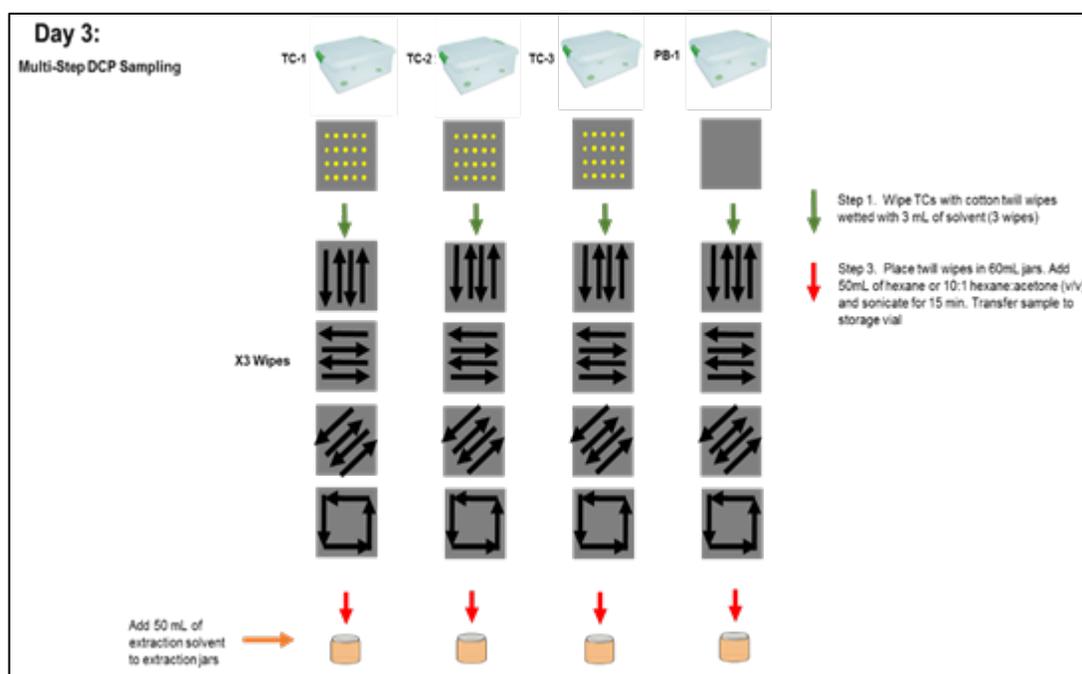


Figure 4-6. Day 3 experimental design and sample flow for multistep DCPs: surface sampling and extractions of TCs and PB.

## 4.2. Surface Sampling and Extraction Methods

This section summarizes types of wiping media, wetting solvents, and amount of wetting solvents for all material-chemical combinations that were used for collection of pre- and post-decontamination surface wipe samples, including PBs. The wipe sampling and extraction procedure was previously evaluated for malathion and carbaryl. The procedure was recently evaluated for concentrations that are typically seen in pesticide misuse applications [7], where the wetting solvent and its volume were optimized for high concentrations of malathion and carbaryl (4 and 24 mg, respectively, per 12 by 12-in test area). Wipe sampling methods were optimized prior to testing as described in Section 3.5.1. Table 4-1

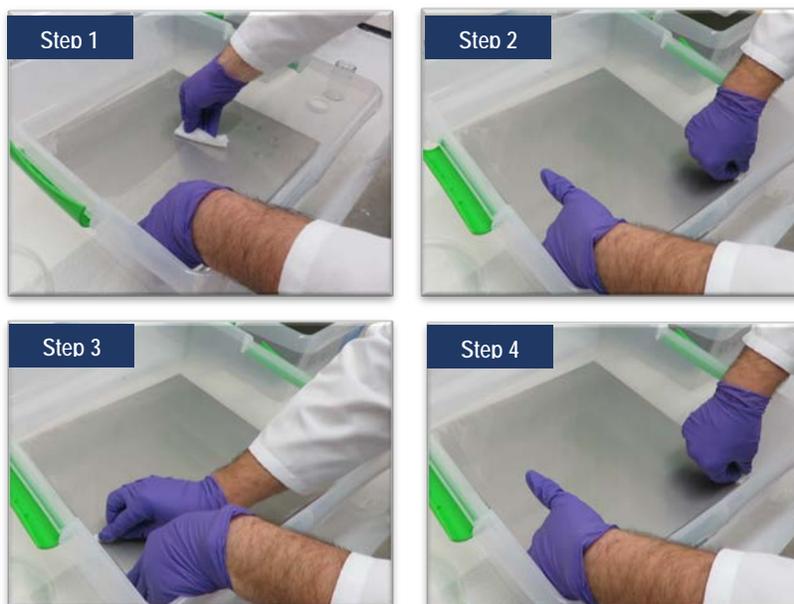
summarizes types of wiping media, wetting solvents, and amount of wetting solvent for all material-pesticide combinations.

**Table 4-1. Wiping media, wetting solvent, and wetting solvent volume for surface sampling**

Chemical/Surface	Wiping Medium	Number of Wipes	Wetting Solvent	Wetting Solvent Volume*
Malathion/stainless steel	Cotton twill wipe	3	Isopropyl alcohol	3 mL
Malathion/painted drywall	Cotton twill wipe	3	Isopropyl alcohol	3 mL
Carbaryl/stainless steel	Cotton twill wipe	3	Isopropyl alcohol	3 mL
Carbaryl/painted drywall	Cotton twill wipe	3	Isopropyl alcohol	3 mL

*\*volume of wetting solvent per wipe*

Each wipe was deployed using a four-step process consisting of a series of horizontal (Step 1, Figure 4-7), vertical (Step 2, Figure 4-7), diagonal (Step 3, Figure 4-7) and perimeter (Step 4, Figure 4-7) wiping strokes, where the wipe was folded over after each step (with contaminated side always inward). The detailed procedure (presented in Appendix B) is based on an internal miscellaneous operating procedure for wet wipe sampling of coupons. Figure 4-7 shows examples of wipe sampling on a horizontal reference material (collection of first wipe out of a total of three used for sampling shown).



**Figure 4-7. Example of surface wipe sampling of stainless steel in horizontal orientation.**

After completion of sampling, three wipes resulting from wiping each coupon were placed in a pre-cleaned 60- or 100-mL wide-mouth extraction jar with polytetrafluoroethylene (PTFE)-lined lids, for composite extraction. Each jar received 50 mL of hexane (Optima™, HPLC/spectrophotometry, gas chromatography/mass spectrometry (GC/MS) and pesticide residue analysis grade, Fisher Chemical, product # H 303-4 UN1208), was capped and was transferred to the sonicator. Note that the hexane term

refers to the mixture, as purchased, of *n*-hexane (45-60%), hexane (isomers) (15-40%) and cyclohexane (3%). Wipe samples were extracted via sonication for 15 minutes. After extraction was completed, a 15-mL aliquot of the extract was transferred to a 20-mL glass vial and refrigerated at  $4\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$  until further processing. The remainder of the sample extract was managed as laboratory waste. Sample preparation for instrumental analysis is described in Section 4.4.

### 4.3. Liquid and Solid Waste Sampling and Extraction Procedures

This section summarizes the liquid waste (runoff) and solid waste (expended sponges, cloths and paint rollers) sampling, and procedures used during tests for chemical uptake by cleaning media and transfer to liquid effluents (described in Section 3.5.3).

Target chemicals from the liquid waste samples (generated in simulated DCP3, DCP4 and DCP5 procedures in which water was used instead of decontaminants; test procedure described in Section 3.5.3) were extracted using a simplified liquid-liquid extraction procedure following the modified extraction procedure described in EPA Method 3571 (Extraction of Solid and Aqueous Samples for Chemical Agents) [9]. The method performance for liquid-liquid extraction of water samples containing malathion was optimized under other ongoing research efforts. A detailed summary of the method is given in Appendix C. Good recoveries were observed for low concentrations ( $0.05\text{ }\mu\text{g/mL}$ ) of malathion from non-preserved and preserved (with L-ascorbic acid, ethylenediaminetetraacetic acid) and non-pH-adjusted, and pH-adjusted (with trisodium salt of potassium dihydrogen citrate) water-waste matrix spike samples ( $100\% \pm 15\%$  SD and  $125\% \pm 3.9\%$  SD, respectively;  $n=3$  for treated and non-treated samples, respectively).

The above extraction method was checked for carbaryl recovery using DI water samples spiked with carbaryl at  $0.2\text{ }\mu\text{g/mL}$ . Carbaryl degrades rapidly at  $\text{pH} > 7$ , with a half-life of approximately 10-17 days at  $\text{pH}$  of 7 down to three hours for  $\text{pH}$  of 9 at  $25\text{ }^{\circ}\text{C}$  [10]. If the aqueous samples containing carbaryl were at  $\text{pH} > 7$ , they should be acidified to  $\text{pH}$  4–5 with  $0.1\text{ N}$  chloroacetic acid [11]. In this study, the uptake by cleaning media and transfer to liquid effluent experiments were performed using DI water ( $\text{pH}$  less than 7); there were no concerns about accelerated degradation of carbaryl. The recovery of carbaryl from (non-pH-adjusted) control samples was  $109\% \pm 1.7\%$  SD ( $n=3$ ). During testing, the  $\text{pH}$  of the liquid waste samples collected was checked immediately after liquid waste samples were collected (recorded  $\text{pH}$  range was 4.0-4.5, depending on the type of cleaning procedure/simulated DCP).

After determination of waste volume for each type of runoff collected (DCP3, DCP4 and DCP5), liquid waste samples were transferred to a clean extraction vial, and an equal volume of hexane was added to each sample (1:1 v/v liquid waste:hexane). Each sample was manually shaken for one minute. After the aqueous and hexane layer separated, the entire hexane layer was carefully collected using a Pasteur pipette and placed into a 15 mL test tube with graduated markings. The total extract volume was recorded. The simulated liquid waste extracts did undergo dilution prior to analysis. One mL of hexane extract was transferred into a 1.8 mL pre-labeled gas chromatographic (GC) amber glass screw-top vial. Samples were refrigerated after preparation and remained refrigerated until prior to shipment to the subcontracting laboratory for analysis. In addition, a 10 mL aliquot of the remaining extract was transferred to a 12 mL vial and archived under the same conditions at  $4\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ .

Solid waste samples (expended sponges, cleaning cloths and paint rollers) were extracted using a modified procedure that was optimized for extraction of cotton wipes from sampling (Section 4.2). The modifications include the following:

1. Extraction solvent volume was increased tenfold to perform large volume extraction, with 1000 mL beakers used instead of small extraction jars. Each beaker was filled with 500 mL of hexane for extraction of malathion, or hexane:acetone (10:1, v/v) for extraction of carbaryl.

2. Due to the large volume of samples, the extraction time was extended to 30 minutes. The level of the extraction solvent was marked using a permanent marker. The beakers were covered with aluminum foil (Heavy Duty Aluminum Foil, Food Service Foil 627; Reynolds Consumer Products, Lake Forest, IL, USA). The temperature of the water in the sonic bath was monitored and noted in the laboratory notebook every five minutes, with some heating of the water bath observed (highest recorded temperature after 30 minutes of sonication was 34.6 °C from the initial temperature of 20.7 °C at the beginning of extraction). The resulting losses in extraction solvent following sonication were compensated by adding solvent to return to the same marked level. No losses were more than 10% for the large-volume extraction procedure. The evaporation-related losses of pesticides were not systematically studied or monitored (e.g., by use of labeled pre-extraction surrogates spiked into samples prior to extractions), but were considered to be negligible due to the minimal evaporation rate and low volatility of the target pesticides.

Immediately after extraction was completed, the entire extract was quantitatively poured into another clean beaker. After the aqueous layer separated from the organic layer, samples were prepared for analysis. Due to the high volume of extraction solvent, most of the samples did not undergo dilution prior to analysis. Only a small subset of samples, those analyzed for carbaryl by the EPA OSL (Section 4.5), were diluted in hexane to concentrations that aligned with the dynamic GC calibration range (100-5000 ng/mL) and spiked with Internal Standard (IS)/surrogate mix. For non-diluted samples, one mL of hexane extract was taken from each secondary beaker and transferred into a 1.8-mL pre-labeled amber glass screw top GC vial and prepared for shipment to the subcontracting laboratory for analysis. Samples were then refrigerated prior to shipment to the subcontracting laboratory for analysis. In addition, a 10 mL aliquot of the remaining extract was transferred to a 12 mL vial and archived at 4 °C ± 2 °C.

#### 4.4. Preparation of Samples for Analysis

Extracts generated from extraction of wipes (Section 4.2) and liquid and solid wastes (Section 4.3) were prepared for analysis in 1.8-mL amber glass GC vials. Depending on the type of sample, extracts underwent up to 20-fold dilution. An aliquot of raw extract was drawn using an appropriate size micropipette and added to a GC vial filled with a premeasured amount of hexane (e.g., 50 microliters (µL) of sample and 950 µL of hexane, for a 20-fold dilution). The control spike samples were also diluted up to 20-fold. Other extracts (PCs from non-reference (painted drywall) material, all decontaminated TCs, blanks, and liquid waste extracts) were submitted to the subcontracting laboratory as is. A 1000-µL aliquot of sample was drawn from each extract using an electronic pipette and added to the GC vial. If analytical results were outside calibration range, the analytical laboratory performed necessary dilutions and reported dilution factors along with quality control (QC) data. The samples were refrigerated at 4 °C ± 2 °C or below prior to shipment. All shipments were accompanied by the chain of custody (COC) form and were inspected by the analytical laboratory upon receipt.

#### 4.5. Instrumental Analysis

Instrumental analyses were performed using modified National Institute for Occupational Safety and Health (NIOSH) Method 5600 [12] by an accredited subcontracting laboratory, EMSL Analytical, Inc.

(Cinnaminson, NJ, USA). Sample extracts were analyzed by means of GC/MS. Malathion was detected using ions of mass 93, 125 and 173 (quantitation with ion of mass 173). Carbaryl was detected using ions of mass 115 and 144; additionally, the ion of mass 144 of a positively identified thermo degradation product, 1-naphthalenol, was also reported (quantitation with combined response for parent compound and degradation product). The quantitation of carbaryl by the EPA OSL was also performed by GC/MS but under different instrumental conditions. The EPA OSL did not observe thermo degradation of carbaryl/formation of 1-naphthalenol. The quantitation of carbaryl was done using isotope dilution, with labeled carbaryl-<sup>13</sup>C<sub>6</sub> (CLM-4682-1.2, Cambridge Isotope Laboratories, Inc., Tewksbury, MA, USA) as internal standard and phenanthrene-D<sub>10</sub> as a surrogate compound (ERS-020-1.2ML, Internal Standards Mixture, Sigma-Aldrich Co. LLC, St. Louis, MO, USA). The instrumental parameters and conditions for GC/MS analyses for both analytes are given in Tables 4-2 through 4-4. Analysis by the EPA OSL was limited to carbaryl samples associated with the chemical uptake tests described in Section 3.5.3.

**Table 4-2. Instrumental parameters and conditions for GC/MS analyses of malathion (EMSL Analytical, Inc.)**

Parameter	Description/Conditions
Instrument	Agilent 6890 Gas Chromatograph equipped with Agilent 5973 Mass Selective Detector (Agilent Technologies, Santa Clara, CA, USA)
Autosampler	Agilent 7683 Automatic Sampler (Agilent Technologies, Santa Clara, CA, USA)
Column	Rtx®-5Sil MS w/5 m Integra-Guard® column, 30 m × 0.25 mm I.D., 0.25 µm film thickness; part no. 13623-124 (Restek Corporation, Bellefonte, PA, USA)
GC column program	100 °C initial temperature, hold 0 min, 15 °C/min to 250 °C, hold 5 min
Carrier gas flow rate	1.0 mL/min
Injection volume/type	1.0 µL/splitless
Inlet temperature	250 °C
MS source temperature	230 °C
MS transfer line	270 °C

**Table 4-3. Instrumental parameters and conditions for GC/MS analyses of carbaryl (EMSL Analytical, Inc.)**

Parameter	Description/Conditions
Instrument	Agilent 6890 Gas Chromatograph equipped with Agilent 5973 Mass Selective Detector (Agilent Technologies, Santa Clara, CA, USA)
Autosampler	Agilent 7683 Automatic Sampler (Agilent Technologies, Santa Clara, CA, USA)
Column	Rtx®-MS5 column, 30 m × 0.32 mm I.D., 0.50 µm df; part no. 13439 (Restek Corporation, Bellefonte, PA, USA)
GC column program	100 °C initial temperature, hold 0 min, 15 °C/min to 250 °C, hold 5 min
Carrier gas flow rate	1.0 mL/min
Injection volume/type	1.0 µL/splitless
Inlet temperature	225 °C
MS source temperature	230 °C
MS transfer line	270 °C

**Table 4-4. Instrumental parameters and conditions for GC/MS analyses of carbaryl (EPA OSL)**

Parameter	Description/Conditions
Instrument	Thermo Trace 1300 Gas Chromatograph GC ISQ™ Mass Spectrometer (Thermo Fisher Scientific, Inc., Waltham, MA, USA)
Autosampler	AS/AI 1310 Autosampler (Thermo Fisher Scientific, Inc., Waltham, MA, USA)
Column	DB-5, 20 m × 0.25 mm I.D., 0.25 µm df; part no. 13439 (Agilent, Santa Clara, CA, USA)
GC column program	80 °C initial temperature, 20 °C/min to 150 °C, 4 °C/min to 190 °C, 30 °C/min to 300 °C, final hold 2 min
Carrier gas flow rate	1.3 mL/min
Injection volume/type	1.0 µL/splitless
Inlet temperature	150 °C
MS source temperature	200 °C
MS transfer line	200 °C

For EMSL Analytical, Inc., the calibration range of 1-100 µg/mL for both analytes (seven-point calibration curve; 1-10-20-40-60-80-100 µg/mL) was used for initial calibration, with reporting limit verification (RLV) and initial calibration verification (ICV) analyses performed at lowest and mid-calibration level, respectively, prior to each analytical run. Due to instabilities in response of the 1 µg/mL carbaryl standard, this standard was excluded from calibration and average response factor calculations (six-point curve was run prior to analysis, and quantitation was performed using two five-point curves depending on sample concentration: 10-20-40-60-80 ug/mL (low-concentration curve, used for analysis of decontaminated samples and blanks) and 20-40-60-80-100 ug/mL (high-concentration curve used for analysis of non-decontaminated samples and control spikes at 100% target concentration). Additionally, analysis of the laboratory control sample (LCS) and the laboratory control sample duplicate (LCSD) was performed prior to each batch of samples. A continuous calibration standard at concentration mid-level was analyzed every ten samples, with a calibration end check performed at the end of each analytical run. Additional QC samples included duplicate injections of test samples and analysis of laboratory blanks. Samples with results below

the lowest calibration point (i.e., 1 µg/mL) were reported as less than the limit of quantitation (<LOQ). Acceptance criteria for QC checks are listed in Table 4-5, below.

**Table 4-5. QC checks for instrumental analyses performed by subcontracting laboratory.**

QC check	Acceptance limits
Initial Calibration (ICAL)	7-point initial calibration prior to analysis*
Reporting Limit Verification at lowest point concentration (RLV)	60-140%
Initial calibration verification at midpoint concentration (ICV mid)	80-120%
Laboratory Control Sample at midpoint concentration (LCS)	70-130%
Laboratory Control Sample Duplicate at midpoint concentration (LCS)	<25% RPD
Continuous calibration (CC) at midpoint concentration	80-120%
End check of calibration (EC) at midpoint concentration	80-120%
Duplicate injections (DUP)	<25% RPD
Laboratory (solvent) blank (BL)	< LOQ
<i>*Recalibrate when continuous calibration fails acceptance criteria and/or after system maintenance; RPD- relative percent difference</i>	

Prior to testing, an initial laboratory proficiency evaluation was performed. Accuracy and precision were determined by analysis of multiple measurements of control spike solutions at concentrations corresponding to 100% and 10% of chemical amount applied to test materials. (n = 3 to 5 for each concentration level; single analytical run).

Two sets of control spike samples were generated by spiking the carbaryl or malathion chemical solution used during the testing directly into the extraction solvent (hexane). All control spikes were sonicated for 10 minutes and then diluted as needed per Section 4.4. Each control spike set was accompanied by one solvent blank sample (1 mL of hexane used for extraction and preparation of samples for analysis). These control spike experiments were used as independent verifications of the results obtained from the external chemical analysis laboratory. The initial and continuing laboratory proficiency results are listed in Table 4-6.

**Table 4-6. The initial and continuing laboratory proficiency results**

Target Chemical	Spike Control A 100% Target Concentration, No Coupon <sup>*</sup> ; n=5	Spike Control B 10% Target Concentration, No Coupon <sup>*</sup> ;n=5	Solvent Blank
	Accuracy and Precision		
<b>EMSL Analytical, Inc.</b>			
Malathion (initial) <sup>a</sup>	95.0%±2.5% SD; RSD=2.6%	79.5%±3.1% SD; RSD=3.9%	<LOQ
Malathion (continuing <sup>b</sup> )	115%±20% SD; RSD=17%	Not prepared <sup>c</sup>	<LOQ
Carbaryl (initial <sup>a</sup> )	107.7%±6.3% SD; RSD=5.9%	91.3%±8.1% SD; RSD=8.8%	<LOQ
Carbaryl (continuing <sup>b</sup> )	69%±16% SD; RSD=24%	Not prepared <sup>c</sup>	<LOQ
<b>EPA OSL</b>			
Carbaryl (initial <sup>d</sup> )	109.2%±1.7% SD; RSD=1.5%	Not prepared	<LOQ
<sup>a</sup> Direct spike into hexane; QC samples prepared for initial laboratory proficiency testing. <sup>b</sup> Direct spike into hexane; QC samples prepared for each analytical batch from core-matrix decontamination test. As per Quality Assurance Project Plan (QAPP), only samples at 100% concentration level were prepared for ongoing checks of laboratory proficiency. <sup>*</sup> Test-specific (transfer to liquid effluents) control samples prepared in water and liquid-liquid extracted with hexane.			

The acceptance criteria for the initial laboratory proficiency tests were established in the QAPP and were 80–120% for accuracy (as recovery compared to theoretical concentration) and < 30% RSD precision for each concentration level for replicate analysis, n = 5 for each concentration target. The ongoing analysis of control spike samples, delivered to the subcontracted analytical laboratory along with each analytical batch was within the above acceptance criteria. Slightly lower recoveries of ongoing laboratory proficiency control spike samples for carbaryl may have been due to longer holding times for analytical batches from the decontamination tests, which were analyzed with longer (six-week) turnaround time (TAT). Other samples were analyzed with 2 to 4 weeks TAT. The results were accepted based on good recovery of test-specific PC samples (Table 3-4) and high accuracy and precision of the analytical measurement reported for control samples prepared by the subcontracting laboratory (accuracy for LCS/LCSD at mid-point concentration was 80% and 82% of true value, respectively; RSD <1%; data not shown).

## 4.6. Data Reduction Procedures

### 4.6.1. Chemical Concentration in Extract Calculations

The GC/MS concentration results ( $\mu\text{g/mL}$ ) were converted to total mass of chemical per sample (mg per sample) by multiplying by the extraction solvent volume and dilution factor (if applicable):

$$M_s = C_s \times V_E \times D_F \times 1000 \quad (1)$$

where:

$M_s$  = mass of chemical in sample (mg)

$C_s$  = concentration ( $\mu\text{g/mL}$ ) from an individual replicate sample

$V_E$  = extraction solvent volume (mL)

$D_F$  = sample dilution factor prior to analysis (if any)

The percent recovery of the chemical from the QC samples (e.g., control spikes) was calculated against theoretical chemical amount spiked into solution:

$$\%R_{QC} = C_{QC} / (V_{SP} \times S_C / V_T / D_F) \times 100\% \quad (2)$$

where:

$\%R_{QC}$  = percent recovery for an individual QC sample (versus theoretical)

$C_{QC}$  = concentration ( $\mu\text{g/mL}$ ) from an individual replicate QC sample

$V_{SP}$  = volume of spike (mL)

$S_C$  = concentration of chemical in spiking solution (8 mg/mL for carbaryl or 6 mg/mL for malathion)

$V_T$  = total sample volume (mL)

$D_F$  = sample dilution factor prior to analysis (if any)

The chemical mass ( $M_s$ ) results used for decontamination efficacy calculations were not adjusted for QC sample recovery ( $\%R_{QC}$ ).

### 4.6.2. Decontamination Cleanup Efficacy Calculations

The decontamination cleanup efficacy was calculated using the mean of the chemical mass recovered from the replicate TC and the mean chemical mass recovered from the associated set of PCs.

$$DE = (1 - \bar{x}_{TC} / \bar{x}_{PC_n}) \times 100\% \quad (3)$$

where:

DE = mean decontamination efficacy (%)

$\bar{x}_{TC_n}$  = mean of chemical amount remaining on replicate TC (decontaminated) coupons (mg)

$\bar{x}_{PC_n}$  = mean of chemical amount remaining on replicate PC (non-decontaminated) coupons (mg)

The mean decontamination efficacy along with the standard deviation was calculated as cumulative decontamination efficacy (or resulting from application of all three procedural steps for each test). 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 TCs and on the PCs. If the average mass of remaining agent on the TC was found to be below the LOQ, the efficacy was calculated using the LOQ value and reported as "greater than" this calculated value.

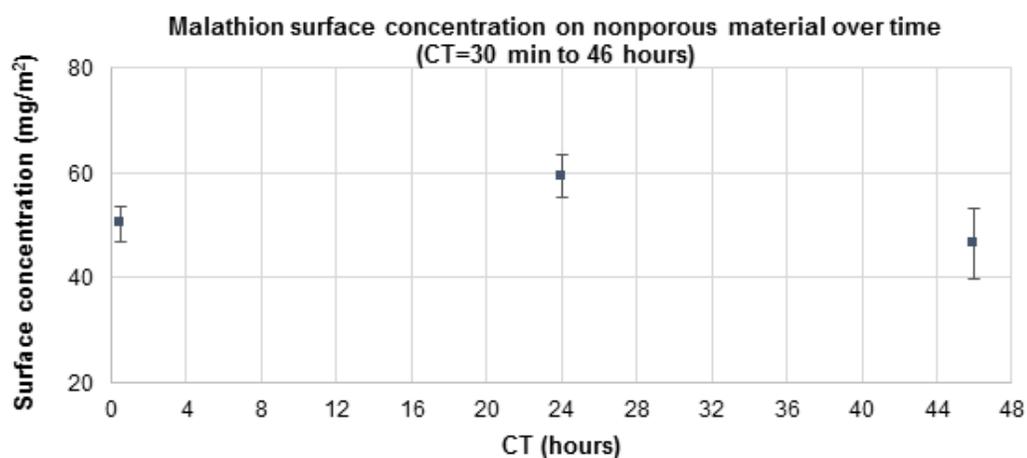
## 5.0. Results

### 5.1. Persistence and Uptake of Chemicals by Nonporous and Semi-Porous Materials.

The results for the persistence and uptake of chemicals by nonporous and semi-porous materials. (experimental approach described in Section 3.5.2) for three chemical-surface-contact times tested ( $CT_{min} = 30 \text{ min}$ ,  $CT_{mid} = 24 \text{ h}$ ,  $CT_{max} = 46 \text{ h}$ ) are given in Table 5-1, below.

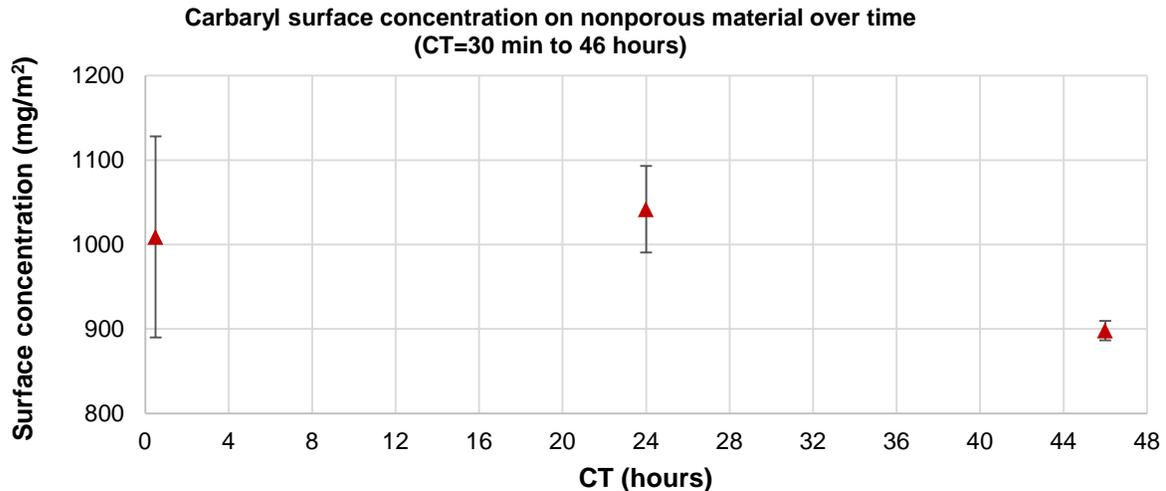
**Table 5-1. Malathion and carbaryl surface concentrations on stainless steel and painted drywall over three contact times tested**

Contact Times	Stainless Steel			Painted Drywall		
	Mean	$\pm$ SD	%RSD	Mean	$\pm$ SD	%RSD
	mg/m <sup>2</sup>			mg/m <sup>2</sup>		
<b>Malathion</b>						
$CT_{min}=30 \text{ min}$	50.2	3.5	7.0%	4.5	1.3	28%
$CT_{mid}=24 \text{ h}$	59.4	4.0	6.7%	3.4	0.50	15%
$CT_{max}=46 \text{ h}$	46.5	6.6	14%	4.1	0.47	11%
<b>Carbaryl</b>						
$CT_{min}=30 \text{ min}$	1010	120	12%	117	21.1	18%
$CT_{mid}=24 \text{ h}$	1040	51.4	4.9%	125	37.3	30%
$CT_{max}=46 \text{ h}$	898	11.4	1.3%	98.4	7.7	7.9%



**Figure 5-1. Malathion surface concentration over time on nonporous material, stainless steel.**

No significant surface dissipation of target chemicals from nonporous materials was observed during 24 hours; the average concentration of malathion and carbaryl at  $CT_{mid}$  was actually slightly higher than chemical film surface concentration measured for  $CT_{min}$  subsets (Table 5-1). The decrease of malathion and carbaryl concentration observed between 24-h and 46-h contact times (Table 5-1, Figure 5-1) was, however, statistically significant ( $p < 0.05$  and  $p < 0.01$ , respectively).



**Figure 5-2. Carbaryl surface concentration over time on nonporous material, stainless steel.**

The low dissipation rates of target pesticides from reference stainless steel material, <20% reduction of chemical surface concentration for approximately two-day contact times are in line with pesticide stability on nonporous surfaces reported in other laboratory and field studies addressing short and long-term building surface persistence of semivolatile compounds with low vapor pressures [13]. The nerve agent VX, an OP compound structurally similar to malathion, was recently reported to be short-term stable on a stainless steel surface contaminated with chemical at a level of milligrams per square meter, with surface loadings reduced by average 11 and 24 percent at 24 and 72 hours post-contamination [14]. For comparison, vapor pressures, which are associated with the volatility of a chemical, of VX and malathion at 25 °C are 0.117 Pascals (Pa) and 0.0053 Pa, respectively [15]. Carbaryl has a lower volatility than malathion (0.00020 Pa) [15] and hence, is even less prone to evaporation-related losses; the literature-reported evaporation-related dissipation of the carbaryl (applied to a soil surface) was estimated to be less than 1% after 50 days post-application [16]. It should be emphasized that pesticide degradation in indoor environments is likely to be accelerated by moisture, sunlight and/or microorganisms, similar to the degradation in the outdoor environment [17]. None of these factors was contributing to pesticide breakdown on the clean and relatively inert surfaces used in this laboratory study.

Analysis of surface concentrations of target pesticides from a representative semi-porous material (painted drywall) was performed in parallel to testing of the nonporous stainless steel (results are in Table 5-1 and are summarized in Figures 5-3 and 5-4). The results indicated that uptake of target chemicals occurred rapidly, with only approximately ten percent of the chemical mass remaining on the surface after 30 minutes post-application (as compared to the equivalent reference material subset; Table 5-1). Over time, the surface-available fraction of the chemical remained relatively constant and did not show a statistically significant surface dissipation trend (Figures 5-3 and 5-4).

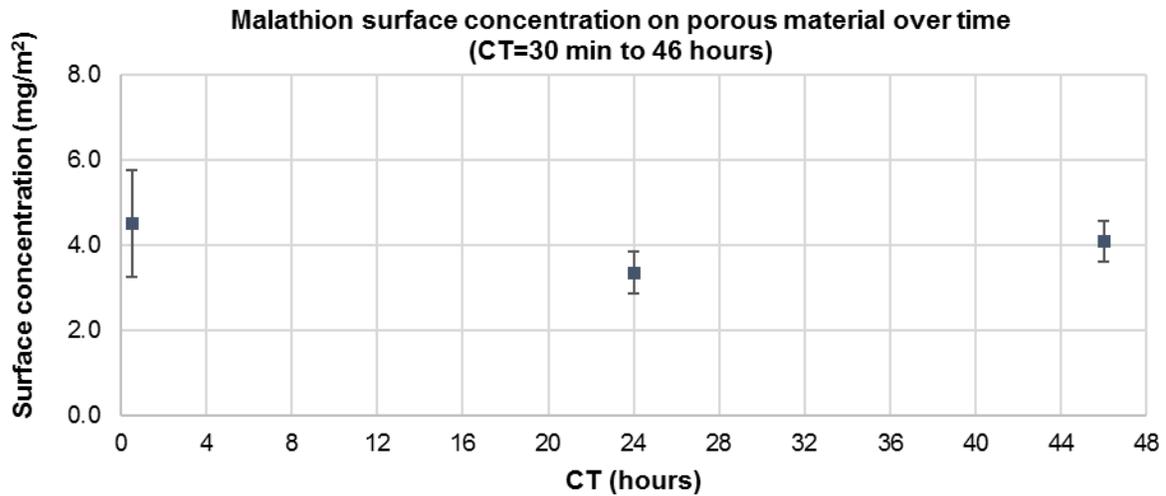


Figure 5-3. Malathion surface concentration over time on semi-porous material, painted drywall.

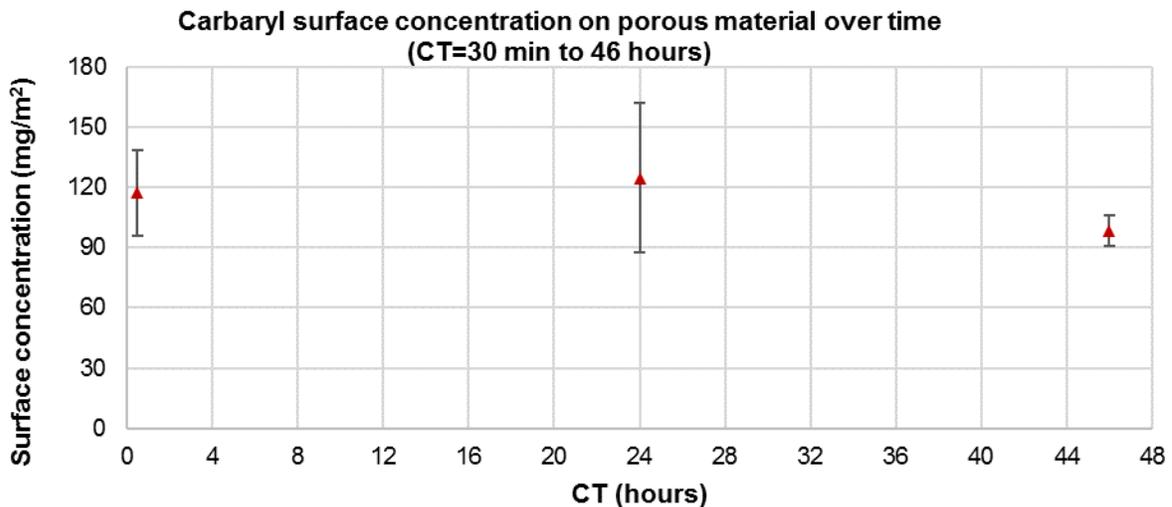


Figure 5-4. Carbaryl surface concentration over time on semi-porous material, painted drywall.

The rapid uptake of persistent organic chemicals is often reported in the context of chemical transport and fate prior to decontamination [2,14]. Since the goal of this study was optimization of decontamination procedures, the decontamination experiments for semi-porous materials were not performed, due to uncertainties in the mechanism of action of decontaminants against permeated chemical agents, including a potential reverse transport of contaminants to the material surface post-decontamination. Studies investigating optimization of decontamination procedures for neutralization of chemical agents absorbed into semi-porous building materials, including possible optimization of decontamination solution delivery methods to subsurface layers of semi-porous materials, are ongoing under a different research effort.

## 5.2. Surface Decontamination Efficacy

Test-specific results of residual surface contamination present on stainless steel before and after decontamination with single- and multistep DCPs, as well as procedure-and-chemical-specific percent decontamination efficacy (%DE; calculated per Section 4.6.2) results are given in Table 5-2 through 5-5. Figures 5-6 and 5-7 summarize overall %DE ( $\pm$ SD) for all DCPs used for cleanup and neutralization of both target chemicals on the reference material.

**Table 5-2. Single- and -multistep bleach decontamination test results for malathion on stainless steel**

Malathion with Bleach											
Cleaning media	Method	ID	Decontaminated Coupons			Positive Controls			Procedural Blank	Decontamination Efficacy	
			Mean	±SD	%RSD	Mean	SD	%RSD		mg/m <sup>2</sup>	%
			mg/m <sup>2</sup>			mg/m <sup>2</sup>					
Pressurized sprayer	Single-step	MA-BL-DCP1-PS-S	<0.54	NA	NA	192	24	13%	<0.54	>99.7	NA
Pressurized sprayer	Multistep	MA-BL-DCP1-PS-M	<0.54	NA	NA					>99.7	NA
Spray bottle	Single-step	MA-BL-DCP2-SB-S	<0.54	NA	NA				<0.54	>99.7	NA
Spray bottle	Multistep	MA-BL-DCP2-SB-M	<0.54	NA	NA					>99.7	NA
Cleaning cloth	Single-step	MA-BL-DCP3-RG-S	<0.54	NA	NA	160	11	7%	<0.54	>99.7	NA
Cleaning cloth	Multistep	MA-BL-DCP3-RG-M	<0.54	NA	NA					>99.7	NA
Cleaning sponge	Single-step	MA-BL-DCP4-SP-S	<0.54	NA	NA				<0.54	>99.7	NA
Cleaning sponge	Multistep	MA-BL-DCP4-SP-M	<0.54	NA	NA					>99.7	NA
Paint roller	Single-step	MA-BL-DCP5-PR-S	<0.54	NA	NA				<0.54	>99.7	NA
Paint roller	Multistep	MA-BL-DCP5-PR-M	<0.54	NA	NA					>99.7	NA

*MA-malathion; BL-bleach; DCP 1 to 5-Decontamination Cleanup Procedure 1 to 5; PS-pressurized sprayer; SB-spray bottle; RG-cotton cloth; SP- sponge; PR-paint roller; S-single-step procedure; M-multistep procedure; < or > values - calculated based on LOQ*

**Table 5-3. Single- and -multistep EasyDECON® DF200 decontamination test results for malathion on stainless steel**

Malathion with EasyDECON® DF200											
Cleaning Media	Method	ID	Decontaminated Coupons			Positive Controls			Procedural Blank	Decontamination Efficacy	
			Mean	±SD	%RSD	Mean	SD	%RSD		mg/m <sup>2</sup>	%
			mg/m <sup>2</sup>			mg/m <sup>2</sup>					
Pressurized sprayer	Single-step	MA-ED-DCP1-PS-S	<0.54	NA	NA	156	14	9.1%	<0.54	>99.7	NA
Pressurized sprayer	Multistep	MA-ED-DCP1-PS-M	<0.54	NA	NA					>99.7	NA
Spray bottle	Single-step	MA-ED-DCP2-SB-S	<0.54	NA	NA				<0.54	>99.7	NA
Spray bottle	Multistep	MA-ED-DCP2-SB-M	<0.54	NA	NA					>99.7	NA
Cleaning cloth	Single-step	MA-ED-DCP3-RG-S	<0.54	NA	NA	142	42	29%	<0.54	>99.7	NA
Cleaning cloth	Multistep	MA-ED-DCP3-RG-M	<0.54	NA	NA					>99.7	NA
Cleaning sponge	Single-step	MA-ED-DCP4-SP-S	<0.54	NA	NA				<0.54	>99.7	NA
Cleaning sponge	Multistep	MA-ED-DCP4-SP-M	<0.54	NA	NA					>99.7	NA
Paint roller	Single-step	MA-ED-DCP5-PR-S	<0.54	NA	NA	<0.54	>99.7	NA			
Paint roller	Multistep	MA-ED-DCP5-PR-M	<0.54	NA	NA		>99.7	NA			

*MA-malathion; ED-EasyDECON® DF200; DCP 1 to 5-Decontamination Cleanup Procedure 1 to 5; PS-pressurized sprayer; SB-spray bottle; RG-cotton cloth; SP- sponge; PR-paint roller; S-single-step procedure; M-multistep procedure; < or > values - calculated based on LOQ*

**Table 5-4. Single- and -multistep bleach decontamination test results for carbaryl on stainless steel**

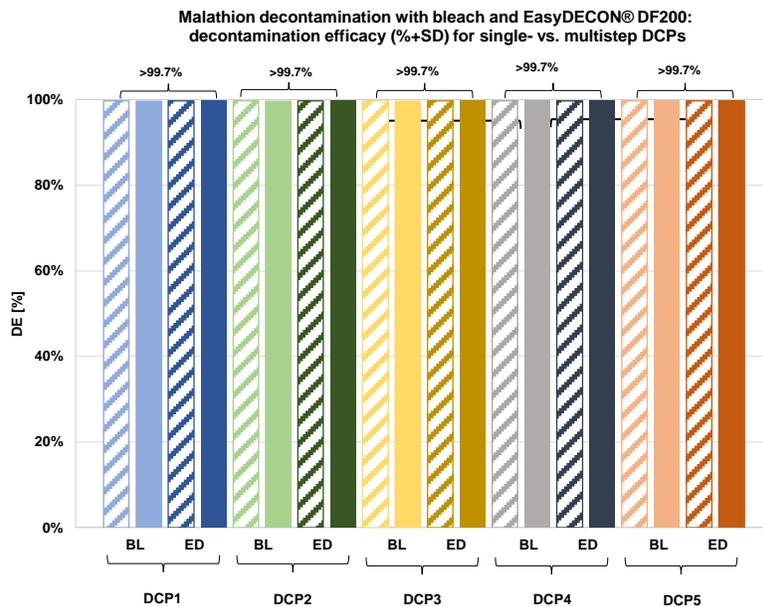
Carbaryl with Bleach											
Cleaning media	Method	ID	Decontaminated Coupons			Positive Controls			Procedural Blank	Decontamination Efficacy	
			Mean	±SD	%RSD	Mean	SD	%RSD		mg/m <sup>2</sup>	%
			mg/m <sup>2</sup>			mg/m <sup>2</sup>					
Pressurized sprayer	Single-step	CA-BL-DCP1-PS-S	59	12	21%	178	49	27%	<5.4	63%	19%
Pressurized sprayer	Multistep	CA-BL-DCP1-PS-M	43	8.8	20%					76%	21%
Spray bottle	Single-step	CA-BL-DCP2-SB-S	66	6.2	9.4%				<5.4	63%	18%
Spray bottle	Multistep	CA-BL-DCP2-SB-M	30	12	39%					83%	24%
Cleaning cloth	Single-step	CA-BL-DCP3-RG-S	52	11	21%	194	7.3	3.8%	<5.4	73%	6.3%
Cleaning cloth	Multistep	CA-BL-DCP3-RG-M	33	26	81%					83%	14%
Cleaning sponge	Single-step	CA-BL-DCP4-SP-S	44	27	61%				<5.4	77%	14%
Cleaning sponge	Multistep	CA-BL-DCP4-SP-M	9.1	3.8	41%					95%	4.1%
Paint roller	Single-step	CA-BL-DCP5-PR-S	47	9.1	19%				<5.4	76%	5.5%
Paint roller	Multistep	CA-BL-DCP5-PR-M	34	11	32%					82%	6.4%

*CA-carbaryl; BL-bleach; DCP 1 to 5-Decontamination Cleanup Procedure 1 to 5; PS-pressurized sprayer; SB-spray bottle; RG-cotton cloth; SP- sponge; PR-paint roller; S-single-step procedure; M-multistep procedure; < or > values - calculated based on LOQ*

**Table 5-5. Single- and -multistep EasyDECON® DF200 decontamination test results for carbaryl on stainless steel**

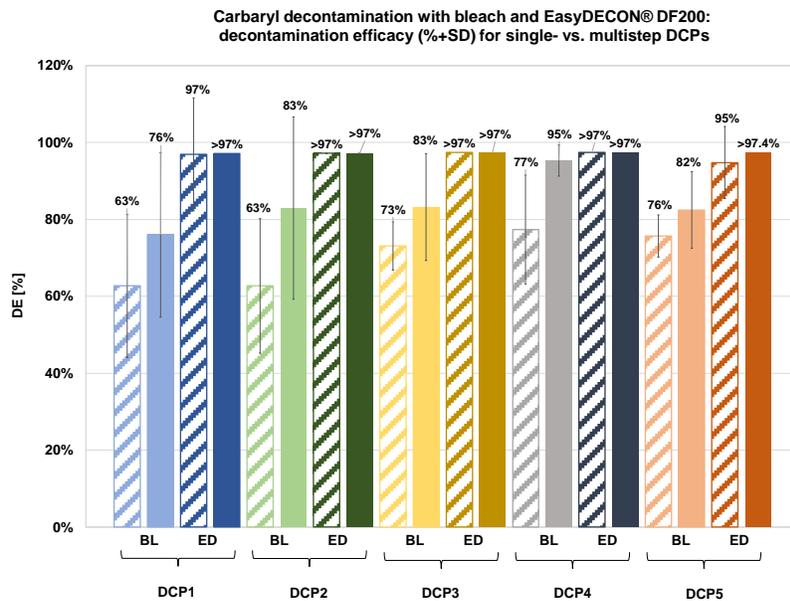
Carbaryl with EasyDECON® DF200											
Cleaning Media	Method	ID	Decontaminated Coupons			Positive Controls			Procedural Blank	Decontamination	
			Mean	±SD	%RSD	Mean	SD	%RSD		mg/m <sup>2</sup>	%
			mg/m <sup>2</sup>			mg/m <sup>2</sup>					
Pressurized sprayer	Single-step	CA-ED-DCP1-PS-S	5.9	0.54	9.1%	193	28	14.6%	<5.4	96.9%	14%
Pressurized sprayer	Multistep	CA-ED-DCP1-PS-M	<5.4	NA	NA					>97.2%	NA
Spray bottle	Single-step	CA-ED-DCP2-SB-S	<5.4	NA	NA					>97.2%	NA
Spray bottle	Multistep	CA-ED-DCP2-SB-M	<5.4	NA	NA					>97.2%	NA
Cleaning cloth	Single-step	CA-ED-DCP3-RG-S	<5.4	NA	NA	210	19	9.2%	<5.4	>97.4%	NA
Cleaning cloth	Multistep	CA-ED-DCP3-RG-M	<5.4	NA	NA					>97.4%	NA
Cleaning sponge	Single-step	CA-ED-DCP4-SP-S	<5.4	NA	NA					>97.4%	NA
Cleaning sponge	Multistep	CA-ED-DCP4-SP-M	<5.4	NA	NA					>97.4%	NA
Paint roller	Single-step	CA-ED-DCP5-PR-S	10.9	7.4	68%	<5.4	>97.4%	94.8%	9.4%		
Paint roller	Multistep	CA-ED-DCP5-PR-M	<5.4	NA	NA					>97.4%	NA

CA-carbaryl; ED-EasyDECON® DF200; DCP 1 to 5-Decontamination Cleanup Procedure 1 to 5; PS-pressurized sprayer; SB-spray bottle; RG-cotton cloth; SP- sponge; PR-paint roller; S-single-step procedure; M-multistep procedure; < or > values - calculated based on LOQ



Legend: BL-bleach; ED-EasyDECON® DF200; DCP 1 to 5-Decontamination Cleanup Procedure 1 to 5;  
Dashed columns-single-step procedure; Solid columns-multistep procedure; > - calculated based on LOQ

**Figure 5-5. Malathion decontamination efficacy (%±SD) for single- vs multistep DCPs**



Legend: BL-bleach; ED-EasyDECON® DF200; DCP 1 to 5-Decontamination Cleanup Procedure 1 to 5;  
Dashed columns-single-step procedure; Solid columns-multistep procedure; > - calculated based on LOQ

**Figure 5-6. Carbaryl decontamination efficacy (%±SD) for single- vs multistep DCPs**

Decontamination test results indicate that chemical surface films of malathion were prone to chemical neutralization with DE > 99.7% for both decontaminants and all types of DCPs deployed. This observation is in line with literature data on oxidation efficacy of OP pesticides, suggesting that OP pesticides undergo relatively rapid chemical oxidation and/or hydrolysis in the presence of various forms of aqueous chlorine (hypochlorous acid, HOCl; hypochlorite ion, OCl<sup>-</sup>) and hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) [18,19]. It should be emphasized that malathion, like many of the commercially available OP pesticides, is a lipophilic phosphorothionate (with one thione moiety (P=S) and three -OR groups attached to a phosphorus atom); its respective oxidized analogs are more polar, characterized by a phosphorus oxygen double bond (P=O), which actually makes the compounds more potent acetylcholinesterase (AChE) inhibitors [18]. These toxic oxidation by-products of OPs, or so-called oxon transformation products, were not analyzed in this study, but based on the literature data, formation of oxo-organophosphates (e.g., diazoxon, maloxon) from organothiophosphates (e.g., malathion) is higher for hypochlorite (NaOCl)- than for hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>)-induced reactions [19]. In addition to the theoretically higher potential for formation of oxons, bleach-based procedures deployed in this study have shown consistent - and very severe - material incompatibilities with stainless steel, causing irreversible damage to the treated surfaces. No material incompatibilities were observed for EasyDECON® DF200. The residue observed after drying out of EasyDECON® DF200 was most likely caused by the cationic surfactant (benzalkonium chloride) used in this formulation, and was easily removed by rinsing with water. Figure 5-7 shows the stainless steel material within an hour after the completion of the multistep decontamination treatment with bleach and EasyDECON® DF200 and rinse.



**Figure 5-7. Appearance of the material surface after multistep treatment with bleach (A) and EasyDecon® DF200 (B).**

Carbaryl had an average %DE ranging from 63% to > 97.4%, depending on type of DCP and decontaminant applied. The overall higher decontamination rates were observed for EasyDECON® DF200 (94.8% to >97.4%), compared to average 63% to 95% DE offered by (concentrated germicidal) bleach. This is in line with literature data on carbamates, and aromatic carbamates, like carbaryl, are not especially prone to oxidation by various chlorine-based oxidants at neutral and alkaline pH, especially at a low concentration of decontaminant [17]. Activated hydrogen peroxide formulas, however, are efficacious for oxidation of carbamates. Hydrogen peroxide in EasyDECON® DF200 is activated by addition of so-called booster - diacetin (glycerol diacetate). The O-bonded acetyl group of the activator reacts with strongly nucleophilic hydroperoxy anions (OOH<sup>-</sup>) to yield peroxygenated species; the peroxygenated species (O=O<sup>-</sup>) is a more efficient oxidizer than hydrogen peroxide alone [20]. The EasyDECON® DF200 decontaminant has an additional advantage in terms of neutralization of carbaryl that is relatively insoluble in water - the addition of so-called solubilizing agent: a quaternary ammonium compound (*n*-alkyl-C<sub>12-16</sub>-N,N-dimethyl-N-benzyl ammonium chloride) [20]. This cationic surfactant “wets-out” (water-insoluble) contamination by suspending

it in a micelle and enhances solubility and availability of carbaryl chemical film to the oxidizing action of activated hydrogen peroxide [19]. It is important to re-emphasize that carbaryl does undergo fast hydrolysis in aqueous alkaline solutions [10]. Both decontaminants in this study have an alkaline pH (Table 3-7) that should theoretically aid decontamination, especially for multistep procedures with no mechanical removal step (DCP1 and DCP2). Carbaryl decontamination tests showed the same type of bleach-stainless steel material incompatibilities as described above for malathion and were characterized by severe corrosion of the polished surface of the stainless steel material (as shown in the example of Figure 5-3).

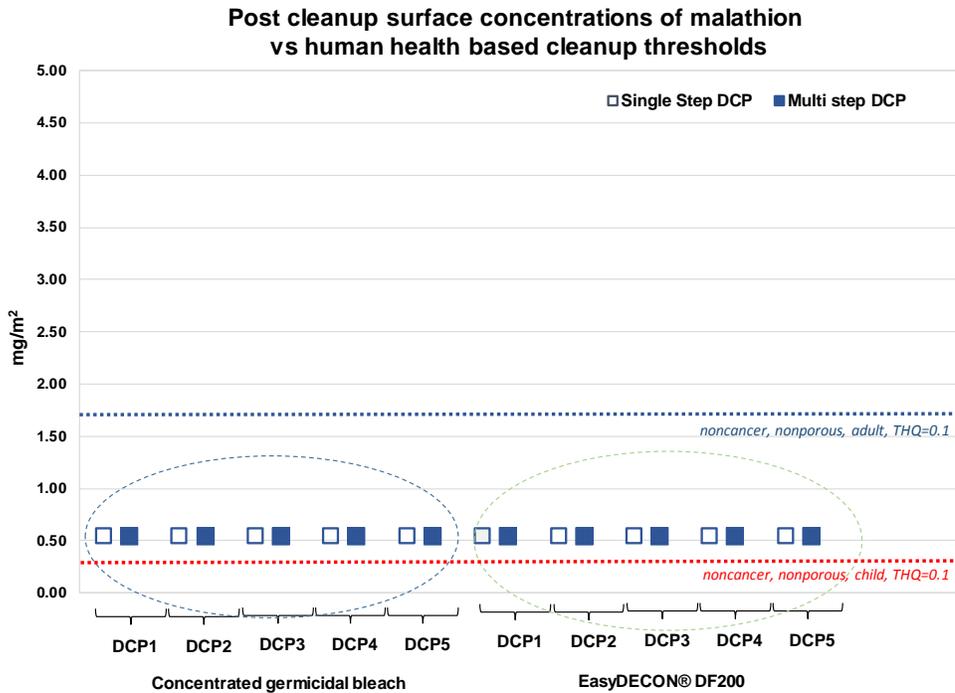
The experimental results demonstrated that, in the case of chemicals that are not very soluble in aqueous solutions, the multistep procedures offered better cleanup efficacy. The re-application step appears to be particularly important for DCPs that did not include the mechanical removal step, i.e., for spray-on DCP1 and DCP2. The re-application of decontaminant resulted in a statistically significant increase in carbaryl decontamination efficacy for non-pressurized spray applications (i.e., for multi- versus single-step DCP2,  $p < 0.01$ ). For methods with a mechanical removal step (DCP3, DCP4 and DCP5), the contribution of a mechanical removal step to overall decontamination was studied for each type of cleaning medium and indicated that mechanical cleaning is less vital to overall decontamination effectiveness than chemical neutralization; results are discussed in Section 5.4.

### 5.3. Residual Pesticides and Cleanup Thresholds

The measured DEs for the evaluated DCPs provide information on the reduction in chemical loading on a surface under controlled laboratory conditions. These DEs provide decision makers with information whether these approaches should be considered in a site specific incident. The other main consideration of any DCP is to decrease the chemical burden to levels that are considered safe for re-entry without specialized protective equipment and ultimately for re-occupation of a building. A direct comparison of residual surface concentrations in the laboratory experiment could be made against an actionable level. However, it does not have a real relevance to a field response clearance goal which would be site and situation specific. Here, the residual surface concentration levels were compared to surface cleanup goals solely as to identify whether a DCP would have been considered successful in reaching a clearance threshold value. It should not be construed that these DCPs will achieve such cleanup level as derived for an actual contamination situation. These cleanup level recommendations are not legally binding on any U.S. EPA program and should be interpreted as suggestions that program offices or individual exposure assessors can consider and modify as needed. Currently, there are no EPA regulatory values for surface cleanup goals. According to the National Contingency Plan (NCP) [21], risk-based cleanup goals are determined on a site- and situation-specific basis. In this study, human health-based screening levels were used for calculation of risk-based cleanup thresholds. The method referenced here for the derivation of risk-based cleanup goals is based upon the information presented in the World Trade Center Indoor Environment Assessment: Selecting Contaminants of Potential Concern and Setting Health-Based Benchmarks [3].

The noncancer risk-based cleanup thresholds were calculated based on various toxicological data, including hazard reference doses for oral and dermal exposures for a child (body weight 15 kilos) and adults (body weight 80 kilos), at assumed 365-day residential exposure scenarios [4] and a conservative target hazard quotient (THQ) of 0.1 [4]. Further details on the risk-based surface cleanup threshold calculations are provided in Appendix A. The calculated surface cleanup thresholds (for semi-porous and nonporous materials) are given in Table A-9 in Appendix A.

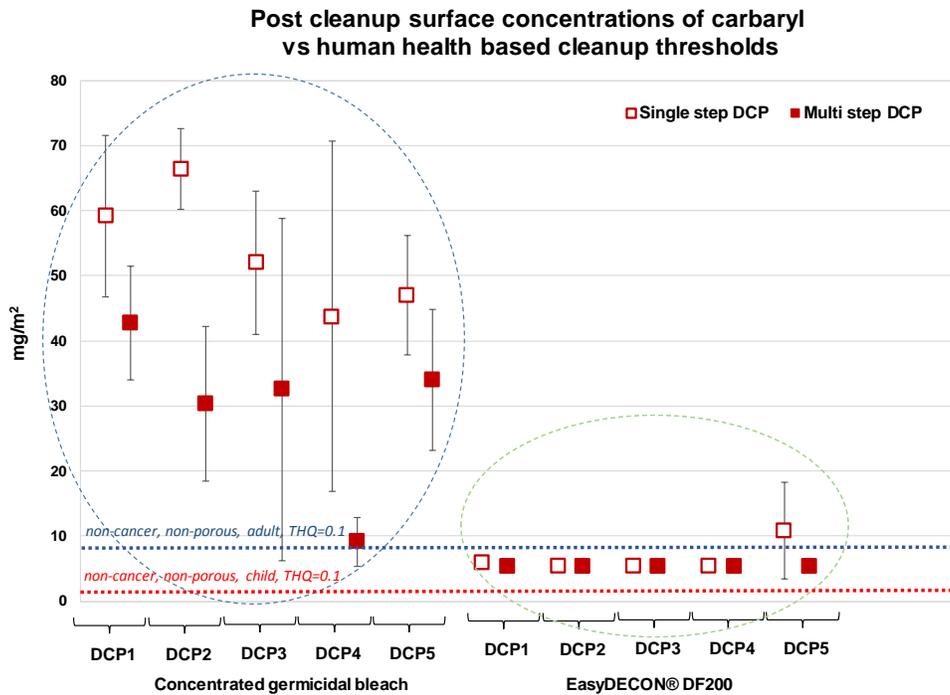
Figures 5-8 and 5-9 show post-cleanup surface concentrations of malathion and carbaryl against calculated human health risk-based cleanup thresholds. For malathion, both decontaminants showed reduction of malathion on the surface below the calculated cleanup threshold for adults, independent of the DCP used (Figure 5-8). The calculated cleanup threshold for a child was lower than the quantification limit in this study and could therefore not be met. As mentioned before, the chemical analysis of the wipes did not assess whether either decontamination process led to the formation of the toxic byproduct malaoxon which was beyond the scope of this study. A separate cleanup threshold calculation would be required to assess the impact of a detectable amount of such toxic decontamination byproduct.



**Figure 5-8. Post cleanup surface concentrations of malathion versus calculated human health risk-based cleanup thresholds – THQ: target hazard quotient [4]**

In the case of carbaryl, only the EasyDECON® DF200 formulation offered cleanup efficacy allowing reduction of the surface chemical burden below the health risk-based threshold for adults (Figure 5-9); only 13% of the test samples treated with the various EasyDECON® DF200-based DCPs showed detectable levels ( $>5.4 \text{ mg/m}^2$ ) of carbaryl, with one out of thirty total test samples reporting approximately two times higher than the noncancer child human health risk-based cleanup threshold for nonporous surfaces (Table A-9). Here again, the calculated carbaryl cleanup threshold for a child was lower than the quantification limit in this study and could therefore not be met. The only bleach-based DCP that reduced carbaryl levels close the human-health (adult) risk-based cleanup threshold for nonporous surfaces was the multistep DCP4. This procedure used a large industrial grade cleaning sponge that allowed the highest surface loading of contaminant among tested procedures, on average, approximately 25 mL of bleach per coupon test area of approximately  $929 \text{ cm}^2$ , or the equivalent of almost 270 mL of bleach per  $\text{m}^2$ . Other bleach-based DCPs, with average computed DEs ranging from 63% to 83%, reduced the carbaryl surface levels to average

concentrations of 30 to 66 mg/m<sup>2</sup>, which are well above the calculated noncancer adult human health risk-based cleanup threshold for nonporous surfaces (8.5 mg/m<sup>2</sup>). Consequently, additional decontamination approaches would be required if bleach was selected to cleanup this nonporous material.



**Figure 5-9. Post cleanup surface concentrations of carbaryl versus calculated human health risk-based cleanup thresholds – THQ: target hazard quotient [4]**

#### 5.4. Transfer of Pesticide to Cleaning Media and Liquid Waste

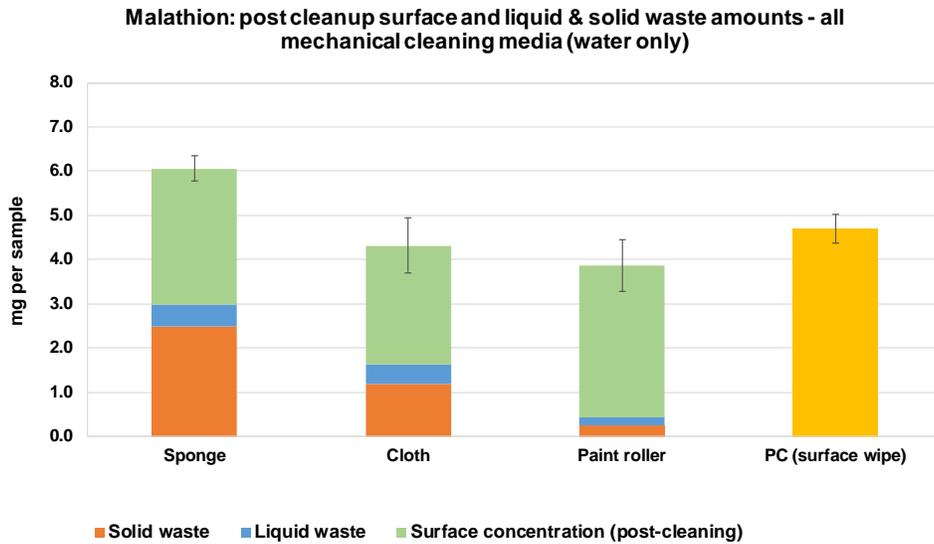
While considering the overall inter-method decontamination effectiveness for field applications, it is important to answer questions on how mechanical cleaning contributes to the removal of the target chemical from a contaminated surface. In addition to a better understanding of the neutralization versus mechanical removal paradigms, the analysis of the chemical residue in expended materials provides insight on the necessity of additional remediation strategies of solid waste prior to disposal. Mechanical removal steps of a contaminant certainly offer an advantage in terms of achieving optimal decontamination efficacy, but mechanical removal of contaminant also leads to generation of large amounts of contaminated solid waste. Figure 5-10 shows the amount of expended cleaning material generated during decontamination of an approximate area of 12 ft<sup>2</sup>, used in decontamination experiments under this project (four samples per each type (3) of cleaning medium; 3 TCs and 1 PB per test plus cleaning media used for water rinses).



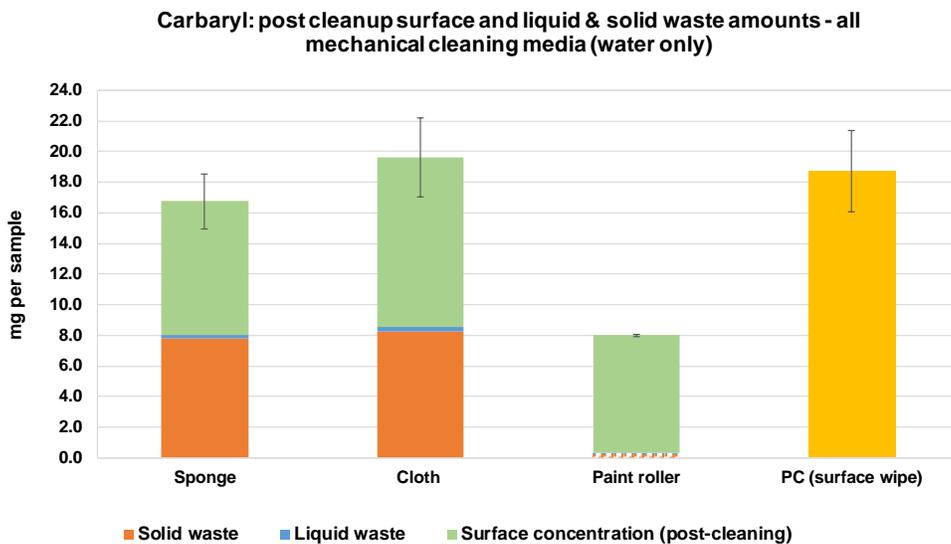
**Figure 5-10. Expended cleaning materials generated during decontamination of an approximate area of 12 ft<sup>2</sup> using three types of cleaning media**

It is also important to consider a possible transport of chemicals to liquid waste, mostly in the context of the immobilization of a chemical/pesticide to a more labile, potentially hazardous, contamination form, and the consequent need for development of proper procedures for handling and disposal of post-decontamination liquid waste. The initial estimates on chemical transfer to solid and liquid waste were performed using testing procedures described in Sections 3.5.3 and 3.5.4. The chemical-specific results of cleaned surface area are given in Appendix A (Table A-10 and A-11) and depicted in Figures 5-11 and 5-12. These tests were conducted without proper PCs as the purpose was to measure the relative distribution of pesticides across surface, media, and liquid effluent, not the absolute concentration in comparison to amount applied. The PC values for malathion and carbaryl in Figures 5-11 and 5-12, respectively, are the best estimates of the amount of pesticide applied to the coupon (derived from values in Table

The post-cleaning concentrations (with a single-step DCP deployed using water only) show that no significant removal of malathion or carbaryl was provided by the mechanical removal step only; the average concentration of the pesticide present on test surfaces post-cleaning was from 29 to 37 and from 83 to 190 mg/m<sup>2</sup> for malathion and carbaryl, respectively. This concentration is in the pre-decontamination surface concentration range (Table 3-4). The highest relative transfer to cleaning media was observed for simulated-DCP4 deployed with a sponge, followed by a cloth. No malathion or carbaryl was detected on paint rollers. No carbaryl was detected in the liquid waste from paint roller-based DCP5.



**Figure 5-11. Malathion post-cleanup surface, liquid and solid waste amounts for all mechanical cleaning media; % contribution calculated based on 1/2 LOQ**



**Figure 5-12. Carbaryl post-cleanup surface, liquid and solid waste amounts for all mechanical cleaning media; dashed bars – no quantification, % contribution calculated based on 1/2 LOQ**

These results suggest that effectiveness of the tested DCPs with decontaminants (Section 5.3) should be attributed mostly to chemical reactivity of the decontaminants, but some cleaning media, especially those characterized by high-liquid decontaminant loading, can take up contaminant via mechanical wet-scrubbing and/or wiping steps. The transfer of the contaminant to liquid waste was minimal for DCPs with mechanical removal steps, with a maximum 10% of the total amount of chemical recovered transferred to runoff.

## 6.0. Quality Assurance/Quality Control

### 6.1. Test Equipment Calibration

All equipment was verified as calibrated at the time of use. Calibration of instruments was done at the frequency shown in Table 6-1. In case of any deficiencies, instruments were adjusted to meet calibration tolerances and/or recalibrated prior to testing. In the case of the GC/MS instrument, any initial calibration deficiencies were noted. The GC/MS was recalibrated prior to analysis. If the calibration tolerances for continuous calibration were not met, the GC/MS was recalibrated and affected samples were re-analyzed.

**Table 6-1. Instrument Calibration Frequency**

Equipment	Calibration/Certification	Expected Tolerance	Results
Thermometer	Compare to independent NIST thermometer (a thermometer that is recertified annually by either NIST or an ISO-17025 facility) value once per quarter.	$\pm 1$ °C	100%
Stopwatch	Compare to official U.S. time @ time.gov every 30 days.	$\pm 1$ min/30 days	100%
Micropipettes	Certified as calibrated at time of use. Recalibrated by gravimetric evaluation of performance to manufacturer's specifications every year.	$\pm 5\%$	100%
Scale	Certified as calibrated at time of use. Calibration verified yearly by the AEMD Metrology Laboratory.	$\pm 1$ g	100%
pH meter	Three-point calibration using NIST-traceable buffer solutions immediately prior to testing.	$\pm 0.1$ pH units	100%
Graduated cylinder	Certified by manufacturer at the time of use.	$\pm 1$ mL	100%
Solvent dispenser	Certified by manufacturer at the time of use. Re-checked volume delivered using graduated cylinder prior to use.	$\pm 1$ mL	100%
GC/MS	6- to 7- point calibration prior to analysis; continuous calibration prior to each analytical run; re-calibrate when continuous calibration fails acceptance criteria and/or after system maintenance; details in Section 4.5.	$\pm 20\%$ at mid-point	Carbaryl: 100% <sup>a*</sup> Malathion: 100% <sup>b</sup>
NIST = National Institute of Standards and Technology; ISO = International Organization for Standardization; <sup>a</sup> malathion decontamination experiments; continuous calibration results: 76-123% at mid-point; average RPD: $\pm 18\%$ ; <sup>b</sup> carbaryl decontamination experiments; continuous calibration results: 80-112% at mid-point; average RPD: $\pm 9.2\%$			

### 6.2. Data Quality Results for Critical Measurements

The following measurements have been deemed critical to accomplishing part or all of the project objectives:

- Initial and post-cleaning surface concentration of malathion and carbaryl in the wipe sampling extracts as determined by GC/MS.
- Contact time and dwell time.
- Hydrogen peroxide concentration and pH of EasyDECOR® DF200 decontamination solution prior to each test.
- Volume of decontaminant (cleaning solution) and rinse water applied.
- Mass of liquid waste (runoff and rinsate).
- Mass of solid waste (DCP 3 through DCP 5 only).
- Volume of extraction solvent.

The data quality indicators (DQIs) for test measurements are provided in Table 6-2. The limited number of results/tests that were not within acceptance criteria (as determined in the project specific QAPP) were not indicative of any systematic error introduced into the experimental results and do not change the general findings of this study.

**Table 6-2. Acceptance Criteria for Critical Measurements and Corresponding Test Results**

Critical Measurement	Target Value and Acceptance Criteria	Results
Contact/weathering time	30 min ± 1 min	All contact times (CTs) within 30 min ± 1 min from spiking; test-specific results are in Appendix A, Tables A-1 through A-8
Dwell time (decontamination interaction time) or rinse drying time	30 min–46 h ± 5 min	All dwell times (DTs) within acceptance criteria; test-specific results are in Appendix A, Tables A-1 through A-8
Delivery of target surface concentration of chemical*	80 -120% of target	The mean spike controls for decontamination tests were 115 ± 20% SD for malathion and 69% ± 16% SD for carbaryl with coefficients of variation ≤30% between tests (both chemicals); results are in Table 4-6.
Recovery of chemical from positive control	60-140% of theoretical target, 30% coefficient of variation for identical test set	All tests had <30% coefficient of variation for non-decontaminated samples resulting from identical test set**; test-specific results for positive control coupons are in Table 3-4.
Recovery of chemical from decontaminated TCs	<30% coefficient of variation for identical test set	6 out of 40 tests had >30% coefficient of variation for decontaminated samples resulting from identical test set; test-specific results are in Table 5-1 through 5-4
Procedural blank	< 5% of the analyte amount recovered from the positive control.	All procedural blank samples within acceptance criteria; all reported <LOQ; test-specific results are in Table 5-1 through 5-4.
Solvent blank	<LOQ	All solvent blanks reported <LOQ
Decontaminant volume applied	± 20% of target volume	Cleaning media and surface loading volumes of decontaminant or water rinse: <ul style="list-style-type: none"> <li>- Pressurized paint sprayer: 20 mL</li> <li>- Spray bottle: 20 mL</li> <li>- Cotton cloth: 150 mL</li> <li>- Cleaning sponge (1/2 sponge): 100 mL</li> <li>- Paint roller (1/2 roller): 50 mL</li> </ul> The volume applied to the cleaning media was pre-measured using a graduated cylinder, therefore constant throughout the entire test matrix. Test-specific results for resulting surface loading volumes are in Tables A-2 through 4-4 in Appendix A and are summarized in Table 3-8.
H <sub>2</sub> O <sub>2</sub> concentration of EasyDECON® DF200****	Go	All finished blend batches passed Go/No Go test prior to testing
pH of EasyDECON® DF200****	9.6–9.9	All finished blend batches were within target pH range prior to testing

*\*As determined by analysis of control spikes; criteria for recovery of chemical from surface; \*\* Optimized method used for decontamination testing; \*\*\*Volumes that were delivered on the surface as determined by gravimetric measurements of test boxes before and after application; \*\*\*\*for finished blend.*

## 7.0. Summary

After comparing the decontamination efficiencies of the single- and multistep decontamination and cleanup procedures deployed onto reference nonporous material (stainless steel), the data indicate the following:

(a) The specialized decontamination formula tested (activated hydrogen peroxide-based, EasyDECON® DF200) had high decontamination effectiveness against the selected pesticides tested in this study: >99.7% DE for malathion and 94.8% to > 97.2% DE for carbaryl. Post-decontamination residual levels of both contaminants were lower than a human-health risk-based cleanup threshold for adults as developed for this study. Human-health risk-based cleanup threshold for a child may not have been reached due to the quantification limits for both contaminants in this study.

(b) The off-the-shelf decontaminant (concentrated germicidal bleach) had > 99.7% DE for malathion, and 63% to 83% DE for carbaryl. Only one DCP (multistep DCP 4 applied by sponge) rendered a residual surface concentration for carbaryl near the project-specific human-health risk-based cleanup threshold for adults. Human-health risk-based cleanup threshold for a child for carbaryl were not reached while for malathion this threshold may not have been reached due to the higher quantification limit for malathion in this study.

(c) The higher solubility in water was linked to generally higher decontamination efficacy observed for malathion. For less water-soluble carbaryl, the addition of surfactant (benzalkonium chloride) in EasyDECON® DF200 formula was considered to influence the performance of EasyDECON® DF200-based DCPs positively.

(d) The natural attenuation of pesticides was confirmed to occur on nonporous materials after 24-hours post-contamination, with significant (>90%) and rapid (within 30-minutes post-contamination) permeation transfer observed on semi-porous material surfaces for both chemicals. These results indicated that further studies are needed for optimization of decontamination procedures for neutralization of chemical agents absorbed into semi-porous building materials, including potential modification of the decontamination solution.

(e) The solid and liquid waste generated when using only water was confirmed to be contaminated with the applied pesticides, with generation rates of chemical mass per cleaned area reaching tens of milligrams per m<sup>2</sup>; the rate of chemical transfer to waste seemed to be mostly related to type of cleaning media used and to a lesser extent, chemical solubility in water. The long-term chemical reactivity of decontaminants in liquid and solid and treatment methods for neutralization of contaminated waste were, however, not addressed in the present study.

The results of this study confirm that decontamination and cleanup methods should be selected based on the reactivity of the chemical agent-active ingredient/ingredients of the decontaminant chemical, as well as the ability of the decontaminant to physically remove the chemical agent on the surface of the material. Such targeted DCPs should ideally consider the physical and chemical properties (e.g., water solubility) of the chemical agent as well as the potential of the chemical to migrate/permeate into semi-porous or porous materials. The multiple cleanup decontamination modes tested in this study were good candidate DCPs for neutralization of high pesticide burdens from nonporous surfaces. Further studies on optimization of decontamination strategies of porous or semi-porous surfaces are currently underway.

This project was a bench scale level study with the limitations that this effort did not address the possible formation of toxic decontamination byproducts. This omission is noteworthy considering the use of malathion as one of the targeted pesticides that may degrade to malaoxon, an oxidation byproduct of equal or higher toxicity than malathion. Further, all materials were clean and prepared specifically for this study; dirt and grime may impact the efficacy. Lastly, pesticides were applied here as a neat film. This application may deviate from the application of a technical formulation containing these pesticides.

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# Appendix A: Supporting Information

**Table A-1. Experimental parameters for malathion decontamination with bleach (single-step procedure)**

Test and Sample ID	Contamination	Decontamination Step				Water Rinse				Wipe sampling	
	Start Time	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time
	(hh:mm:ss)		(g)		(hh:mm:ss)		(g)		(hh:mm:ss)		
Single-step DCP 1 Hand-held pressurized sprayer											
DCP1S-MA-SS-BL-PS-1-TC-1-190117	10:10:00	10:10:00	10:10:10	20.0	31	11:10:00	11:10:10	20.0	17	11:10:00	11:12:49
DCP1S-MA-SS-BL-PS-1-TC-2-190117	10:20:00	10:20:00	10:20:11	20.0	30	11:20:00	11:20:11	20.0	16	11:20:00	11:22:44
DCP1S-MA-SS-BL-PS-1-TC-3-190117	10:30:00	10:30:00	10:30:10	20.0	27	11:30:00	11:30:10	20.0	17	11:30:00	11:32:52
Single-step DCP 2 Spray bottle											
DCP2S-MA-SS-BL-SB-1-TC-1-190117	10:40:00	10:40:00	10:40:10	19.6	19	11:40:00	11:40:10	20.6	18	11:40:00	11:42:48
DCP2S-MA-SS-BL-SB-1-TC-2-190117	10:50:00	10:50:00	10:50:10	20.0	19	11:50:00	11:50:11	19.7	17	11:50:00	11:52:47
DCP2S-MA-SS-BL-SB-1-TC-3-190117	11:00:00	11:00:00	11:00:11	20.0	18	12:00:00	12:00:10	20.2	16	12:00:00	12:03:01
Single-step DCP 3 Cleaning cloth											
DCP3S-MA-SS-BL-RG-1-TC-1-120117	10:24:00	10:24:00	10:24:10	4.5	4.0	11:24:00	11:24:10	2.7	2	11:24:00	11:27:01
DCP3S-MA-SS-BL-RG-1-TC-2-120117	10:31:00	10:31:00	10:31:10	4.4	3.0	11:31:00	11:31:10	3.1	2	11:31:00	11:34:05
DCP3S-MA-SS-BL-RG-1-TC-3-120117	10:38:00	10:38:00	10:38:10	3.5	4.0	11:38:00	11:38:10	3.2	2	11:38:00	11:41:08
Single-step DCP 4 Cleaning sponge											
DCP4S-MA-SS-BL-SP-1-TC-1-120117	10:45:00	10:45:00	10:45:10	35.7	35	11:45:00	11:45:10	16.2	15	11:45:00	11:48:20
DCP4S-MA-SS-BL-SP-1-TC-2-120117	10:52:00	10:52:00	10:52:10	32.9	31	11:52:00	11:52:10	14.7	13	11:52:00	11:55:06
DCP4S-MA-SS-BL-SP-1-TC-3-120117	10:59:00	10:59:00	10:59:10	34.8	33	11:59:00	11:59:10	7.5	5	11:59:00	12:02:18
Single-step DCP 5 Paint roller											
DCP5S-MA-SS-BL-PR-1-TC-1-120117	11:06:00	11:06:00	11:06:10	2.8	3.0	12:06:00	12:06:10	5.1	4	12:06:00	12:08:57
DCP5S-MA-SS-BL-PR-1-TC-2-120117	11:13:00	11:13:00	11:13:10	3.7	4.0	12:13:00	12:13:10	4.2	4	12:13:00	12:16:11
DCP5S-MA-SS-BL-PR-1-TC-3-120117	11:20:00	11:20:00	11:20:10	3.1	3.0	12:20:00	12:20:10	3.4	33	12:20:00	12:23:03

**Table A-2. Experimental parameters for malathion decontamination with bleach (multistep procedure)**

Test and Sample ID	Contamination	Decontamination Step 1				Decontamination Step 2				Water Rinse				Wipe sampling	
	Start Time	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time
	(hh:mm:ss)	(hh:mm:ss)	(hh:mm:ss)	(g)	(g)	(hh:mm:ss)	(hh:mm:ss)	(g)	(g)	(hh:mm:ss)	(hh:mm:ss)	(g)	(g)	(hh:mm:ss)	(hh:mm:ss)
Multistep DCP 1 Hand-held pressurized sprayer															
DCP1M-MA-SS-BL-PS-4/24-TC-1-200117	8:50:00	8:50:00	8:50:10	20	23	12:50:00	12:50:10	20	18	12:50:00	12:50:11	20	21	12:50:00	12:52:48
DCP1M-MA-SS-BL-PS-4/24-TC-2-200117	9:00:00	9:00:00	9:00:10	20	23	13:00:00	13:00:12	20	22	13:00:00	13:00:10	20	20	13:00:00	13:02:43
DCP1M-MA-SS-BL-PS-4/24-TC-3-200117	9:10:00	9:10:00	9:10:10	20	21	13:10:00	13:10:10	20	26	13:10:00	13:10:12	20	19	13:10:00	13:12:56
DCP2M-MA-SS-BL-PS-4/24-PB-1-200117	----	9:20:00	9:20:10	20	24	13:20:00	13:20:15	20	29	13:20:00	13:20:10	20	21	13:20:00	13:22:39
Multistep DCP 2 Spray bottle															
DCP2M-MA-SS-BL-SB-4/24-TC-1-200117	9:30:00	9:30:00	9:30:13	23.0	21	13:30:00	13:30:13	19.7	19	13:30:00	13:30:15	20.2	19	13:30:00	13:32:54
DCP2M-MA-SS-BL-SB-4/24-TC-2-200117	9:40:00	9:40:00	9:40:12	20.9	19	13:40:00	13:40:12	19.8	19	13:40:00	13:40:15	19.6	18	13:40:00	13:42:56
DCP2M-MA-SS-BL-SB-4/24-TC-3-200117	9:50:00	9:50:00	9:50:12	21.9	20	13:50:00	13:50:12	20.0	18	13:50:00	13:50:16	20.4	19	13:50:00	13:52:51
DCP2M-MA-SS-BL-SB-4/24-PB-1-200117	----	10:00:00	10:00:12	22.1	22	14:00:00	14:00:10	20.3	20	14:00:00	14:00:13	19.7	19	14:00:00	14:02:55
Multistep DCP 3 Cleaning cloth															
DCP3M-MA-SS-BL-RG-4/24-TC-1-130117	9:00:00	9:00:00	9:00:10	3.5	2.0	13:00:00	13:00:10	3.9	3	13:00:00	13:00:10	3.7	2	13:00:00	13:03:45
DCP3M-MA-SS-BL-RG-4/24-TC-2-130117	9:07:00	9:07:00	9:07:10	3.8	3.0	13:07:00	13:07:10	4.5	3	13:07:00	13:07:10	3.3	2	13:07:00	13:09:55
DCP3M-MA-SS-BL-RG-4/24-TC-3-130117	9:14:00	9:14:00	9:14:10	4.4	3.0	13:14:00	13:14:10	4.3	4	13:14:00	13:14:10	2.9	3	13:14:00	13:17:03
DCP3M-MA-SS-BL-RG-4/24-PB-1-130117	----	9:21:00	9:21:10	4.3	3.0	13:21:00	13:21:10	3.7	2	13:21:00	13:21:10	3.3	2	13:21:00	13:24:13
Multistep DCP 4 Cleaning sponge															
DCP4M-MA-SS-BL-SP-4/24-TC-1-130117	9:28:00	9:28:00	9:28:10	28.7	27	13:28:00	13:28:10	34.1	33	13:28:00	13:28:10	12.4	11	13:28:00	13:31:06
DCP4M-MA-SS-BL-SP-4/24-TC-2-130117	9:35:00	9:35:00	9:35:10	21.2	21	13:35:00	13:35:10	37.1	36	13:35:00	13:35:10	11.8	11	13:35:00	13:38:03
DCP4M-MA-SS-BL-SP-4/24-TC-3-130117	9:42:00	9:42:00	9:42:10	21.6	21	13:42:00	13:42:10	28.3	27	13:42:00	13:42:10	26.8	26	13:42:00	13:45:11
DCP4M-MA-SS-BL-SP-4/24-PB-1-130117	----	9:49:00	9:49:10	32.5	32	13:49:00	13:49:10	24.0	23	13:49:00	13:49:10	8.3	8	13:49:00	13:52:02
Multistep DCP 5 Paint roller															
DCP5M-MA-SS-BL-PR-4/24-TC-1-130117	9:56:00	9:56:00	9:56:10	4.0	4.0	13:56:00	13:56:10	2.4	2	13:56:00	13:56:10	1.2	4	13:56:00	13:59:01
DCP5M-MA-SS-BL-PR-4/24-TC-2-130117	10:03:00	10:03:00	10:03:10	8.4	3.0	14:03:00	14:03:10	6.1	6	14:03:00	14:03:10	3.5	3	14:03:00	14:06:00
DCP5M-MA-SS-BL-PR-4/24-TC-3-130117	10:10:00	10:10:00	10:10:10	2.6	0	14:10:00	14:10:10	2.8	2	14:10:00	14:10:10	3.4	3	14:10:00	14:12:58
DCP5M-MA-SS-BL-PR-4/24-PB-1-130117	----	10:17:00	10:17:10	5.0	6.0	14:17:00	14:17:10	6.8	7	14:17:00	14:17:10	3.8	4	14:17:00	14:19:59

**Table A-3. Experimental parameters for carbaryl decontamination with bleach (single-step procedure)**

Test and Sample ID	Contamination	Decontamination Step				Water Rinse				Wipe sampling	
	Start Time	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time
	(hh:mm:ss)		(g)		(hh:mm:ss)		(g)		(hh:mm:ss)		
Single-step DCP 1 Hand-held pressurized sprayer											
DCP1S-CA-SS-BL-PS-1-TC-1-080217	10:10:00	10:10:00	10:10:00	20	28	11:10:00	11:10:10	20	23	11:10:00	11:12:58
DCP1S-CA-SS-BL-PS-1-TC-2-080217	10:20:00	10:20:00	10:20:10	20	26	11:20:00	11:20:11	20	24	11:20:00	11:22:51
DCP1S-CA-SS-BL-PS-1-TC-3-080217	10:30:00	10:30:00	10:30:10	20	27	11:30:00	11:30:10	20	22	11:30:00	11:33:00
Single-step DCP 2 Spray bottle											
DCP2S-CA-SS-BL-SB-1-TC-1-080217	10:40:00	10:40:00	10:40:10	20.7	21	11:40:00	11:40:22	20.5	18	11:40:00	11:42:51
DCP2S-CA-SS-BL-SB-1-TC-2-080217	10:50:00	10:50:00	10:50:10	20.9	19	11:50:00	11:50:15	20.8	19	11:50:00	11:53:02
DCP2S-CA-SS-BL-SB-1-TC-3-080217	11:00:00	11:00:00	11:00:10	19.9	19	12:00:00	12:00:35	19.5	18	12:00:00	12:02:58
Single-step DCP 3 Cleaning cloth											
DCP3S-CA-SS-BL-RG-1-TC-1-150217	10:24:00	10:24:00	10:24:10	3.0	2.0	11:24:00	11:24:10	2	1	11:24:00	11:26:57
DCP3S-CA-SS-BL-RG-1-TC-2-150217	10:31:00	10:31:00	10:31:10	3.1	2.0	11:31:00	11:31:10	2.1	1	11:31:00	11:33:58
DCP3S-CA-SS-BL-RG-1-TC-3-150217	10:38:00	10:38:00	10:38:10	3.5	3.0	11:38:00	11:38:10	2.8	2	11:38:00	11:40:51
Single-step DCP 4 Cleaning sponge											
DCP4S-CA-SS-BL-SP-1-TC-1-150217	10:45:00	10:45:00	10:45:10	16	15	11:45:00	11:45:10	16.9	15	11:45:00	11:47:48
DCP4S-CA-SS-BL-SP-1-TC-2-150217	10:52:00	10:52:00	10:52:10	15.3	14	11:52:00	11:52:10	16.9	16	11:52:00	11:55:03
DCP4S-CA-SS-BL-SP-1-TC-3-150217	10:59:00	10:59:00	10:59:10	19.7	19	11:59:00	11:59:10	14.7	14	11:59:00	12:02:59
Single-step DCP 5 Paint roller											
DCP5S-CA-SS-BL-PR-1-TC-1-150217	11:06:00	11:06:00	11:06:10	5.1	5.0	12:06:00	12:06:10	5.1	4	12:06:00	12:08:56
DCP5S-CA-SS-BL-PR-1-TC-2-150217	11:13:00	11:13:00	11:13:10	6.7	6.0	12:13:00	12:13:10	3.4	3	12:13:00	12:15:52
DCP5S-CA-SS-BL-PR-1-TC-3-150217	11:20:00	11:20:00	11:20:10	4.0	4.0	12:20:00	12:20:10	2.8	2	12:20:00	12:22:49

**Table A-4. Experimental parameters for carbaryl decontamination with bleach (multistep procedure)**

Test and Sample ID	Contamination	Decontamination Step 1				Decontamination Step 2				Water Rinse				Wipe sampling	
	Start Time	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time
	(hh:mm:ss)	(hh:mm:ss)	(hh:mm:ss)	(g)	(g)	(hh:mm:ss)	(hh:mm:ss)	(g)	(g)	(hh:mm:ss)	(hh:mm:ss)	(g)	(g)	(hh:mm:ss)	(hh:mm:ss)
<b>Multistep DCP 1 Hand-held pressurized sprayer</b>															
DCP1M-CA-SS-BL-PS-4/24-TC-1-090217	8:50:00	8:50:00	8:50:10	20	22	12:50:00	12:50:10	20	27	12:50:00	12:50:10	20	23	12:50:00	12:52:43
DCP1M-CA-SS-BL-PS-4/24-TC-2-090217	9:00:00	9:00:00	9:00:10	20	24	13:00:00	13:00:10	20	26	13:00:00	13:00:10	20	21	13:00:00	13:03:07
DCP1M-CA-SS-BL-PS-4/24-TC-3-090217	9:10:00	9:10:00	9:10:08	20	24	13:10:00	13:10:10	20	24	13:10:00	13:10:10	20	22	13:10:00	13:12:55
DCP2M-CA-SS-BL-SB-4/24-PB-1-090217	----	9:20:00	9:20:10	20	23	13:20:00	13:20:10	20	24	13:20:00	13:20:10	20	22	13:20:00	13:23:01
<b>Multistep DCP 2 Spray bottle</b>															
DCP2M-CA-SS-BL-SB-4/24-TC-1-090217	9:30:00	9:30:00	9:30:12	21.9	20	13:30:00	13:30:10	20.4	20	13:30:00	13:30:30	20.2	19	13:30:00	13:33:00
DCP2M-CA-SS-BL-SB-4/24-TC-2-090217	9:40:00	9:40:00	9:40:11	20.0	19	13:40:00	13:40:10	20.4	20	13:40:00	13:40:44	20.7	19	13:40:00	13:42:58
DCP2M-CA-SS-BL-SB-4/24-TC-3-090217	9:50:00	9:50:00	9:50:10	19.6	19	13:50:00	13:50:10	20.0	20	13:50:00	13:50:46	20.2	19	13:50:00	13:53:05
DCP2M-CA-SS-BL-SB-4/24-PB-1-090217	----	10:00:00	10:00:11	20.3	19	14:00:00	14:00:33	20.7	20	14:00:00	14:00:40	21.0	19	14:00:00	14:03:04
<b>Multistep DCP 3 Cleaning cloth</b>															
DCP3M-CA-SS-BL-RG-4/24-TC-1-160217	9:00:00	9:00:00	9:00:10	3.5	3.0	13:00:00	13:00:10	3.8	2	13:00:00	13:00:10	1.9	1	13:00:00	13:02:45
DCP3M-CA-SS-BL-RG-4/24-TC-2-160217	9:07:00	9:07:00	9:07:10	3.8	3.0	13:07:00	13:07:10	3.7	2	13:07:00	13:07:10	2.1	2	13:07:00	13:09:52
DCP3M-CA-SS-BL-RG-4/24-TC-3-160217	9:14:00	9:14:00	9:14:10	4.0	4.0	13:14:00	13:14:10	2.9	1	13:14:00	13:14:10	2.2	2	13:14:00	13:16:50
DCP3M-CA-SS-BL-RG-4/24-PB-1-160217	----	9:21:00	9:21:10	3.5	3.0	13:21:00	13:21:10	2.9	2	13:21:00	13:21:10	2.7	2	13:21:00	13:24:00
<b>Multistep DCP 4 Cleaning sponge</b>															
DCP4M-CA-SS-BL-SP-4/24-TC-1-160217	9:28:00	9:28:00	9:28:10	40.3	40	13:28:00	13:28:10	31.1	30	13:28:00	13:28:10	27	26	13:28:00	13:31:00
DCP4M-CA-SS-BL-SP-4/24-TC-2-160217	9:35:00	9:35:00	9:35:10	28.5	27	13:35:00	13:35:10	19	17	13:35:00	13:35:10	15	13	13:35:00	13:37:45
DCP4M-CA-SS-BL-SP-4/24-TC-3-160217	9:42:00	9:42:00	9:42:10	21.9	21	13:42:00	13:42:10	29.6	28	13:42:00	13:42:10	15.3	16	13:42:00	13:44:51
DCP4M-CA-SS-BL-SP-4/24-PB-1-160217	----	9:49:00	9:49:10	16.4	15	13:49:00	13:49:10	29.9	27	13:49:00	13:49:10	22.5	21	13:49:00	13:52:00
<b>Multistep DCP 5 Paint roller</b>															
DCP5M-CA-SS-BL-PR-4/24-TC-1-160217	9:56:00	9:56:00	9:56:10	4.7	3.0	13:56:00	13:56:10	4.4	3	13:56:00	13:56:10	2.9	2	13:56:00	13:58:41
DCP5M-CA-SS-BL-PR-4/24-TC-2-160217	10:03:00	10:03:00	10:03:10	4.5	4.0	14:03:00	14:03:10	5.5	6	14:03:00	14:03:10	4.8	4	14:03:00	14:05:56
DCP5M-CA-SS-BL-PR-4/24-TC-3-160217	10:10:00	10:10:00	10:10:10	6.4	6.0	14:10:00	14:10:10	4.4	4	14:10:00	14:10:10	2.5	3	14:10:00	14:12:49
DCP5M-CA-SS-BL-PR-4/24-PB-1-160217	----	10:17:00	10:17:10	4.9	5.0	14:17:00	14:17:10	4.3	4	14:17:00	14:17:10	2.7	3	14:17:00	14:19:47

**Table A-5. Experimental parameters for malathion decontamination with EasyDECON® DF200 (single-step procedure)**

Test and Sample ID	Contamination	Decontamination Step				Water Rinse				Wipe sampling	
	Start Time	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time
	(hh:mm:ss)		(g)		(hh:mm:ss)		(g)		(hh:mm:ss)		
Single-step DCP 1 Hand-held pressurized sprayer											
DCP1S-MA-SS-ED-PS-24-TC-1-291216	10:10:00	10:10:05	10:10:15	20	48	11:10:00	11:10:10	20	23	11:10:00	11:13:06
DCP1S-MA-SS-ED-PS-24-TC-2-291216	10:20:00	10:20:00	10:20:19	20	37	11:20:00	11:20:13	20	18	11:20:00	11:23:09
DCP1S-MA-SS-ED-PS-24-TC-3-291216	10:30:00	10:30:00	10:30:18	20	39	11:30:00	11:30:13	20	18	11:30:00	11:33:31
Single-step DCP 2 Spray bottle											
DCP2S-MA-SS-ED-SB-24-TC-1-291216	10:40:00	10:40:00	10:40:15	20.7	18	11:40:30	11:40:43	21.7	19	11:40:00	11:43:32
DCP2S-MA-SS-ED-SB-24-TC-2-291216	10:50:00	10:50:00	10:50:15	19.8	17	11:50:00	11:50:19	21.5	18	11:50:00	11:53:28
DCP2S-MA-SS-ED-SB-24-TC-3-291216	11:00:00	11:00:00	11:00:22	20.4	21	12:00:00	12:00:16	21.2	18	12:00:00	12:03:21
Single-step DCP 3 Cleaning cloth											
DCP3S-MA-SS-ED-RG-1-TC-1-050117	10:24:00	10:24:00	10:24:10	11.3	9.0	11:24:00	11:24:10	0.2	0	11:24:00	11:26:57
DCP3S-MA-SS-ED-RG-1-TC-2-050117	10:31:00	10:31:00	10:31:10	6.3	4.0	11:31:00	11:31:10	2.2	1	11:31:00	11:34:01
DCP3S-MA-SS-ED-RG-1-TC-3-050117	10:38:00	10:38:00	10:38:10	5.0	2.0	11:38:00	11:38:10	7.5	1	11:38:00	11:40:52
Single-step DCP 4 Cleaning sponge											
DCP4S-MA-SS-ED-SP-1-TC-1-050117	10:45:00	10:45:00	10:45:10	16.8	16	11:45:00	11:45:10	9.0	6	11:45:00	11:47:47
DCP4S-MA-SS-ED-SP-1-TC-2-050117	10:52:00	10:52:00	10:52:10	21.0	20	11:52:00	11:52:10	19.1	18	11:52:00	11:55:00
DCP4S-MA-SS-ED-SP-1-TC-3-050117	10:59:00	10:59:00	10:59:10	22.6	20	11:59:00	11:59:10	14.5	12	11:59:00	12:02:01
Single-step DCP 5 Paint roller											
DCP5S-MA-SS-ED-PR-1-TC-1-050117	11:06:00	11:06:00	11:06:10	4.8	4.0	12:06:00	12:06:10	6.4	6	12:06:00	12:08:51
DCP5S-MA-SS-ED-PR-1-TC-2-050118	11:13:00	11:13:00	11:13:10	4.3	4.0	12:13:00	12:13:10	7.6	7	12:13:00	12:15:47
DCP5S-MA-SS-ED-PR-1-TC-3-050119	11:20:00	11:20:00	11:20:10	5.6	4.0	12:20:00	12:20:10	8.6	8	12:20:00	12:23:02

**Table A-6. Experimental parameters for malathion decontamination with EasyDECON® DF200 (multistep procedure)**

Test and Sample ID	Contamination	Decontamination Step 1				Decontamination Step 2				Water Rinse				Wipe sampling	
	Start Time	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time
	(hh:mm:ss)	(hh:mm:ss)	(hh:mm:ss)	(g)	(g)	(hh:mm:ss)	(hh:mm:ss)	(g)	(g)	(hh:mm:ss)	(hh:mm:ss)	(g)	(g)	(hh:mm:ss)	(hh:mm:ss)
<b>Multistep DCP 1 Hand-held pressurized sprayer</b>															
DCP1M-MA-SS-ED-PS-4/24-TC-1-301216	9:00:00	9:00:00	9:00:10	20	46	13:00:00	13:00:12	20	55	13:00:00	13:00:11	20	20	13:00:00	13:03:24
DCP1M-MA-SS-ED-PS-4/24-TC-2-301216	9:10:00	9:10:00	9:10:14	20	49	13:10:00	13:10:14	20	55	13:10:00	13:10:14	20	19	13:10:00	13:13:32
DCP1M-MA-SS-ED-PS-4/24-TC-3-301216	9:20:00	9:20:00	9:20:11	20	30	13:20:00	13:20:15	20	54	13:20:00	13:20:10	20	20	13:20:00	13:23:26
<b>Multistep DCP 2 Spray bottle</b>															
DCP2M-MA-SS-ED-SB-4/24-TC-1-301216	9:30:00	9:30:00	9:30:16	20.3	18	13:30:00	13:30:17	20.2	19	13:30:00	13:30:16	20.3	18	13:30:00	13:33:16
DCP2M-MA-SS-ED-SB-4/24-TC-2-301216	9:40:00	9:40:00	9:40:16	20.1	17	13:40:00	13:40:18	20.1	19	13:40:00	13:40:13	20.5	18	13:40:00	13:43:16
DCP2M-MA-SS-ED-SB-4/24-TC-3-301216	9:50:00	9:50:00	9:50:19	21.2	20	13:50:00	13:50:14	19.5	17	13:50:00	13:50:17	20.3	18	13:50:00	13:53:11
DCP2M-MA-SS-ED-SB-4/24-PB-1-301216	----	10:00:00	10:00:18	20.5	19	14:00:00	14:00:14	19.7	18	14:00:00	14:00:16	20.9	17	14:00:00	14:03:09
<b>Multistep DCP 3 Cleaning cloth</b>															
DCP3M-MA-SS-ED-RG-4/24-TC-1-060117	9:00:00	9:00:00	9:00:10	7.0	3.0	13:00:00	13:00:10	5.3	1.0	13:00:00	13:00:10	9.4	1	13:00:00	13:03:15
DCP3M-MA-SS-ED-RG-4/24-TC-2-060117	9:07:00	9:07:00	9:07:10	4.7	3.0	13:07:00	13:07:10	4.8	3.0	13:07:00	13:07:10	2.2	2	13:07:00	13:10:05
DCP3M-MA-SS-ED-RG-4/24-TC-3-060117	9:14:00	9:14:00	9:14:10	3.8	1.0	13:14:00	13:14:10	7.6	5.0	13:14:00	13:14:10	1.6	1	13:14:00	13:17:09
DCP3M-MA-SS-ED-RG-4/24-PB-1-060117	----	9:21:00	9:21:10	4.7	2.0	13:21:00	13:21:10	5.3	2.0	13:21:00	13:21:10	2.1	1	13:21:00	13:24:17
<b>Multistep DCP 4 Cleaning sponge</b>															
DCP4M-MA-SS-ED-SP-4/24-TC-1-060117	9:28:00	9:28:00	9:28:10	34.4	32	13:28:00	13:28:10	12.9	11	13:28:00	13:28:10	11.4	10	13:28:00	13:31:24
DCP4M-MA-SS-ED-SP-4/24-TC-2-060117	9:35:00	9:35:00	9:35:10	20.4	18	13:35:00	13:35:10	8.9	7	13:35:00	13:35:10	7.4	6	13:35:00	13:38:18
DCP4M-MA-SS-ED-SP-4/24-TC-3-060117	9:42:00	9:42:00	9:42:10	29.8	27	13:42:00	13:42:10	19.2	18	13:42:00	13:42:10	5.0	4	13:42:00	13:45:08
DCP4M-MA-SS-ED-SP-4/24-PB-1-060117	----	9:49:00	9:49:10	27.2	29	13:49:00	13:49:10	13.0	10	13:49:00	13:49:10	6.8	5	13:49:00	13:52:18
<b>Multistep DCP 5 Paint roller</b>															
DCP5M-MA-SS-ED-PR-4/24-TC-1-060117	9:56:00	9:56:00	9:56:10	2.5	2.0	13:56:00	13:56:10	7.1	7.0	13:56:00	13:56:10	2.9	2	13:56:00	13:59:08
DCP5M-MA-SS-ED-PR-4/24-TC-2-060117	10:03:00	10:03:00	10:03:10	3.3	3.0	14:03:00	14:03:10	9.9	10	14:03:00	14:03:10	3.8	4	14:03:00	14:06:06
DCP5M-MA-SS-ED-PR-4/24-TC-3-060117	10:10:00	10:10:00	10:10:10	2.8	3.0	14:10:00	14:10:10	6.1	5.0	14:10:00	14:10:10	6.5	5	14:10:00	14:13:21
DCP5M-MA-SS-ED-PR-4/24-PB-1-060117	----	10:17:00	10:17:10	2.2	2.0	14:17:00	14:17:10	7.2	6.0	14:17:00	14:17:10	7.3	6	14:17:00	14:20:03

**Table A-7. Experimental parameters for carbaryl decontamination with EasyDECON® DF200 (single-step procedure)**

Test and Sample ID	Contamination	Decontamination Step				Water Rinse				Wipe sampling	
	Start Time	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time
	(hh:mm:ss)		(g)		(hh:mm:ss)		(g)		(hh:mm:ss)		
Single-step DCP 1 Hand-held pressurized sprayer											
DCP1S-CA-SS-ED-PS-1-TC-1-250117	10:10:00	10:10:00	10:10:10	20	21	11:10:00	11:10:10	20	23	11:10:00	11:13:08
DCP1S-CA-SS-ED-PS-1-TC-2-250117	10:20:00	10:20:00	10:20:12	20	22	11:20:00	11:20:13	20	20	11:20:00	11:23:08
DCP1S-CA-SS-ED-PS-1-TC-3-250117	10:30:00	10:30:00	10:30:13	20	22	11:30:00	11:30:10	20	21	11:30:00	11:33:03
Single-step DCP 2 Hand-held pressurized sprayer											
DCP2S-CA-SS-ED-SB-1-TC-1-250117	10:40:00	10:40:00	10:40:19	20.0	19	11:40:00	11:40:20	19.5	20	11:40:00	11:43:04
DCP2S-CA-SS-ED-SB-1-TC-2-250117	10:50:00	10:50:00	10:50:18	20.6	19	11:50:00	11:50:13	19.9	20	11:50:00	11:53:03
DCP2S-CA-SS-ED-SB-1-TC-3-250117	11:00:00	11:00:00	11:00:24	19.4	19	12:00:00	12:00:13	19.7	20	12:00:00	12:02:59
Single-step DCP 3 Cleaning cloth											
DCP3S-CA-SS-ED-RG-1-TC-1-010217	10:24:00	10:24:00	10:24:10	8.5	7.0	11:24:00	11:24:10	1.9	1	11:24:00	11:27:02
DCP3S-CA-SS-ED-RG-1-TC-2-010217	10:31:00	10:31:00	10:31:10	9.3	8.0	11:31:00	11:31:10	2.2	1	11:31:00	11:33:57
DCP3S-CA-SS-ED-RG-1-TC-3-010217	10:38:00	10:38:00	10:38:10	9.0	7.0	11:38:00	11:38:10	2.5	1	11:38:00	11:40:40
Single-step DCP 4 Cleaning sponge											
DCP4S-CA-SS-ED-SP-1-TC-1-010217	10:45:00	10:45:00	10:45:10	49.1	47	11:45:00	11:45:10	18.6	17	11:45:00	11:47:45
DCP4S-CA-SS-ED-SP-1-TC-2-010217	10:52:00	10:52:00	10:52:10	31.7	29	11:52:00	11:52:10	22.0	21	11:52:00	11:54:35
DCP4S-CA-SS-ED-SP-1-TC-3-010217	10:59:00	10:59:00	10:59:10	38.4	35	11:59:00	11:59:10	27.7	26	11:59:00	12:01:45
Single-step DCP 5 Paint roller											
DCP5S-CA-SS-ED-PR-1-TC-1-010217	11:06:00	11:06:00	11:06:10	19.0*	19*	12:06:00	12:06:10	9.2	8	12:06:00	12:08:38
DCP5S-CA-SS-ED-PR-1-TC-2-010217	11:13:00	11:13:00	11:13:10	3.5	3.0	12:13:00	12:13:10	16.1	15	12:13:00	12:15:40
DCP5S-CA-SS-ED-PR-1-TC-3-010217	11:20:00	11:20:00	11:20:10	3.9	3.0	12:20:00	12:20:10	20.6	19	12:20:00	12:22:50

\*No analytical notes on reason for the high loading; the outlier excluded from average loading calculations

**Table A-8. Experimental parameters for carbaryl decontamination with EasyDECON® DF200 (multistep procedure)**

Sample ID	Contamination	Decontamination Step 1				Decontamination Step 2				Water Rinse				Wipe sampling	
	Start Time	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time	Amount Applied	Waste collected	Start Time	Stop Time
	(hh:mm:ss)	(hh:mm:ss)	(hh:mm:ss)	(g)	(g)	(hh:mm:ss)	(hh:mm:ss)	(g)	(g)	(hh:mm:ss)	(hh:mm:ss)	(g)	(g)	(hh:mm:ss)	(hh:mm:ss)
<b>Multistep DCP 1 Hand-held pressurized sprayer</b>															
DCP1M-CA-SS-ED-PS-4/24-TC-1-260117	8:50:00	8:50:00	8:50:13	20	26	12:50:00	12:50:10	20	22	12:50:00	12:50:10	20	22	12:50:00	12:53:00
DCP1M-CA-SS-ED-PS-4/24-TC-2-260117	9:00:00	9:00:00	9:00:12	20	31	13:00:00	13:00:10	20	23	13:00:00	13:00:08	20	23	13:00:00	13:02:50
DCP1M-CA-SS-ED-PS-4/24-TC-3-260117	9:10:00	9:10:00	9:10:15	20	31	13:10:00	13:10:10	20	23	13:10:00	13:10:09	20	23	13:10:00	13:12:43
DCP1M-CA-SS-ED-PS-4/24-PB-1-260117	----	9:20:00	9:20:11	20	31	13:20:00	13:20:10	20	23	13:20:00	13:20:10	20	23	13:20:00	13:22:45
<b>Multistep DCP 2 Spray bottle</b>															
DCP2M-CA-SS-ED-SB-4/24-TC-1-260117	9:30:00	9:30:00	9:30:35	22.5	31	13:30:00	13:30:35	21.0	21	13:30:00	13:30:35	19.6	21	13:30:00	13:32:43
DCP2M-CA-SS-ED-SB-4/24-TC-2-260117	9:40:00	9:40:00	9:40:37	20.9	20	13:40:00	13:40:35	19.6	19	13:40:00	13:40:31	20.4	19	13:40:00	13:42:53
DCP2M-CA-SS-ED-SB-4/24-TC-3-260117	9:50:00	9:50:00	9:50:35	22.9	19	13:50:00	13:50:38	20.0	19	13:50:00	13:50:38	20.3	19	13:50:00	13:52:48
DCP2M-CA-SS-ED-SB-4/24-PB-1-260117	----	10:00:00	10:00:28	19.8	21	14:00:00	14:00:30	19.9	19	14:00:00	14:00:45	21.9	19	14:00:00	14:02:44
<b>Multistep DCP 3 Cleaning cloth</b>															
DCP3M-CA-SS-ED-RG-4/24-TC-1-020217	9:00:00	9:00:00	9:00:10	8.4	6.0	13:00:00	13:00:10	8.0	6.0	13:00:00	13:00:10	3.2	3	13:00:00	13:02:45
DCP3M-CA-SS-ED-RG-4/24-TC-2-020217	9:07:00	9:07:00	9:07:10	6.6	5.0	13:07:00	13:07:10	8.1	7.0	13:07:00	13:07:10	2.0	1	13:07:00	13:09:43
DCP3M-CA-SS-ED-RG-4/24-TC-3-020217	9:14:00	9:14:00	9:14:10	8.2	6.0	13:14:00	13:14:10	8.8	6.0	13:14:00	13:14:10	2.1	1	13:14:00	13:16:48
DCP3M-CA-SS-ED-RG-4/24-PB-1-020217	----	9:21:00	9:21:10	7.4	5.0	13:21:00	13:21:10	8.3	7.0	13:21:00	13:21:10	2.0	0	13:21:00	13:23:51
<b>Multistep DCP 4 Cleaning sponge</b>															
DCP4M-CA-SS-ED-SP-4/24-TC-1-020217	9:28:00	9:28:00	9:28:10	24.5	21	13:28:00	13:28:10	36.9	35	13:28:00	13:28:10	19.9	19	13:28:00	13:30:40
DCP4M-CA-SS-ED-SP-4/24-TC-2-020217	9:35:00	9:35:00	9:35:10	27.2	25	13:35:00	13:35:10	17.3	14	13:35:00	13:35:10	12.1	11	13:35:00	13:37:50
DCP4M-CA-SS-ED-SP-4/24-TC-3-020217	9:42:00	9:42:00	9:42:10	40.6	38	13:42:00	13:42:10	17.0	13	13:42:00	13:42:10	11.5	11	13:42:00	13:44:39
DCP4M-CA-SS-ED-SP-4/24-PB-1-020217	----	9:49:00	9:49:10	30.6	27	13:49:00	13:49:10	38.0	34	13:49:00	13:49:10	11.8	12	13:49:00	13:51:35
<b>Multistep DCP 5 Paint roller</b>															
DCP5M-CA-SS-ED-PR-4/24-TC-1-020217	9:56:00	9:56:00	9:56:10	12.1 *	12	13:56:00	13:56:10	11.4	11	13:56:00	13:56:10	8.5	14	13:56:00	13:58:50
DCP5M-CA-SS-ED-PR-4/24-TC-2-020217	10:03:00	10:03:00	10:03:10	8.2	8.0	14:03:00	14:03:10	17.7	17	14:03:00	14:03:10	10.6	10	14:03:00	14:05:52
DCP5M-CA-SS-ED-PR-4/24-TC-3-020217	10:10:00	10:10:00	10:10:10	9.1	8.0	14:10:00	14:10:10	16.9	17	14:10:00	14:10:10	9.4	9	14:10:00	14:12:35
DCP5M-CA-SS-ED-PR-4/24-PB-1-020217	----	10:17:00	10:17:10	4.6	4.0	14:17:00	14:17:10	13.6	13	14:17:00	14:17:10	7.6	6	14:17:00	14:19:41

\*No analytical notes on reason for the high loading; not an outlier

**Table A-9. Calculated risk-based surface cleanup thresholds for malathion and carbaryl.**

Malathion surface cleanup threshold (noncancer)*		
Surface	(ug/100 cm <sup>2</sup> )	(mg/m <sup>2</sup> )
Child		
Nonporous	2.92	0.29
Porous	14.6	1.5
Adult		
Nonporous	17.0	1.7
Porous	85.1	8.5
Carbaryl surface cleanup threshold (noncancer)*		
Surface	(ug/100 cm <sup>2</sup> )	(mg/m <sup>2</sup> )
Child		
Nonporous	14.6	1.5
Porous	73.1	7.3
Adult		
Nonporous	85.1	8.5
Porous	426	43
*THQ = 0.1; equations for risk-based calculations are below		

**Risk-based surface goal for non-carcinogens:**

Adopted from reference [19]:

$$\text{Risk-based Surface Goal}_N (\mu\text{g} / \text{cm}^2) = \frac{\text{Target hazard quotient}}{\text{Noncancer hazard}_{\text{Oral}} + \text{Noncancer hazard}_{\text{Dermal}}} \quad [\text{A1}]$$

**Noncancer hazard from oral exposure:**

$$\text{Noncancer Hazard}_{\text{Oral}} = \frac{1/RfD_o \times ED \times EF \times MCF \times STF \times UC \times MSA \times MF \times SE \times ET}{BW \times AT_N \times TCF} \quad [\text{A2}]$$

**Noncancer hazard from dermal exposure:**

$$\text{Noncancer Hazard}_{\text{Dermal}} = \frac{1/RfD_D \times ED \times EF \times MCF \times STF \times UC \times CR \times ABS_D \times ET}{BW \times AT_N \times TCF} \quad [\text{A3}]$$

where:

$$RfD_D = RfD_o \times ABS_{GI} \quad [\text{A4}]$$

## Equation Parameters and Values

Parameter	Definition	Exposure Scenario		Reference
		Industrial	Residential	
<b>ED</b>	Exposure duration (years)	25	6 Child 24 Adult	a,b,c
<b>EF</b>	Exposure frequency (days/year)	250	350	a,b,c
<b>MCF</b>	Mass conversion factor (mg/μg)	0.001	0.001	
<b>STF</b>	Skin transfer factor (unitless)	0.25 nonporous 0.05 porous	0.25 nonporous 0.05 porous	d
<b>UC</b>	Unit concentration (μg/cm <sup>2</sup> )	1.0	1.0	
<b>MSA</b>	Mouthing surface area (cm <sup>2</sup> /event)	45	15 Child 45 Adult 39 Age Adj.	d
<b>MF</b>	Mouthing frequency (events/hour)	2	9 Child 2 Adult 3.4 Age Adj.	d
<b>SE</b>	Saliva extraction factor (unitless)	0.5	0.5	d
<b>ET</b>	Exposure time (hours/day)	8	16	a,b,c
<b>BW</b>	Body weight (kg)	80	15 Child 80 Adult 59 Age Adj.	e
<b>AT<sub>N</sub></b>	Average time for noncarcinogens (years)	25	6 Child 24 Adult	a,b,c
<b>TCF</b>	Time conversion factor (days/year)	365	365	a,b,c
<b>CR</b>	Contact rate (cm <sup>2</sup> /hour)	2000	2000	b
<b>ABS<sub>GI</sub></b>	Gastrointestinal absorption factor (unitless)	Chemical-specific	1 (malathion) 1 (carbaryl)	f
<b>ABS<sub>D</sub></b>	Dermal absorption factor (unitless)	Chemical-specific	0.1 (malathion) 0.1 (carbaryl)	f
<b>RfD<sub>O</sub></b>	Oral reference dose (mg/kg-day)	Chemical-specific	0.02 (malathion) 0.1 (carbaryl)	g
<b>RfD<sub>D</sub></b>	Dermal reference dose (mg/kg-day)	Calculated	Calculated	Eq. A4

#### Table References:

- a U.S. Environmental Protection Agency (USEPA), 1991, Risk Assessment Guidance for Superfund, Vol. 1: Human Health Evaluation Manual, Part B, Development of Risk-Based Preliminary Remediation Goals. Office of Emergency and Remedial Response, Washington, DC. EPA/540/R-92/003 <https://www.epa.gov/risk/risk-assessment-guidance-superfund-rags-part-b> Last accessed October 19, 2017
- b U.S. Environmental Protection Agency (USEPA), 2004, Risk Assessment Guidance for Superfund, Vol. 1: Human Health Evaluation Manual, Part E, Supplemental Guidance for Dermal Risk Assessment. Office of Superfund Remediation and Technology Innovation, Washington, DC. EPA/540/R/99/005 <https://www.epa.gov/risk/risk-assessment-guidance-superfund-rags-part-e> Last accessed October 19, 2017
- c U.S. Environmental Protection Agency (USEPA), 2002, Supplemental Guidance for Developing Soil Screening Levels for Superfund Sites. Office of Solid Waste and Emergency Response, Washington DC. OSWER 9355.4-24 <https://www.epa.gov/superfund/superfund-soil-screening-guidance> Last accessed October 19, 2017
- d U.S. Environmental Protection Agency (USEPA), 2003, World Trade Center Indoor Environment Assessment: Selecting Contaminants of Potential concern and Setting Health-Based Benchmarks. Prepared by the Contaminants of Potential Concern (COPC) Committee of the World Trade Center Indoor Air Task Force Working Group. [http://www.epa.gov/WTC/copc\\_study.htm](http://www.epa.gov/WTC/copc_study.htm) Last accessed October 19, 2017
- e U.S. Environmental Protection Agency (USEPA), 2011, Exposure Factors Handbook: 2011 Edition, Chapter 8, Body Weight Studies. Office of Research and Development, Washington, DC. EPA/600/R-09/052F <https://cfpub.epa.gov/ncea/risk/recordisplay.cfm?deid=236252> Last accessed October 19, 2017
- f Oak Ridge National Laboratory (ORNL), 2005, Risk Assessment Information System, Oak Ridge National Laboratory, Life Science Division, Oak Ridge, TN. <https://rais.ornl.gov/> Last accessed October 19, 2017
- g U.S. Environmental Protection Agency (USEPA), 2017, Regional Screening Level Summary Tables June 2017. <https://www.epa.gov/risk/regional-screening-levels-rsls> Last accessed October 19, 2017

**Table A-10. Concentration of malathion in cleaning media (solid waste), liquid waste, and post-cleanup surface concentration - all mechanical cleaning media (DCP deployed using water only)**

Cleaning Medium Used	Sponge	Cloth	Paint Roller
Type of Sample	Malathion Concentration per Area Cleaned		
	(mg/m <sup>2</sup> )		
Cleaning media (solid waste)	28	20	<5.4
	43	13	<5.4
	10	<5.4	<5.4
Mean	26.9	12.7	<5.4
±SD	16.17	7.27	NA
%RSD	60%	57%	NA
Liquid waste	4.2	4.5	1.8
	4.5	3.8	1.4
	7.0	5.8	2.7
Mean	5.2	4.7	2.0
±SD	1.5	1.03	0.66
%RSD	29%	22%	33%
Surface concentration post-cleaning*	34	22	44
	30	32	32
	36	34	35
Mean	33	29	37
±SD	3.1	6.6	6.4
%RSD	9.3%	23%	17%
<i>*Cleaning with water only, no decontaminant.</i>			

**Table A-11. Concentration of carbaryl in cleaning media (solid waste), liquid waste, and post-cleanup surface concentration - all mechanical cleaning media (DCP deployed using water-only)**

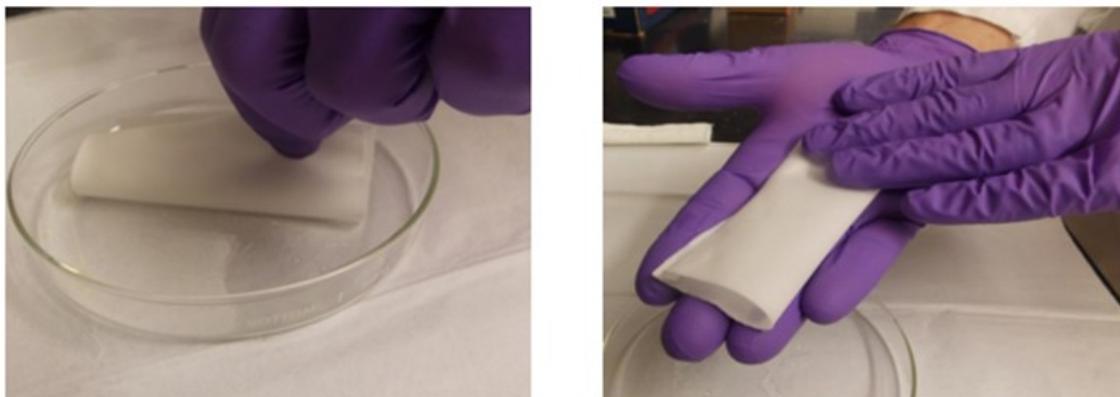
Cleaning Medium Used	Sponge	Cloth	Paint Roller
Type of Sample	Carbaryl Concentration per Area Cleaned		
	(mg/m <sup>2</sup> )		
Cleaning media (solid waste)	81	140	<5.4
	111	32	<5.4
	60	95	<5.4
Mean	84.0	89.2	<5.4
±SD	25.6	54.1	NA
%RSD	31%	61%	NA
Liquid waste	3.6	<0.11	No sample**
	1.5	<0.11	0.75
	2.0	<0.11	0.64
Mean	2.4	<0.11	0.69
±SD	1.1	NA	0.08
%RSD	46%	NA	12%
Surface concentration post-cleaning*	91	100	No sample**
	76	336	83
	115	139	82
Mean	94	190	83
±SD	20	130	0.70
%RSD	21%	66%	0.8%
<i>*Cleaning with water -only, no decontaminant; **Due to sample mislabeling error</i>			

## Appendix B: Wipe Sampling Procedure

The details on the wipe sampling procedure are for a surface that is 12 in × 12 in. Note: Photographs are showing hexane-based wipe sampling; for acetone sampling used in the optimized method in this study, latex gloves were worn over nitrile gloves.

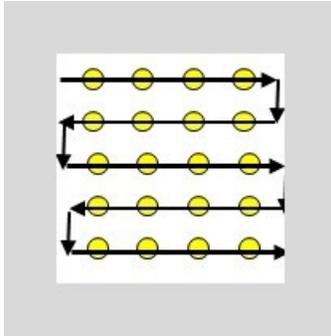
This multistep sampling procedure is summarized below:

1. Prepare sampling wipes:
  - Don disposable nitrile gloves.
  - Using forceps, remove one clean wipe from the storage container and place it on a clean Petri dish.
  - Pipette 3 mL of wetting solvent (IPA, hexane, acetone, etc.) onto the center of the wipe, cover the dish, and allow the solvent to disperse into the wipe material.
  - Proceed immediately to wipe sampling.
2. Don a fresh pair of nitrile gloves.
3. Grasp the wetted decontamination wipe with one hand, and use the other hand to gently fold the wipe (Figure B-1). Do not squeeze the wipe to avoid loss of the wetting solvent.



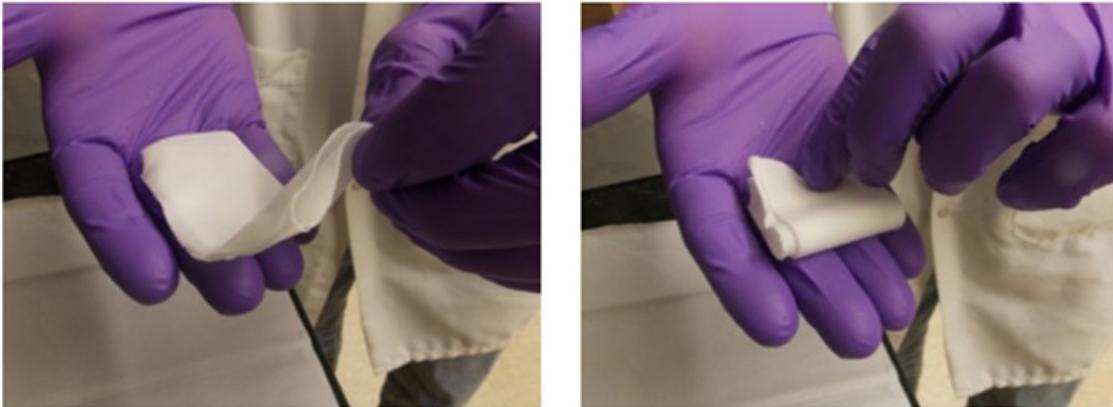
**Figure B-1. Folding wipe for sampling the first wiping pathway (horizontal).**

4. Starting in the top left corner, wipe the surface horizontally, working downward, to cover the surface completely. The horizontal wipe sampling pathway is shown in Figure B-2.



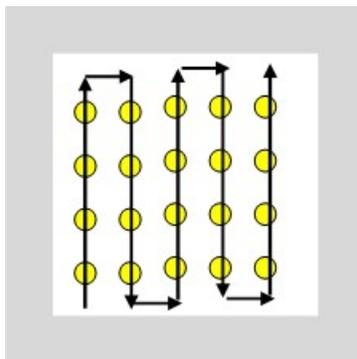
**Figure B-2. Horizontal wiping pathway.**

5. Using both hands, gently refold the wipe so that that the surface used for the horizontal wipe sampling is now on the inside (Figure B-3).



**Figure B-3. Folding wipe for sampling the second wiping pathway (vertical).**

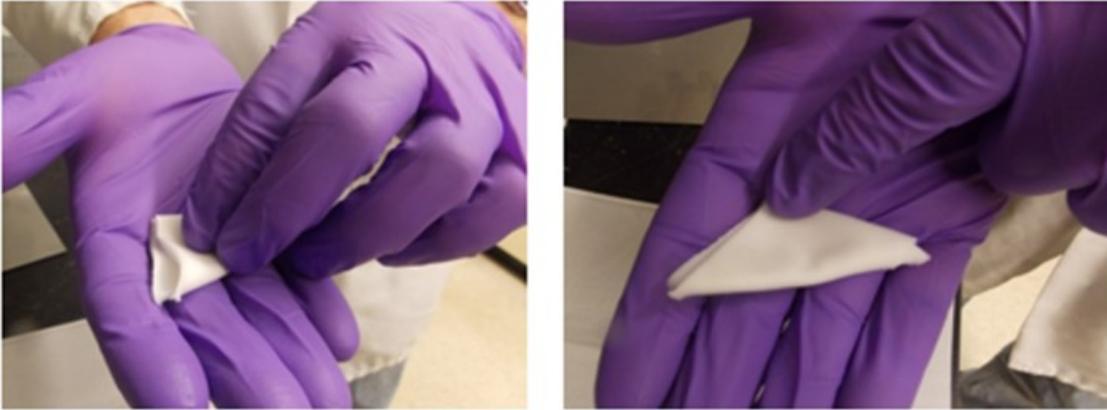
6. Starting in the bottom left corner, wipe the surface vertically, working toward the right, to completely cover the surface. The vertical wipe sampling pathway is shown in Figure B-4.



**Figure B-4. Vertical wiping pathway.**

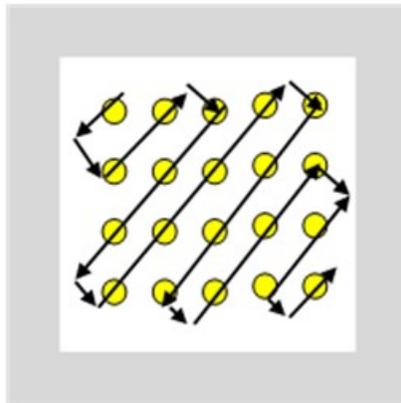
7. Using both hands, gently refold the wipe diagonally, so that that surface used for the vertical

wipe sampling is now on the inside (Figure B-5).



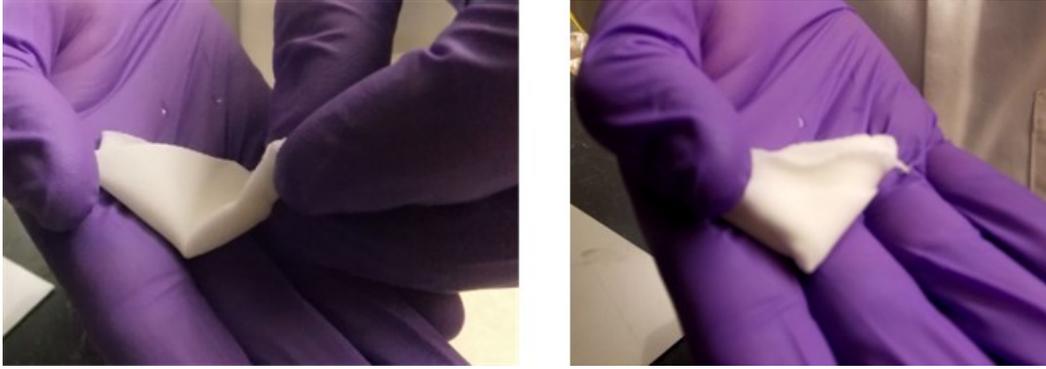
**Figure B-5. Folding wipe for sampling the third wiping pathway (diagonal).**

8. Starting in the top left corner, wipe the surface diagonally, working toward the bottom right corner, to completely cover the surface. The diagonal wipe sampling pathway is shown in Figure 4-6.



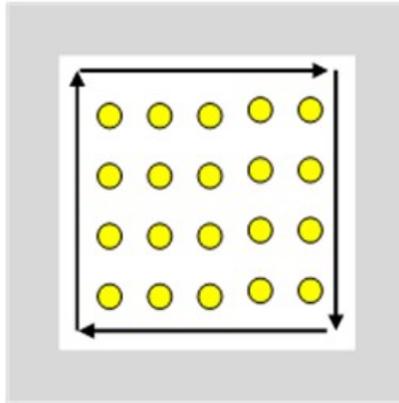
**Figure B-6. Diagonal wiping pathway.**

Using both hands, gently refold the wipe so that that surface used for the diagonal wipe sampling is now on the inside (Figure B-7).



**Figure B-7. Folding wipe for sampling the fourth pathway (perimeter).**

9. Starting in any corner, wipe the perimeter of the coupon. The perimeter wipe sampling pathway is shown in Figure B-8.



**Figure B-8. Perimeter wiping pathway.**

10. Repeat steps 1-9 for repeated wipe sampling of the same surface area.

## Appendix C: Method Development for Liquid Waste Extraction

Liquid waste in this study was extracted using the modified extraction procedure described in EPA Method 3571 (Extraction of Solid and Aqueous Samples for Chemical Agents) [8]. The method-recommended extraction solvent of 10% IPA/dichloromethane was replaced with hexane. Method performance was demonstrated using simulated liquid waste samples (water only, no decontaminant; addition of soap to optimize for detergent-containing samples) spiked with malathion solutions. Two target concentrations (low and high) were tested in these matrix spike samples: 0.05 mg/mL and 5 mg/mL. There were six samples for each target chemical-concentration combination, three with preservative (L-ascorbic acid, ethylenediaminetetraacetic acid) added and three without preservative, accompanied by one PB, total of twelve samples. There was one solvent blank sample (hexane). The preservation-no-preservation test design is below:

1. Six samples, three at 0.05 mg/L and three at 5 mg/L concentration, were preserved with L-ascorbic acid, ethylenediaminetetraacetic acid, and pH-adjusted with the trisodium salt of potassium dihydrogen citrate to pH 3.8 to slow alkaline hydrolysis of malathion. Metabolites of malathion resulting from hydrolysis include malaoxon, malathion alpha and beta monoacid, diethyl fumarate, diethyl thiomalate, O,O-dimethylphosphorodithioic acid, diethylthiomalate, and O,O-dimethylphosphorothionic acid) [9].
2. Preserved samples were each spiked with 30  $\mu$ L of 20x concentrated L-ascorbic acid, ethylenediaminetetraacetic acid, and pH-adjusted with the trisodium salt of potassium dihydrogen citrate solutions. Six samples, three at 0.05 mg/L and three at 5 mg/L concentration, were prepared without preservatives. In this study, liquid preserved and unpreserved samples were extracted with 5 mL of hexane immediately after collection to avoid losses of target chemicals. Hexane extracts were analyzed for malathion via GC/MS.



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