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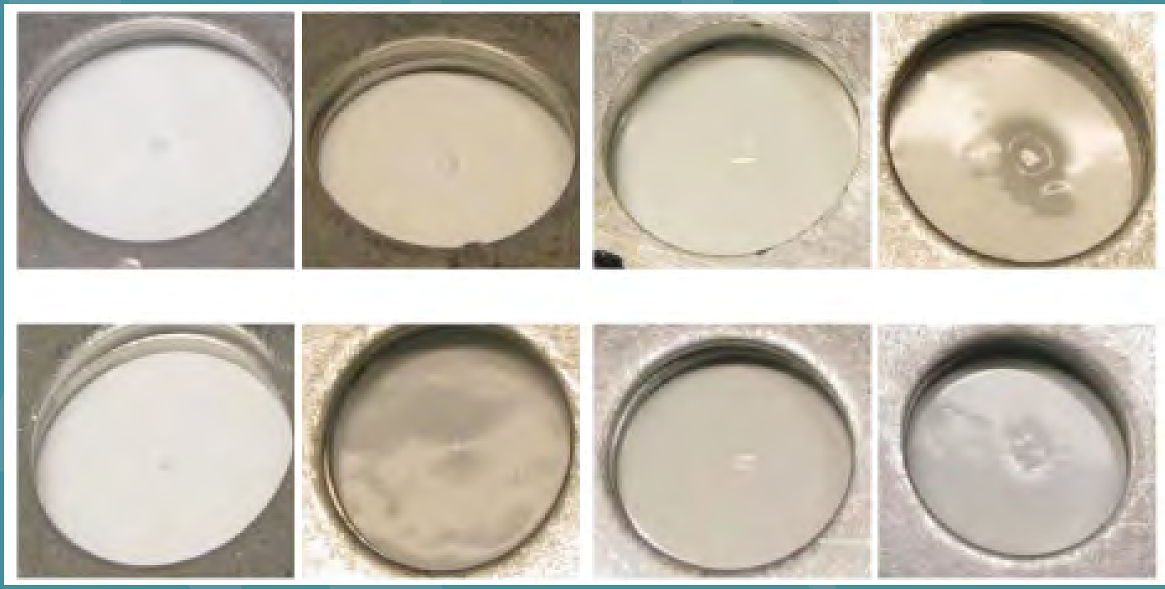
Decontamination Options for Surface Layers Containing Permeated Chemical Warfare Agents HD and VX and Pesticides Malathion and Fipronil



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Decontamination Options for Surface Layers Containing Permeated Chemical Warfare Agents HD and VX and Pesticides Malathion and Fipronil

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DISCLAIMER

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FOREWORD

The U.S. Environmental Protection Agency (EPA) is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. To meet this mandate, EPA's research program is providing data and technical support for solving environmental problems today and building a science knowledge base necessary to manage our ecological resources wisely, understand how pollutants affect our health, and prevent or reduce environmental risks in the future.

The Center for Environmental Solutions and Emergency Response (CESER) within the Office of Research and Development (ORD) conducts applied, stakeholder-driven research and provides responsive technical support to help solve the Nation's environmental challenges. The Center's research focuses on innovative approaches to address environmental challenges associated with the built environment. We develop technologies and decision-support tools to help safeguard public water systems and groundwater, guide sustainable materials management, remediate sites from traditional contamination sources and emerging environmental stressors, and address potential threats from terrorism and natural disasters. CESER collaborates with both public and private sector partners to foster technologies that improve the effectiveness and reduce the cost of compliance, while anticipating emerging problems. We provide technical support to EPA regions and programs, states, tribal nations, and federal partners, and serve as the interagency liaison for EPA in homeland security research and technology. The Center is a leader in providing scientific solutions to protect human health and the environment.

This report assesses various decontamination technologies and strategies to degrade toxic persistent chemicals on the surface of permeable layers (paints and sealants) and any chemical that permeated past the coatings and into underlying porous materials.

Gregory Sayles, Director
Center for Environmental Solutions and Emergency Response

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This research is part of the U.S. Environmental Protection Agency's (EPA's) Homeland Security Research Program's (HSRP) efforts to seek improvements in the decontamination of porous or permeable materials that are contaminated with a persistent chemical warfare agent. This effort was directed by the principal investigator (PI) from the Office of Research and Development's (ORD's) Homeland Security and Materials Management Division (HSMMD) within the Center for Environmental Solutions and Emergency Response (CESER). The contributions of the following individuals have been a valued asset throughout this effort.

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EXECUTIVE SUMMARY

Under the U.S. Environmental Protection Agency's (EPA's) Homeland Security Research Program (HSRP), research is being conducted necessary for identification of methods and technologies that can be used during hazardous materials remediation and cleanup efforts. The threat of a chemical warfare agent (CWA) or pesticide release into the environment is driving EPA's HSRP to systematically evaluate potential decontamination technologies for CWAs and mis- or overused pesticides. The efficacy of many liquid decontaminants has been observed to be material-dependent, attributable in part to the permeability or porosity of the materials to which the decontaminants are applied. Transport of CWA/pesticide into a permeable material often leaves the material more difficult to decontaminate, as water-based decontaminants may not be capable of similar penetration to reach the CWA/pesticide. Adequate decontamination then often becomes even more difficult to achieve if CWA/pesticide permeates into a porous material under a surface film or coating (e.g., paint or sealant).

The purpose here was to evaluate the efficacy of various liquid-based decontamination technologies to degrade CWAs and pesticides on the surface of coating layers (films), within the layer, and into an underlying porous material. Prior to decontamination testing, fate and transport testing was performed for CWAs and pesticides to quantify the amount of each target chemical that remained on the paint or sealant film surface, permeated into (and remained in) the film, and permeated through the film to the porous material during a selected contact time. Decontaminants were then first tested (baseline decontamination condition) to determine which would be efficacious in decontamination of CWAs and pesticides from two freestanding coating layers, paint and sealant. The decontaminants that were initially used for efficacy testing of surfaces contaminated with the CWAs HD and VX included: bleach, Dahlgren Decon, and Decon7 (D7). The decontaminants that were initially used for efficacy testing of surfaces contaminated with the pesticides malathion and fipronil included: 10x diluted bleach and D7.

Following initial baseline decontamination technology testing, two decontaminants (full strength bleach and Dahlgren Decon) were used in additional efficacy testing, with three evaluated decontamination approach modifications. The modified approaches were:

- CWA Decontamination Modification 1: a double (120-minute (min)) decontamination dwell time;
- CWA Decontamination Modification 2: application of 2-butoxyethanol (Chemical A) or Zep® Foaming Wall Cleaner (Chemical B), followed by a water rinse, and 60-minute bleach dwell time; and
- CWA Decontamination Modification 3: application of Chemical A, followed by a water rinse, and 60-minute Dahlgren Decon dwell time.

Selection of Chemicals A and B was based on their use in paint strippers and as household cleaners with stated ability to lift stains from painted surfaces.

For pesticides, one decontaminant (D7) was included in the additional efficacy testing, with two decontamination approach modifications evaluated:

- Pesticide Decontamination Modification 1: 60-minute D7 dwell time, followed by a water rinse, and second 60-minute D7 dwell time.
- Pesticide Decontamination Modification 2: 120-minute D7 time.

The average total decontamination efficacy (combined efficacy based on the sum of chemical mass recoveries from wipe sampling, film extraction, and solid-phase extraction (SPE) disk) measured during CWA testing was low (≤ 50% efficacy) across all the baseline and modification testing for both paint and sealant films; see Table E 1 for a summary of the highest CWA total decontamination efficacies. The highest decontamination efficacy measured for HD-contaminated paint films was demonstrated during Modification 2 testing of bleach with Chemical A (35% average efficacy); the highest decontamination efficacy for HD-contaminated sealant films was demonstrated during Modification 2 testing of bleach with Chemical B (14% average efficacy). The highest decontamination efficacy measured for VX-contaminated paint films was demonstrated during Modification 1 testing with full strength bleach (approximately 6% hypochlorite solution, with 39% average efficacy), and for VX-contaminated sealant films was demonstrated during baseline testing with bleach (50% average efficacy). For comparison, efficacies for decontamination of only the surface ranged from 91% to 99.99% (see Table E 1) indicating that the degradation of these CWAs occurs but is limited to the agent on the surface. The inclusion of Chemical A or B in the decontamination approach for the tested paint and sealant did not assist in the reversed transfer of HD or VX to the surface for degradation of any of the tested decontaminants. The same was absorbed for chemical transferred into the porous sublayer (SPE disk).

Table E 1. Highest HD and VX Average Total Decontamination Efficacy

Analyte	Material	Decontamination Measurement	Decontaminant	Optimum Decontamination Scenario	Average Decontamination Efficacy
HD	Paint	Surface	Bleach	Mod 2 w/ Chemical B	91%
		Total	Bleach	Mod 2 w/ Chemical A	35%
	Sealant	Surface	None	None	0%[1]
		Total	Bleach	Mod 2 w/ Chemical B	14%
VX	Paint	Surface	Bleach	Mod 2 w/ Chemical B	99.9%
		Total	Bleach	Modification 1	39%
	Sealant	Surface	Bleach	Modification 1	99.99%
		Total	Bleach	Baseline	50%

[1] Artificially low because HD was not detected for all positive control wipe samples.

Figure E 1 and Figure E 2 summarize the average total percent decontamination efficacy measured for each test condition during baseline and modification testing for CWA.

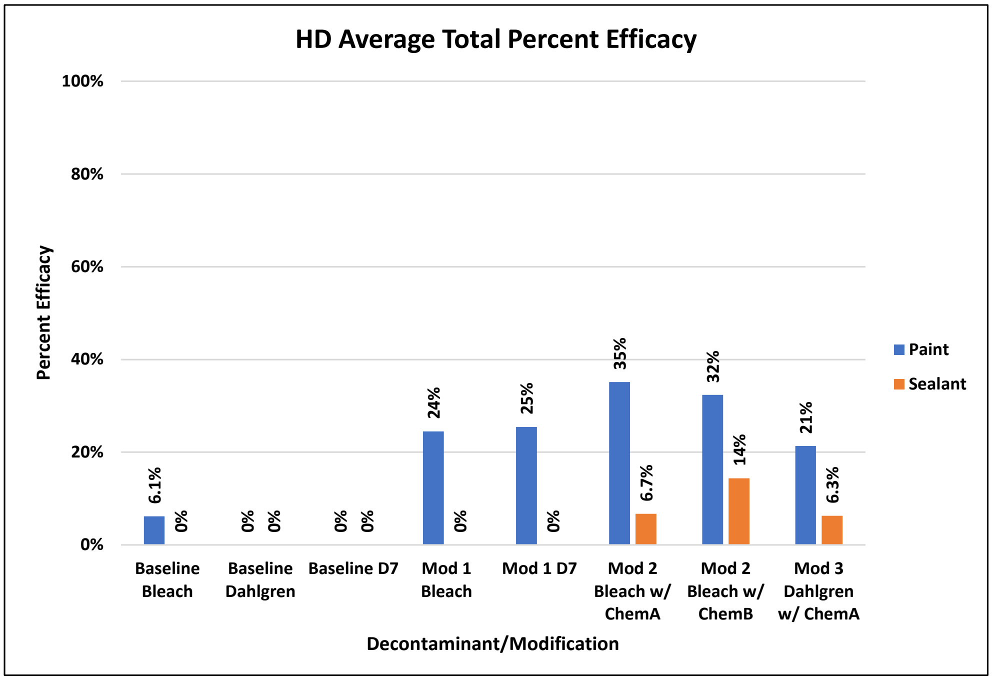

Figure E 1. Average Total Decontamination Efficacy for HD

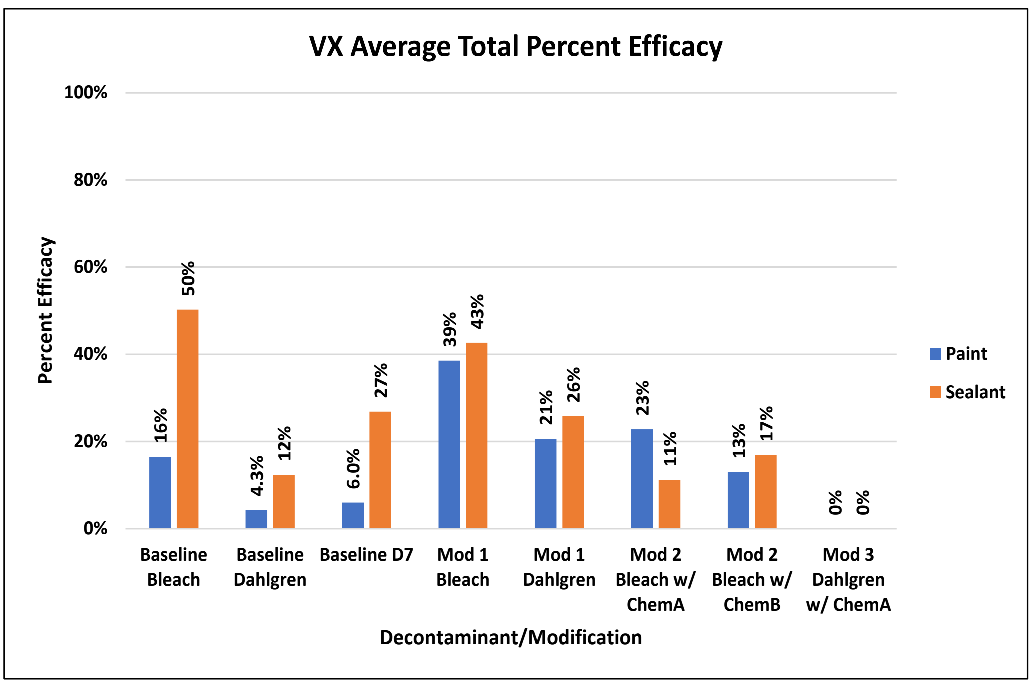

Figure E 2. Average Total Decontamination Efficacy for VX

The average total decontamination efficacies measured during pesticide testing with malathion were low across all the baseline and modification testing for paint films, with the highest efficacy demonstrated during baseline testing using 10x diluted bleach (6.8% average efficacy). The average total decontamination efficacy measured during pesticide testing with malathion were also highest during Modification 1 testing for sealant films using D7 (43% average efficacy). The average total decontamination efficacies measured during pesticide testing with fipronil were mostly high (>90%) across all the baseline and modification testing for both paint and sealant films. The highest decontamination efficacy measured for fipronil-contaminated paint films was demonstrated during Modification 1 testing with D7 (98% average efficacy), and for fipronil contaminated sealant films was demonstrated during baseline testing with 10x diluted bleach (99.1% average efficacy). Fipronil does not transfer appreciably into the paint or sealant layer in comparison to malathion. See Table E 2 for a summary of the highest pesticide surface decontamination efficacy and total decontamination efficacy. As with CWAs, the degradation of these pesticides occurs but is limited to analyte on the surface of the paint or sealant.

Table E 2. Highest Malathion and Fipronil Average Total Decontamination Efficacy

Analyte	Material	Decontamination Measurement	Decontaminant	Optimum Decontamination Scenario	Average Decontamination Efficacy
Malathion	Paint	Surface	None	None	0%[1]
		Total	10x Diluted Bleach	Baseline	6.8%
	Sealant	Surface	D7	Modification 1	>79%
		Total	D7	Modification 1	43%
Fipronil	Paint	Surface	D7	Modification 1	99%
		Total	D7	Modification 1	98%
	Sealant	Surface	10x Diluted Bleach	Baseline	99.3%
		Total	10x Diluted Bleach	Baseline	99.1%

[1] Artificially low because malathion was not detected for all positive control wipe samples.

Figure E 3 and Figure E 4 summarize the average total percent decontamination efficacy measured for each test condition during baseline and modification testing for pesticides.

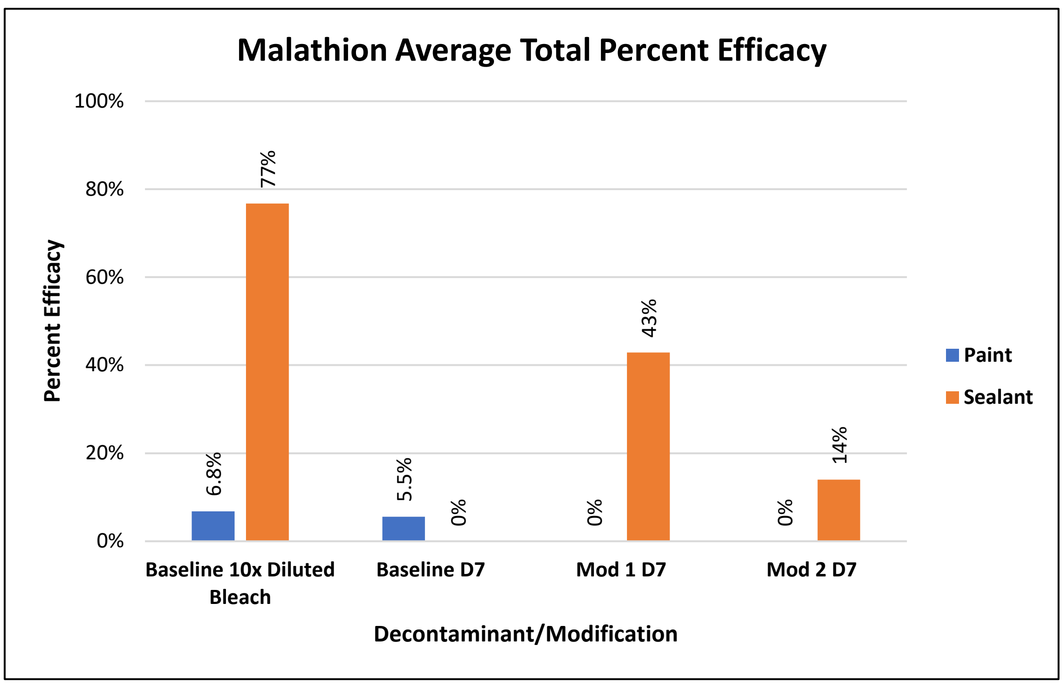

Figure E 3. Average Total Decontamination Efficacy for Malathion

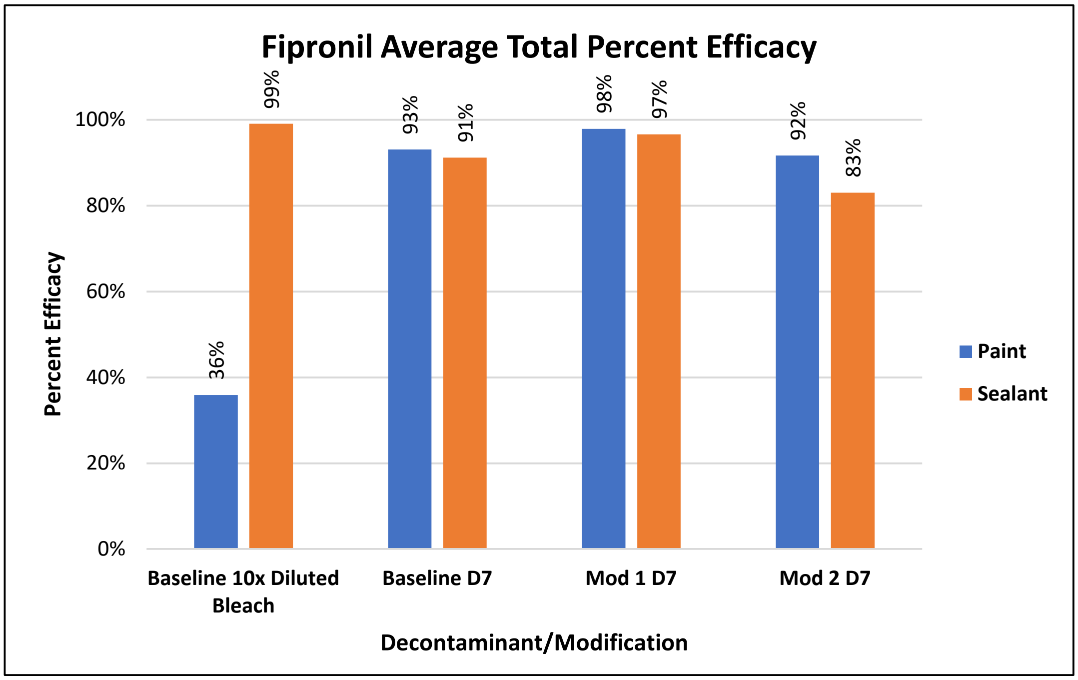

Figure E 4. Average Total Decontamination Efficacy for Fipronil

Efficacy results for the evaluated decontamination products, both during baseline and modification testing indicate that degradation of a chemical agent that had permeated into a paint or sealant remains a challenge. The addition of a household cleaner with stated ability to lift stains from a paint or sealant prior to the decontaminant application did not result in an appreciable improvement in total decontamination efficacy. Following surface decontamination, chemicals that permeated into the paint or sealant layer may eventually resurface over time thereby recreating the hazard.

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LIST OF ACRONYMS AND ABBREVIATIONS

°C	Degree(s) Celsius
°F	Degree(s) Fahrenheit
µg	microgram(s)
µL	microliter(s)
µm	micrometer(s)
AMC	Army Materiel Command
ANOVA	Analysis of Variance
ASTM	American Society for Testing and Materials (ASTM) International
BE	2-butoxyethanol
CAS	Chemical Abstract Services
CCDC	Combat Capabilities Development Command
CCV	Continuing Calibration Verification
CESER	Center for Environmental Solutions and Emergency Response
cm	centimeter(s)
cm^2	square centimeter(s)
CoC	Chain of Custody
CWA	Chemical Warfare Agent
DFTPP	decafluorotriphenylphosphine
EPA	U.S. Environmental Protection Agency
FID	flame ionization detector
FWC	Foaming Wall Cleaner
g	gram
GC/MS	gas chromatography/mass spectrometry
HD	Sulfur Mustard
HMRC	Hazardous Materials Research Center
HPLC	high performance liquid chromatography
HSMMD	Homeland Security and Materials Management Division
HSRP	Homeland Security Research Program
ID	inside diameter
in-lb	inch pound(s)
IPA	isopropyl alcohol
IS	internal standard
LB	laboratory blank
LC-MS/MS	liquid chromatography - tandem mass spectrometry
LLOQ	lower limit of quantitation
LRB	laboratory record book
LVAP	Low Volatility Agent Permeation
M	molar
m^2	square meter(s)
mg	milligram(s)

min	minute(s)
mL	milliliter(s)
mm	millimeter(s)
MQL	method quantification limit
MRM	multiple reaction monitoring
NIST	National Institute of Standards and Testing
OD	outside diameter
OP	organophosphate
ORD	Office of Research and Development (EPA)
PB	procedural blank
PC	positive control
PFPP	pentafluorophenylpropyl
PI	principal investigator
PTFE	polytetrafluoroethylene
QA	quality assurance
QAPP	Quality Assurance Project Plan
r^2	coefficient of determination
RH	relative humidity
rpm	revolution(s) per minute
RSD	relative standard deviation
SC	spike control
SEM	scanning electron microscope
SD	standard deviation
SDS	Safety Data Sheet
SIM	selected ion monitoring
SOP	standard operating procedure
SPE	solid phase extraction
STREAMS	Scientific, Technical, Research, Engineering, and Modeling Support
STS	sodium thiosulfate
TPCS	test parameter control sheet
TSA	Technical System Audit
VR	Russian VX; O-(iso-Butyl) S-(2-diethylaminoethyl) methylphosphonothiolate
VX	O-ethyl S-(2-[diisopropylamino]-ethyl) methylphosphonothioate

A. INTRODUCTION

The efficacy of liquid decontaminants for surfaces contaminated with chemical warfare agents (CWAs) or pesticides has been observed in many cases (both experimentally and during field use) to be material dependent, attributable in part to the permeability or porosity of the materials to which the decontaminants are applied. Liquid decontaminants are often water-based, and many are capable of high efficacy if applied to CWA contamination on nonporous materials. However, transfer of CWA into a permeable material often leaves the material more difficult to decontaminate, as water-based decontaminants may not be capable of similar penetration to reach the permeated CWA. Adequate decontamination generally becomes even more difficult to achieve if the CWA permeates into a porous material underneath a film or other coating on the surface (e.g., paint or sealant). A CWA permeated into porous materials may also resurface sometime after surface decontamination has taken place, recreating a potential hazard. Decontamination technologies/approaches that can reach and degrade CWA contamination that has permeated through surface coatings and into porous subsurfaces are therefore needed, and this need has been identified by United States Environmental Protection Agency (EPA) as a high priority research gap.

Testing conducted previously [1] to study CWA fate and transport characteristics utilized a layered test sample system consisting of a freestanding film of a selected paint or sealant placed over a solid-phase extraction (SPE) disk to simulate a permeable coating on a porous substrate (e.g., painted wood or sealed concrete). Representative sample setups referred to as low volatility agent permeation (LVAP) assemblies were designed for those studies. This approach allows to readily separate the paint or sealant from the substrate below without impacting the CWA mass distribution. The freestanding film was contaminated with either sulfur mustard (bis(2-chloroethyl) sulfide; HD) or O-ethyl S-(2-[diisopropylamino]-ethyl) methylphosphonothioate (VX), the CWA was allowed to permeate the film for a predetermined time period, and the LVAP assembly components (film surface, film, and SPE disk) were subsequently sampled via wipe collection or solvent extraction to detect and quantify HD or VX in each component. Results indicated that the CWAs absorbed into the permeable surface coatings and transfer into the underlying porous SPE disks.

A.1 Project Objectives

Testing conducted during this study utilized methods based on methods developed during previous studies [1]. Objectives of this study were twofold:

- Assess the efficacy of selected decontamination technologies applied as liquids to degrade CWA or pesticide contamination on the surface of coating layers, as well as CWA or pesticide that has permeated through the coatings and into underlying porous materials; and

- Evaluate modifications to the selected decontaminants and/or decontaminant application procedures to improve the initially measured efficacy.

Fate and transport testing was performed according to procedures developed during earlier studies with HD and VX while using a modified LVAP assembly (refer to Section C.3). HD in film wipe samples and extracts of films and SPE disks was quantified using gas chromatography/mass spectrometry (GC/MS). VX was quantified in wipe and LVAP component extracts using liquid chromatography – tandem mass spectrometry (LC-MS/MS).

Following fate and transport testing, a decontamination step was incorporated into the test procedure, and any changes in CWA amounts recovered from the film surface (via wipe sampling), the permeable film itself (via solvent extraction), and the underlying porous material (SPE disk, via solvent extraction) were measured to assess decontamination efficacy. Modifications to the decontamination step were made to attempt to improve the initially measured efficacy of the tested decontaminants. Demonstration of measured improvements in decontamination efficacy of the test decontaminants when applied to contaminated permeable materials was the main focus of the testing conducted in this work.

Testing included the persistent CWAs HD and VX, as well as selected pesticides reported to be misused in situations leading to remedial action, including malathion and fipronil. Malathion is an organophosphate (OP)-based insecticide widely used in agriculture, outdoor pest control, and residential landscaping. Fipronil is a broad-spectrum insecticide that belongs to the phenylpyrazole family.

A.2 Quality Objectives and Criteria

This work was performed under a Quality Assurance Project Plan (QAPP) [2].

The quality objectives and performance criteria described in the QAPP provide the requirements for determining the adequacy of data generated during this project. Methods were considered acceptable and valid data were assumed if the quality objectives for the test measurements were met, and the Technical System Audit (TSA) and data quality audits show acceptable results. Accuracy was ensured by the calibration of the instruments used during testing, including the GC/MS and LC-MS/MS systems as described in Sections C.7 and C.8, respectively.

The representativeness and uniformity of the test materials were critical attributes to assure reliable test results. For this study, representativeness meant that the freestanding paint/sealant films used in LVAP assemblies were typical of films of the selected paints/sealants commonly encountered in "real world" settings (e.g., painted wood or drywall, or sealed concrete) in terms of quality, surface characteristics, thickness, etc. Uniformity meant that all films (per paint/sealant type) were essentially equivalent for the purposes of testing. Replicate films were obtained using as few "production batches" as possible (refer to Section C.2) so that the replicate films were presumed to have uniform characteristics. Films and SPE disks were visually examined and any with abnormalities on the test surfaces were rejected from use as control or test samples.

A.3 Test Facility Description

All testing was performed at Battelle's Hazardous Materials Research Center (HMRC) located in West Jefferson, Ohio. The HMRC is certified to work with chemical surety materials under a provisioning agreement with oversight by the U.S. Army Materiel Command (AMC; Provisioning Agreement Battelle-1). Wherever applicable and required, the reporting requirements of this agreement were followed.

B. RESEARCH APPROACH

The study objectives were achieved through execution of a series of tests, completed in phases that evaluated:

- Fate and transport of the CWAs HD and VX and the pesticides malathion and fipronil through selected paints and sealants. Fate and transport tests were performed using modified LVAP assemblies. Pesticide fate and transport testing was conducted for 24 hours and 72 hours to ascertain the degree of malathion and fipronil permeation during these times.
- Efficacy of select decontaminants to destroy CWAs/pesticides on the surface of paint and sealant, as well as CWAs/pesticides that have permeated the paint/sealant layers and subsequently migrated into underlying porous materials. Measurement of the mass of CWA/pesticide on the surface of paint/sealant films, within the films, and below the surface of the films (within the underlying SPE disks) were performed both with decontamination (test samples) and without decontamination (positive controls). The efficacy of the decontamination procedures was determined by comparison of the results.
- Modifications (designed and developed based on the initially measured decontamination efficacy) made to the selected decontamination technologies, the decontaminant application methods, or both, to improve efficacy of the decontaminants to degrade CWA/pesticide contamination on the surface of permeable layers as well as CWA/pesticide that has permeated into underlying porous materials.

Prior to fate and transport and decontamination efficacy testing, methods demonstration was performed to ensure:

- Acceptable recovery of CWAs/pesticides from the surface of freestanding films of one (1) selected paint and one (1) selected sealant via wipe sampling.
- Acceptable recovery of CWAs/pesticides that have permeated into freestanding paint/sealant films and SPE disks via solvent extraction.
- Neutralization (quench) of the reactions of tested decontaminants (i.e., decontaminant quench), so that decontamination efficacies could be measured as a function of decontaminant dwell time and valid efficacy data were generated.
- As part of quench testing, no extracted compounds (from wipes, freestanding paint/sealant films, or SPE disks), test decontaminants, or quench agents present in test samples interfere with accurate quantitation of CWAs or pesticides by LC-MS/MS and GC/MS.

During this study, test articles consisted of freestanding films of the paint and sealant selected for testing positioned over (and held in close contact to) SPE disks in the LVAP assemblies. Refer to Section C.2 for more information on the specific paint and sealant that were evaluated during testing, the methods that were used to produce freestanding films of the paint and sealant, and the methods for measurement of the thickness of the freestanding paint/sealant films that were produced. This test sample setup was intended to mimic the characteristics of commonly encountered painted or sealed porous materials (e.g., painted wood or drywall, or sealed concrete). Refer to reference 1 for more information on the design of the LVAP assembly and the arrangement of the assembly components.

C. MATERIALS AND METHODS

C.1 CWAs and Pesticides

HD (Chemical Abstract Services (CAS) # 505-60-2) and VX (CAS # 50782-69-9) used for this testing were synthesized at Battelle's HMRC under Chemical Weapons Convention program guidelines, with accountability through the U.S. AMC. All CWAs originated from the same synthesis lot.

HD and VX purity were determined by dissolving a known mass of the neat CWA into a known volume of solvent (targets of 3,500 µg/milliliter (mL) concentration for HD and 900 µg/mL concentration for VX) and analyzing the samples by GC/flame ionization detector (FID) to determine the relative abundance of HD or VX as determined by peak area and reported as percent purity. Solvent blanks were used to correct for possible solvent contaminants. Measured purities are shown in Table 1. HD and VX purity were > 90% for all testing per QAPP requirements.

Table 1. CWA Purity Sample GC/FID Analysis Method Parameters

CWA	Lot Number	Analysis Date	Purity
HD	C066-2	7/16/2020	100%
	C066-2	10/27/2020	100%
	C066-2	12/15/2020	98.9%
	C066-2	1/11/2021	98.9%
	C066-2	3/22/2021	99.4%
VX	C070-7-1	7/16/2020	94.7%
	C070-7-1	8/19/2020	94.5%
	C070-7-1	11/2/2020	92.4%
	C070-7-2	12/2/2020	93.3%
	C070-7-2	3/25/2021	91.6%

A commercial formulation of malathion (Ortho® Max® Malathion) was purchased for use in testing. This formulation contained 50% malathion (CAS # 121-75-5) and 50% other ingredients; the Ortho Max Malathion Safety Data Sheet (SDS) identified the other ingredients as solvent naphtha (petroleum), CAS # 64742-95-6. A commercial formulation of fipronil (Termidor® SC) was purchased for use in testing. This formulation contained 9.1% fipronil (CAS # 120068-37-3); two other ingredients were listed on the SDS: 0.3 – 1.0% sodium N-methyl-N-oleoyltaurate (CAS # 137-20-2) and <0.1% 1,2-Benzisothiazol-3(2H)-one (CAS # 2634-33-5); the balance of components was not identified on the SDS. The Ortho Max and Termidor SC were stored under ambient laboratory conditions. Termidor SC was kept in its original container; however, the Ortho Max was transferred to a new polypropylene container as the cap on the original container leaked.

During all phases of testing involving deposition of neat CWA onto paint and sealant films, CWA was applied using a 100-µL Hamilton syringe equipped with a repeating dispenser. The dispenser delivered 1/50th of the syringe volume with each actuation, thus each "click" delivered a 2-µL droplet. During wipe sampling and solvent extraction methods demonstration testing, fate and transport testing, and all decontamination efficacy testing conducted during this project, paint and sealant films were each contaminated with a single 2-µL droplet deposited in the middle of the film. Mass contamination targets were thus 2,540 µg of HD and 2,016 µg of VX based on density. These contamination amounts are equivalent to 2.65 and 2.10 g/ meters squared (m^2) surface concentrations, respectively, using the exposed area of the freestanding film in the LVAP assembly (approximately 9.6 cm^2). Actual mass contamination depended on percent purity of the CWAs, as well as the precision and accuracy of the deposition method; the purity was verified experimentally during each test through generation of three spike control samples per test. Spike controls were generated by delivering the same quantity of CWA (2 µL) directly into an extraction jar (i.e., onto the side-surface of the inside of the jar) and dissolution of the CWA in 10 mL of extraction solvent. The three spike controls were prepared at the start, middle, and end of each test, bracketing sample spiking. Following preparation, spike controls were sonicated and aliquoted as described below for wipe, film, and SPE disk extracts. The spike control extract was then analyzed alongside the test and control samples.

A malathion mass contamination of 4.0 µg/centimeters squared (cm^2) and a fipronil contamination density of 1.5 µg/cm^2 were targeted. These values were based on highest observed surface concentrations in two pesticide misuse cases that required remediation. Based on this target contamination mass level and the exposed area of the freestanding film in the LVAP assembly (approximately 9.6 cm^2), a target contamination mass of 38.3 µg of malathion and 14.4 µg of fipronil needed to be applied to the surface of films during testing. Solutions of each pesticide were prepared in distilled water (Crystal Springs, Lakeland, FL) at the highest concentration recommended by each manufacturer. Note that malathion is only very slightly soluble in water [3] and fipronil has very poor solubility in water [4]. To minimize the volume of prepared pesticide solutions, three final volumes were evaluated: 4 L, 500 mL, and 100 mL. Multiple spiking studies were performed with prepared solutions to determine the correct spiking volume of pesticide solution. For fipronil, dilution of 1.2 mL of Termidor SC into a final volume of 100 mL of water, and a 12-µL volume application to a surface resulted in an average mass of 12.6 µg of fipronil (88% of the target). This preparation was selected for all testing with fipronil. For malathion, dilution of 0.78 mL of Ortho Max into a final volume of 100 mL of water, and a 12-µL volume application to a surface resulted in an average mass of 34.8 µg of malathion (91% of target). A calibrated positive displacement pipette was used to apply a single 12 µL droplet in the coupon center for all testing.

As with CWA application operations, three spike controls were prepared during each test to verify the mass of pesticide applied to test and control samples. Fresh pesticide solutions were prepared for each day of testing.

While initial spike determination studies for malathion resulted in accurate and reproducible surface mass loadings, the malathion surface wipe method development study indicated high and variable mass loadings for the three spike controls (326% average recovery and 127% relative standard deviation (RSD)). The preparation of the Ortho Max solution was then revisited. When prepared, the Ortho Max solution was white and cloudy, and it was noted that after being allowed to sit overnight, a white precipitate formed (see Figure 1). The cloudiness of the preparation may be due to the naphtha, which has very low water solubility.

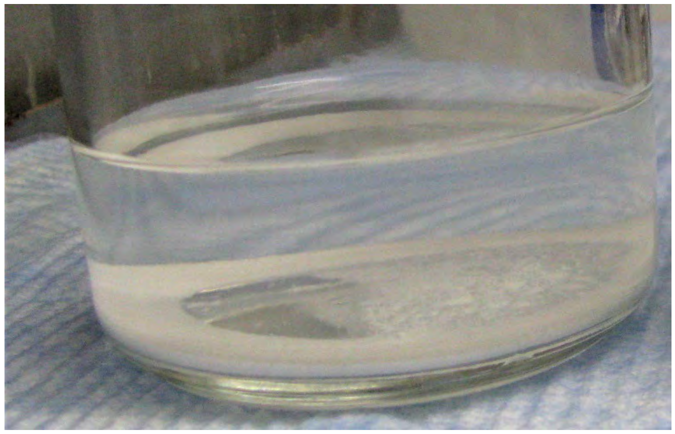

Figure 1. Ortho Max Solution After 1 Day

Based on the observation of this precipitate, the Ortho Max solution was stirred with a stir bar prior to spiking samples, and continued to be stirred while spiking samples, to help ensure a uniform mixture. To evaluate this stirring approach, a second spiking study was performed with three separate Ortho Max solution preparations. The average malathion recoveries for these three solutions were 91%, 88%, and 88% with RSDs of 19%, 5.4%, and 19%, respectively. Based on these results, continuous stirring of the solution was implemented. Additionally, the solution was stirred for at least 1 hour after preparation prior to spiking samples.

C.2 Freestanding Paint and Sealant Films

The permeable surface coatings that were evaluated during this testing were:

- Behr® Premium Plus Low Odor, Paint and Primer in One Semi-Gloss Enamel
- Rust-Oleum® 6711 System Waterborne Oil-Modified Polyurethane Floor Coating.

Acrylic enamel is a low-odor coating appropriate for application onto brick, drywall, masonry, plaster, stucco, vinyl, and wood, and the acrylic enamel is less permeable to some solvents, resulting in easier cleaning in its intended application (the permeability of this coating to the CWAs and pesticides is not known). Polyurethane coating/sealant is generally considered to be highly flexible and elastic, making it suitable for application onto high traffic areas. See Table 2 for additional information.

Table 2. Paint and Sealant Information

Coating	Product	Part No.	Vendor, Location
Paint	Behr® Premium Plus Low Odor, Paint and Primer in One Semi-Gloss Enamel (Ultra-Pure White), 100% acrylic	202761530	Home Depot, Atlanta, GA
Sealant	Rust-Oleum® 6711 System Waterborne Oil-Modified Polyurethane Floor Coating (Clear)	4MG61	Grainger, Lake Forest, IL

Quality and uniformity of free films, both paint and sealant, are highly dependent on the rheological behavior of the liquid, the application method, and the release substrate to which they are applied. Freestanding films of the paint and sealant shown in Table 2 were prepared using ASTM International (ASTM) method D823 "Standard Practices for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels" [5]. A Universal Blade Applicator (AP-G08, Paul N. Gardner Company, Pompano Beach, FL) was used.

Each gallon of paint/sealant was placed on a standard paint shaker (Northern Tool, Burnsville, MN) for 10 minutes to ensure a homogenous mix. Due to the excessive air entrapped in the coatings during this process, a de-gas step was also used. Small batches of approximately 80 grams (g) were de-gassed in a Speed Mixer™ (FlackTeck, Louisville, CO) for 3 minutes at 3,000 revolutions per minute (rpm) to remove entrapped air.

A polytetrafluoroethylene (PTFE) (Teflon®) (Chemfab CF1, Saint-Gobain, Elk Grove, IL) coated fabric was selected as the release substrate for the Behr® semi-gloss enamel and a polyethylene film (Silthene, Siliconature, Caledonia, MI) was selected as the release substrate for the Rust-Oleum® polyurethane coating. Additional substrate surface preparation techniques in the form of plasma treatment (to promote good surface wetting and film quality during the drying process) were required. An AtomFlo 500 (Surfx, Redondo Beach, CA) plasma treater using helium and oxygen plasma gases at 160 watts of power was used.

Following preparation, all freestanding films were visually examined and any areas with obvious abnormalities on the test surface, such as bubbles, pinholes, or visible contaminants in the paint were rejected from use as test samples. Thickness of the freestanding paint and sealant films was measured using an eddy current gauge (PosiTector® 6000, DeFelsko Corporation, Ogdensburg, NY) according to ASTM method E376 "Standard Practice for Measuring Coating Thickness by Magnetic-Field or Eddy-Current (Electromagnetic) Test Methods" [6]. Thickness was measured in each of six (6) unique areas on each film sheet, as identified in Figure 2.

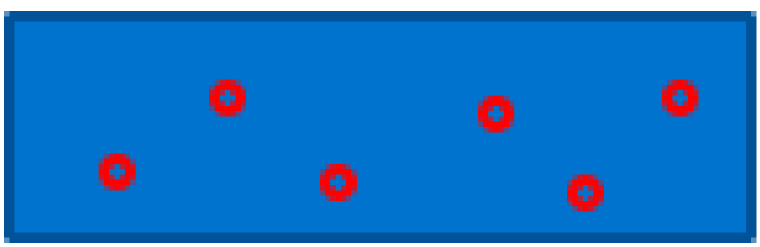

Figure 2. Film Thickness Measurement Locations

Individual coupons were cut from the film sheets using a 4.5-centimeter (cm) diameter die, then allowed to cure for a minimum of 14 days at constant temperature (24 °C) and relative humidity (50% RH). Table 3 and Table 4 provide the thickness measurements collected for each of the freestanding films prepared, and the number of coupons collected from each film. The thickness across each sheet was quite consistent, typically < 6% RSD. Average film thickness was also consistent across sheets: 5.3% RSD for paint sheets and 4.3% RSD for sealant sheets. A total of 382 paint coupons and 336 sealant coupons were prepared. Note that coupons were cut from

Sheets B1 and B5 on 1/21/21, approximately 6 months after other coupons used in testing. These two sheets had been held at 24 °C and 50% RH prior to cutting. The "new" coupons were used during malathion and fipronil developmental testing. Paint and sealant films were inspected visually prior to testing to ensure no coupons with surface anomalies were used. As discussed below, no notable differences in performance of these sheets were obvious in the data.

Table 3. Prepared Paint Films and Measured Thickness

Sheet ID	Draw Down Date	Wet Film Thickness (mils)	Dry Film Thickness (mils)								No. Coupons Collected
			1	2	3	4	5	6	Average	RSD	
B1	6/5/2020	10	2.27	2.29	2.13	2.26	2.41	2.20	2.3	3.9%	29
B2	6/5/2020	10	2.25	2.30	2.31	2.18	2.33	2.39	2.3	3.0%	Uncut
B3	6/5/2020	10	2.15	2.12	2.19	2.39	2.21	2.25	2.2	4.5%	Uncut
B4	6/5/2020	10	2.21	2.25	2.33	2.25	2.23	2.18	2.2	2.3%	Uncut
B5	6/8/2020	10	2.32	2.20	2.40	2.26	2.32	2.23	2.3	3.0%	34
B6	6/8/2020	10	2.39	2.23	2.55	2.19	2.35	2.20	2.3	6.1%	Uncut
B7	6/9/2020	10	2.53	2.47	2.30	2.27	2.50	2.45	2.4	4.6%	Uncut
B8	6/9/2020	10	2.51	2.38	2.47	2.48	2.69	2.32	2.5	5.2%	Uncut
B9	6/9/2020	10	2.74	2.53	2.82	2.56	2.59	2.62	2.6	4.2%	Uncut
B10	6/10/2020	10	2.56	2.43	2.52	2.80	2.75	2.43	2.6	6.2%	34
B11	6/10/2020	10	2.48	2.36	2.60	2.48	2.57	2.63	2.5	4.0%	42
B12	6/10/2020	10	2.35	2.38	2.40	2.30	2.53	2.58	2.4	4.6%	48
B13	6/10/2020	10	2.23	2.37	2.24	2.30	2.31	2.27	2.3	2.2%	32
B14	6/10/2020	10	2.20	2.25	2.23	2.52	2.31	2.28	2.3	5.2%	29
B15	6/10/2020	10	2.21	2.48	2.65	2.59	2.38	2.42	2.5	6.4%	45
B16	6/10/2020	10	2.43	2.52	2.63	2.59	2.62	2.42	2.5	3.6%	45
B17	6/10/2020	10	2.56	2.34	2.56	2.36	2.31	2.47	2.4	4.6%	44

Table 4. Prepared Sealant Films and Measured Thickness

Sheet ID	Draw Down Date	Wet Film Thickness (mils)	Dry Film Thickness (mils)								No. Coupons Collected
			1	2	3	4	5	6	Average	RSD	
R1	6/5/2020	10	2.51	2.68	2.58	2.53	2.45	2.56	2.6	3.1%	33
R2	6/5/2020	10	2.23	2.19	2.36	2.25	2.27	2.29	2.3	2.6%	43
R3	6/5/2020	10	2.47	2.28	2.23	2.54	2.49	2.61	2.4	6.3%	38
R4	6/5/2020	10	2.57	2.42	2.44	2.63	2.51	2.48	2.5	3.2%	38
R5	6/5/2020	10	2.20	2.55	2.39	2.27	2.17	1.55	2.2	15%	38
R6	6/5/2020	10	2.44	2.23	2.48	2.20	2.53	2.53	2.4	6.3%	36
R7	6/5/2020	10	2.58	2.49	2.36	2.44	2.31	2.45	2.4	4.2%	26
R8	6/8/2020	10	2.28	2.26	2.50	2.27	2.38	2.52	2.4	5.0%	50
R9	6/8/2020	10	2.28	2.31	2.53	2.49	2.35	2.45	2.4	4.2%	34
R10	6/8/2020	10	2.47	2.62	2.50	2.46	2.27	2.62	2.5	5.2%	Uncut
R11	6/8/2020	10	2.55	2.61	2.32	2.24	2.51	2.43	2.4	5.8%	Uncut

Scanning electron microscope (SEM) images were taken of paint and sealant film samples. SEM images were taken in August 2020 (just prior to the start of testing) and again in February 2021 (see Figure 3 and Figure 4). No significant differences were observed in the images taken at the two time points, indicating that the films were not physically changing over time. Closeups of paint and sealant films reveal localized but isolated depressions (2-10 µm in diameter).

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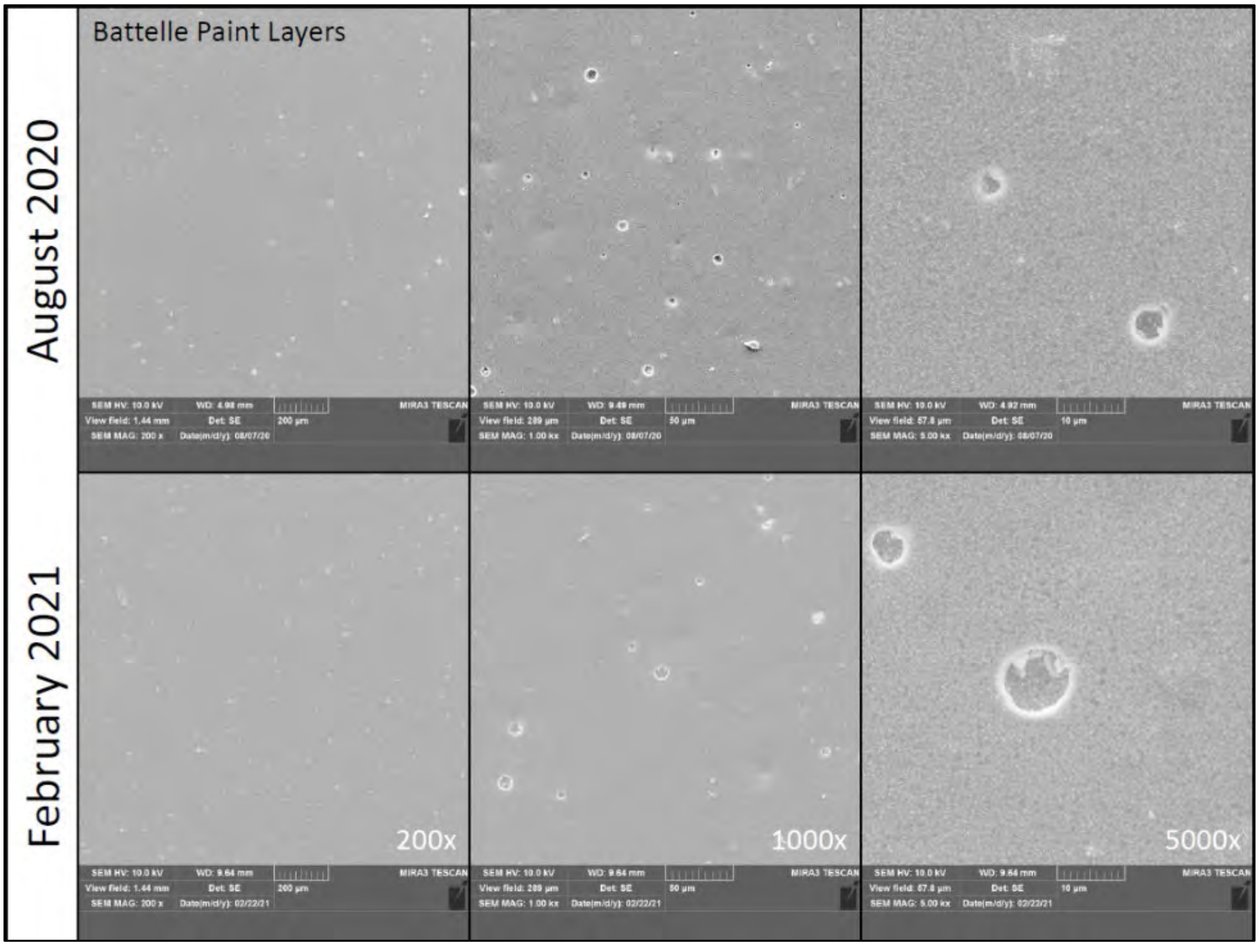


Figure 3. SEM Image of Paint Films at Increased Magnifications (left to right).

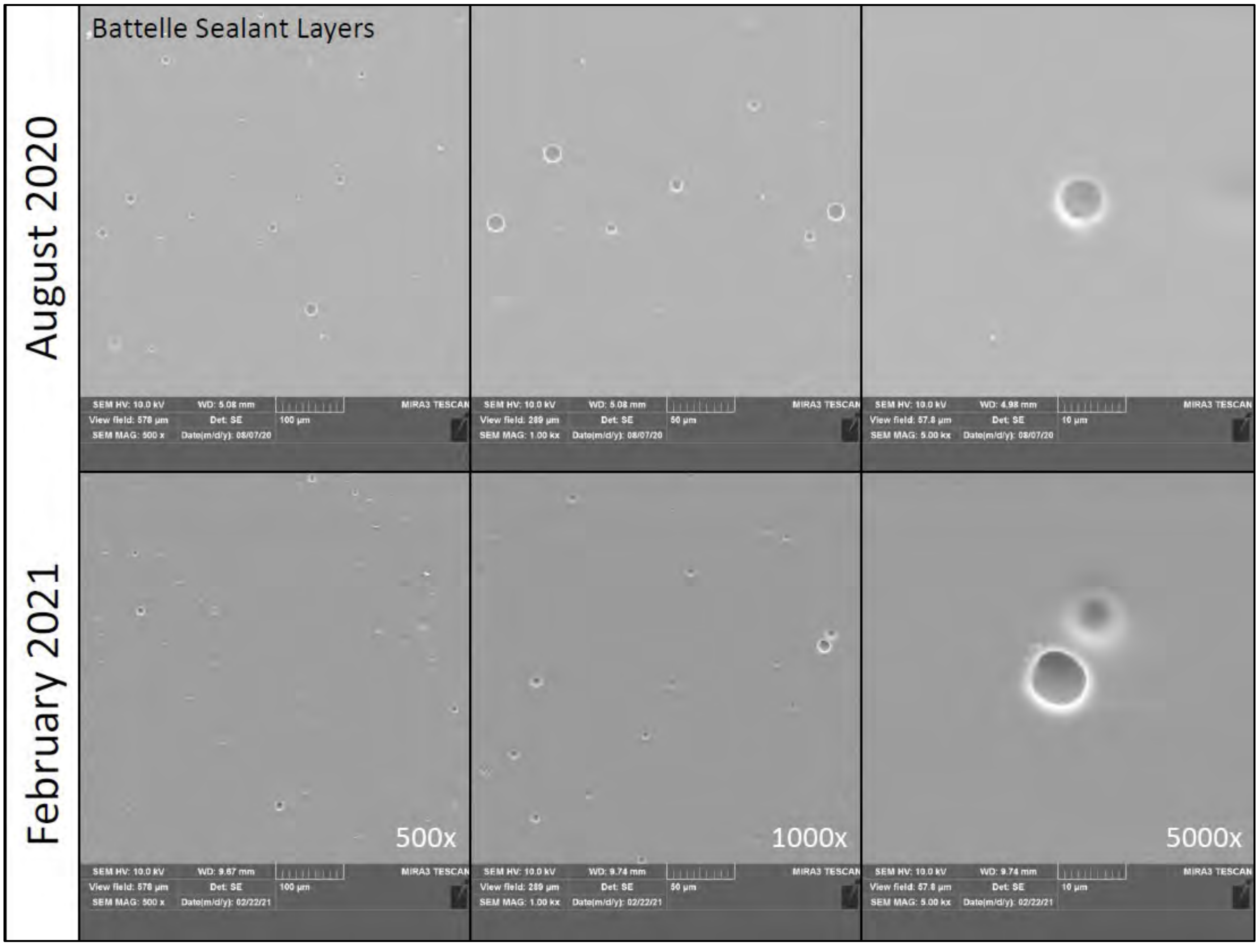


Figure 4. SEM Image of Sealant Films at Increased Magnifications (left to right).

C.3 LVAP Assemblies

The LVAP test methodology was used during previous studies [1] to evaluate fate and transport of HD and VX on, into, and across films of various paints and sealants in contact with SPE disks (to simulate a permeable coating over a porous subsurface). The CWA was spiked onto the surface of paint/sealant films, and as CWA permeated through the films, it was absorbed and retained by the underlying SPE disk. A weight placed on top of the LVAP assembly compressed the assembly components and held the paint/sealant film and SPE disk in close contact.

A modified version of the LVAP arrangement used during previous studies was used for this work that eliminated the need for a weight to be placed on top of the LVAP assembly to compress the assembly components, so that formation of a limited headspace above the CWA/pesticide-contaminated film was avoided. Rather than using a weight, a top compression plate was placed onto bolts attached to a bottom support plate. The bottom support plate was constructed of Type 316 stainless steel and measured 3 inches by 3 inches with ¼-inch thickness. Four 1-inch long ¼-20 thread hex head screws were attached to the bottom support plate at each side. A 5-cm diameter PTFE disk was placed on the bottom support plate centered between the hex screws. As used previously, a CDS Empore™ SDB-XC SPE disk (a poly(styrenedivinylbenzene) copolymer used as a reversed phase sorbent for SPE) was used to simulate a porous subsurface. Each 47-mm diameter SPE disk was die cut to a diameter of 36 mm to provide a contact area of approximately 10 cm^2 beneath the paint/sealant film and the SPE disk was centered on top of the PTFE disk. A latex gasket die cut to 36 mm inside diameter [ID], 51 mm outside diameter [OD], was placed around the SPE disk. A freestanding film coupon (4.5-cm diameter) of the paint or sealant selected for evaluation was then placed directly on top of the SPE disk/latex gasket and held in close contact using a steel washer (1.375-inch ID, 1.875-inch OD, 0.125-inch thickness, type 316 stainless steel). The exposed surface area of the paint/sealant film coupon inside the steel washer was approximately 9.6 cm^2.

The steel washer worked in conjunction with the latex gasket around the SPE disk to isolate the outside edge of the SPE disk and prevent fugitive CWA or pesticide from reaching the SPE disk, mitigating the possibility of false positive results. A top compression was placed on top of the steel washer onto the screws attached to the bottom support plate to compress the assembly and ensure adequate contact between the paint/sealant film and the underlying SPE disk. Nuts threaded onto the screws secured the top compression plate in place. A torque wrench was used to tighten the nuts (target torque of 4 inch-pounds [in-lb]) to ensure consistent compression across the plate. The opening in the top compression plate allowed films to be spiked with the compression plate in place. Figure 5 depicts the step-by-step procedure for loading a freestanding paint film coupon into an LVAP assembly for fate and transport testing.

Figure 5. Paint Film Built into LVAP Assembly

The completed LVAP assemblies were placed in 3.75-inch square (1.5-inch deep) hard clear acrylic boxes during the CWA/pesticide contact period to prevent cross contamination of the films by fugitive CWA/pesticide vapors from other nearby LVAP assemblies. The acrylic boxes were loosely closed so that any CWA/pesticide that evaporated from the surface of the film could slowly and passively escape the box (drawn toward the back of the chemical fume hood in which testing was performed) so buildup of vapor in the local test environment was avoided, and buildup of vapor within the box was avoided. The acrylic boxes used to contain LVAP assemblies were disposed of following each test.

Following application of CWA or pesticide to the film coupon surface, samples remained undisturbed under ambient laboratory conditions. Laboratory temperature and relative humidity were monitored and recorded for the duration of all tests but not controlled. Appendix B provides the starting temperature and RH for each trial.

C.4 Decontamination Technologies

The following three decontaminant technologies were selected for testing:

1. Bleach (Arocep Ultra Bleach, Champion Packaging & Distribution Inc., Woodridge, IL). Commercially available 6% sodium hypochlorite solution. Purchased as 1-gallon containers and used as received.
2. Decon7 (D7), (Part # 7001702, Decon7 Systems, Scottsdale, AZ) is a three-component decontaminant system that is purchased as premeasured components (Part 1, Part 2, Part 3) and mixed in a ratio of 49:49:2. The components include surfactants/inorganic salts (Part 1), hydrogen peroxide (Part 2), and diacetin (Part 3) as a hydrogen peroxide booster.
3. Dahlgren Decon (DD-006-RTU, First Line Technology, Chantilly, VA) is a three-component decontaminant system including water and a surfactant package (Part A), sodium hydroxide (Part B1), and peracetyl borate (active ingredient; Part B2; releases peracetic acid upon dissolution in water). Normally, Part A comes as a solid and must be dissolved in water before mixing with Parts B1 and B2, but for this testing a "ready-to-use" (RTU) version was used that provides Part A already dissolved in water from the manufacturer.

Fresh solutions of Decon7 and Dahlgren Decon were prepared prior to use on each day of testing. The pH and activity of each test decontaminant (following any necessary preparation) were measured prior to use on each day of testing (see Appendix A). The pH of each decontaminant was measured using a calibrated pH meter (Orion Star™ A221 pH portable meter, STARA2210). Hypochlorite concentration for bleach was measured with a Hach model CN-HRDT hypochlorite test kit. Bleach hypochlorite concentration was quite consistent across all testing, ranging from 5.5 – 6.4%. Hypochlorite concentrations for bleach diluted 10-fold were correspondingly lower, ranging from 0.63 – 0.67%. Hydrogen peroxide concentration for D7 was measured with a Hach model HYP-1 hydrogen peroxide test kit. D7 hydrogen peroxide was generally consistent, ranging from 3.9 – 5.8%. However, for the pesticide quench study the hydrogen peroxide activity was only 1.2%. This low reading ultimately did not impact test result interpretation, as it apparently still provided the needed stoichiometric excess of oxidant. The peracetic acid concentration in Dahlgren Decon was measured using a titration approach provided by First Line Technology (see Appendix C for the titration procedure, list of chemicals and materials, and the equation for calculating percent peracetic acid). Dahlgren Decon peracetic acid levels ranged from 6.3 – 11%.

An oil mister/sprayer (CHEFVANTAGE, model# S02-P02-V20-SS-2) was used to apply decontaminant to the surface of paint and sealant film coupons in the LVAP assemblies with an application approach based on the approach used during previous testing [7]. A different sprayer was used for each test decontaminant. New sprayers were used for each test day and then disposed of after one use. The following procedure was used to apply decontaminant:

1. The sprayer was filled to approximately half full (~80 mL) with decontaminant. The sprayer reservoir was marked to indicate the fill level. The sprayer was then pumped twenty times to pressurize.
2. The sprayer was held approximately 10 inches directly above the film coupon in the LVAP assembly. The sash of the test hood was set to a height of 10 inches and the operator used the sash as a guide to ensure the sprayer standoff distance was maintained throughout each application.
3. The sprayer was actuated to begin spray delivery of decontaminant and the sprayer was passed over the film in the LVAP assembly three times (using a "back, forth, back" motion/pattern) to completely cover the film surface. Only one LVAP assembly was sprayed at a time.
4. The sprayer was pumped three additional times after each set of three replicate three-pass applications to maintain pressurization of the sprayer.
5. Before each test, each sprayer filled with decontaminant was checked to verify reproducible spray application (RSD ≤ 15%). Three replicate Teflon disks (1.65" diameter) were sprayed with each decontaminant following the same spray procedure used for the film coupons. The weight of the applied decontaminant on each Teflon disk was recorded, and RSD was calculated.

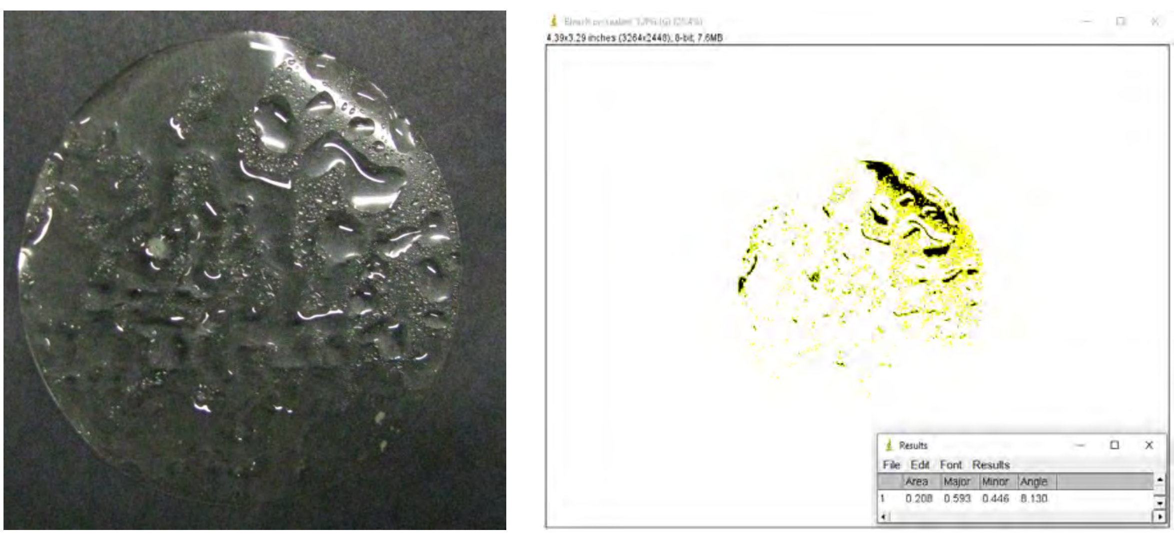
Given the nature of the LVAP assembly, the decontaminant applied to the film coupons remained within the ID of the stainless-steel washer compressed against the paint/sealant film.

Water was used to perform sprayer characterization using five sprayer units. PTFE disks were installed in LVAP assemblies, and the above procedure was followed. Following each three-pass application of water, the PTFE disks were removed from LVAP assemblies and weighed to determine the mass of water delivered to the exposed disk area, which was then converted to volume of water. One LVAP assembly was repeatedly set up with PTFE disks to allow collection of three replicates with a single sprayer (Collection Set 1), after which the sprayer was pumped three times to maintain pressure, and then an additional three replicates were collected (Collection Set 2). Average measured masses for the three replicates are shown in Table 5 along with the mass converted to volume. The average volume of water applied from the five sprayers was 111 µL (13% RSD) for Collection Set 1 and 110 µL (7.9% RSD) for Collection Set 2. These results indicated consistent water application between sprayers and following re-pressurization of the sprayers.

Table 5. Sprayer Characterization Results

Collection Set	Water Delivered	Sprayer 1	Sprayer 2	Sprayer 3	Sprayer 4	Sprayer 5	Average All Sprayers
1	Average Mass (g)	0.1046	0.1026	0.1289	0.0973	0.1236	0.1114
	Average Volume (µL)	105	103	129	97	124	111
	RSD	3.3%	8.1%	6.1%	19%	16%	13%
Pumped three times to maintain pressure							
2	Average Mass (g)	0.1074	0.0972	0.1179	0.1096	0.1182	0.1101
	Average Volume (µL)	107	97	118	110	118	110
	RSD	22%	22%	13%	12%	7.7%	7.9%

Representative photographs of paint and sealant film coupons covered with decontaminant in LVAP assemblies following spray-application were taken and processed using ImageJ image processing software. Only bleach was evaluated in this manner because D7 and Dahlgren Decon visually exhibited uniform coverage. Five pictures of the same sealant coupon were individually evaluated using the ImageJ software. There was some variation based on reflection between each of the pictures. On average, the film coupon was measured to be 88±3% wet (see example photo and ImageJ evaluation in Figure 6). The Figure 6 image evaluation determined 0.208 square inches of dry surface for the 2.40 square inch-coupon or 91% of the coupon being wet.



Bleach on Sealant Coupon Photo

ImageJ Software Evaluation of Photo

Figure 6. Example of the Photo Contrast Evaluation of Bleach on Sealant

Following application, the decontaminants were allowed to remain undisturbed on the surface of films for a predetermined period of time (60 minutes during baseline decontamination efficacy testing). Films were left uncovered in LVAP assemblies during the decontaminant dwell period. Visual observations of the wetness of each film surface at the end of the decontaminant dwell period (prior to wipe sampling) was recorded. Following the decontaminant dwell period, the surface of each film was sampled via wiping according to procedures described in Section C.5.

C.5 Wipe Sampling

The wipe-sampling method used during this testing to recover HD, VX, malathion, and fipronil from the surface of freestanding paint and sealant film coupons was consistent with the method developed during previous studies to recover VX and HD from the surface of films of the same paint and sealant selected for evaluation during this work:

- Lint-free 2-inch by 2-inch, four (4)-ply rayon/polyester blend (gauze) sponges (22-037-921, Fisher Scientific, Pittsburgh, PA) were used.
- Wipes used to sample HD and malathion-contaminated films were wetted with 1.5 mL of hexane. Wipes used to sample VX and fipronil-contaminated films were wetted with 1.5 mL of isopropyl alcohol (IPA). This wetting volume is half of the volume of solvent determined experimentally during previous studies to be necessary to saturate the gauze wipe. Compatibility of the solvents with the paint and sealant films had been demonstrated during previous studies.
- During fate and transport and decontamination efficacy testing, film coupons were wiped while still built into LVAP assemblies, i.e., LVAP assemblies were not disassembled, and films removed prior to wipe sampling.
- Films were wiped using a defined 3-pass wipe pattern including four horizontal strokes, four vertical strokes, and a perimeter stroke. Wipes were folded between passes so that the CWA-/pesticide-exposed surface of the wipe was folded into the wipe. Figure 7 illustrates the wipe sampling pattern that was used.

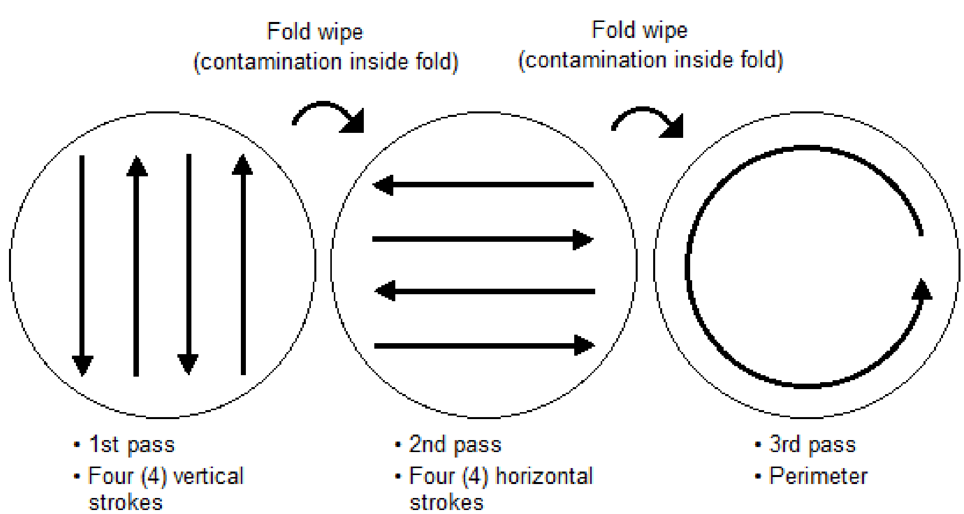


Figure 7. Wipe Sample Collection Pattern

- Given the small surface area of the film coupons inside the stainless-steel washer in the LVAP assemblies, wipe passes were short (film length/width) and partially placed on top of each other.

- During most of the decontamination efficacy testing, no blotting or rinsing of any excess liquid decontaminant remaining on the surface of films was performed. The excess decontaminant was absorbed into the wipe during the wiping action. As discussed in Section D.3, adequate methods for quenching the decontaminant reactions were demonstrated prior to decontamination efficacy testing.

Following collection, wipes were extracted in solvent using the same method used to extract freestanding films and SPE disks, as described in Section C.6, using the same solvents as those used to wet the wipes. Wipe extracts were analyzed via GC/MS (HD and malathion) or LC-MS/MS (VX and fipronil) as described in Sections C.7 and C.8, respectively.

C.6 Solvent Extraction

Recovery of HD, VX, malathion, and fipronil via solvent extraction from wipes, freestanding film coupons of paint and sealant, and SPE disks during this testing was accomplished using the extraction methods developed and used in previous studies to recover HD and VX from the same films and SPE disks selected for this work.

Wipes, film coupons, and SPE disks were extracted by placing each into a separate 60-mL glass jar containing 10 mL of extraction solvent. *n*-Hexane (Fisher Scientific, Part # H306-4) was selected for extraction of HD and malathion from wipes and films; isopropanol (Fisher Scientific, Part # A464-4) was selected for extraction of VX and fipronil from wipes and films; and acetone (Fisher Scientific, Part # A929-4) was selected for extraction of all target chemicals from SPE disks. All solvents were high-performance liquid chromatography (HPLC)-grade or better. Prior to extraction of samples, 5 mL of a 3 molar (M) solution of sodium thiosulfate (STS, Fisher Scientific, Part # S446-500) in water was included in each jar with the solvent as a quench for the decontaminants. Wipes, film coupons, and SPE disks were able to lie flat within the inside diameter of the extraction jars fully submerged in the extraction solvent.

Following addition of each wipe, film coupon, and SPE disk to the extraction solvent, the jars were swirled by hand for approximately 5 to 10 seconds and placed into an ultrasonic bath. Extraction jars were sonicated at 40 to 60 kHz for 10 min. The temperature of the water in the ultrasonic bath was not allowed to increase by more than 10 °C above ambient temperature. Within 30 minutes of completing sonication, approximately 0.5 mL aliquots from each extraction jar were transferred to duplicate GC or LC autosampler vials and capped. One vial served as a primary analysis sample, and the second was stored as an archive sample. Samples that were not analyzed the day of preparation were stored at -20 ± 10 °C until analysis was performed. Samples were analyzed within 3 days for CWAs or 4 days for pesticides after they were prepared during testing (in accordance with the decontaminant quench and sample preservation method demonstrated during method development testing, as described in Section D.3).

C.7 Quantitative Analysis by GC/MS

Extracts of wipe samples, film coupons, and SPE disks from samples contaminated with HD and malathion were analyzed in selected ion monitoring (SIM) mode on an Agilent 7890 or 8890 GC using an Agilent 5977A or 5977B Agilent mass selective detector. Data were acquired using

Agilent MassHunter Software. Naphthalene-d_8 was used as an internal standard (IS) for HD mass quantification while malathion-d_{10} was used as the internal stand for malathion mass quantification. Malaoxon (CAS # 1634-78-2), a malathion degradation product, was also quantified; malathion-d_{10} was used as the IS for malaoxon. Quantification and qualifier ions for each analyte are shown in Table 6.

Table 6. Expected Analyte Ion Transitions

Analyte	Quantification m/z	Qualifier m/z
HD	109	111, 158, 160
Naphthalene-d_8	136	134, 137, 108
Malathion	173	127, 99
Malathion-d_{10}	183	132, 100
Malaoxon	99	127, 142

The GC/MS was tuned initially and as needed following manufacturer's guidelines. Prior to running each set of samples, a decafluorotriphenylphosphine (DFTPP) tune check analysis was performed with the MS run in the full scan mode to ensure proper MS operation. A 12-hour tune time was not employed.

Table 7. GC/MS Conditions for All Analyses

Parameter	DFTPP Analysis	HD Analysis	Malathion Analysis
Column	Rxi-5Sil MS, 30.0 meters by 0.25 millimeters (mm), 0.25 micrometers (µm) film thickness		
Liner Type	4 mm split/splitless		
Carrier Gas Flow	1.5 mL/min	1.2 mL/min	1.5 mL/min
Column Temperature	50 °C initial temp., hold 0.0 minutes, 30 °C/minute to 280 °C, hold 3.0 minutes	50 °C initial temp., hold 0.50 minutes, 30 °C/minute to 280 °C, hold 0.50 minutes.	100 °C initial temp., hold 0.50 minutes, 20 °C/minute to 250 °C, hold 1.0 minute, 30 °C/minute to 280 °C, hold 0.50 minutes.
Injection Volume	1.0 µL	2.0 µL	2.0 µL
Injection Temp.	250 °C		
MS Quad Temp.	150 °C		
MS Source Temp.	230 °C		

HD stock solutions were prepared in hexane and acetone from HD Lot Number C066-2. Stock solution concentrations were calculated using Equation 1:

$$C_A = \frac{V_A \times D_A \times P_A}{V_S} \qquad (1)$$

where:

C_A = analyte concentration in the solution (milligrams (mg)/mL)

V_A = volume of the analyte added to the solvent (µL)

D_A = density of the analyte (mg/µL)

P_A = purity of the analyte (refer to Table 1)

V_S = volume of the solvent (mL)

Naphthalene-d_8 was purchased from Supelco (part # 442716, 250 mg). A 505 µg/mL naphthalene-d_8 solution was prepared in *n*-hexane. Malathion was purchased from Sigma-Aldrich (part # 36143, 100 mg) as was malaoxon (part # 36142, 100 mg vial). Stock solutions containing a cocktail of malathion and malaoxon at an equal concentration were prepared by dissolving a known mass of each analyte in a known volume of solvent. Malathion/malaoxon stock solutions were prepared in both hexane and acetone. Malathion-d_{10} was purchased from Sigma-Aldrich (part # 3514, 10 mg). A 250-micrograms (µg)/mL malathion-d_{10} solution was prepared in *n*-hexane.

Stock solutions were used to prepare separate HD and malathion/malaoxon calibration standards in both *n*-hexane and acetone to allow matrix matching of calibration standards with sample extracts. A six-point calibration was analyzed prior to each set of sample analyses; concentration levels for both HD, malathion, and malaoxon were: 0.10, 0.50, 1.0, 2.5, 5.0, and 10 µg/mL. Continuing calibration verification (CCV) standards were prepared by a second analyst at 0.10 and 5.0 µg/mL. CCV analysis was performed after every five samples, alternating between low and high concentration CCVs, and at the end of the analytical sequence. Prior to analysis calibration standards, CCVs, and samples were spiked with naphthalene-d_8 or malathion-d_{10} at a final concentration of 5.0 µg/ml. The expiration date for HD and malathion calibration standards was six months, while CCVs had a three-month expiration date. Calibration standards and CCVs were stored in a freezer at -20 ± 10 °C when not in use.

A linear or quadratic regression was used to describe the data with 1/x or $1/x^2$ weighting with the origin excluded. Refer to Table 8 for analysis performance parameters and acceptance criteria. The GC/MS was recalibrated if the coefficient of determination (r^2) from the regression analysis of the standards was less than 0.990. Each calibration standard or CCV was required to calculate back (using the established calibration curve) to within ±15% of the nominal standard concentration. The lowest calibration standard and CCV were required to be within ±25% of the nominal standard concentration. Following analysis of the calibration standards at the beginning of each analytical run, a solvent blank sample was analyzed to confirm that no target analyte carryover above the lowest calibration standard had occurred. Internal standard area in the test samples was compared to the area of internal standard in the nearest passing calibration standard or passing CCV.

Table 8. Analysis Performance Parameters and Acceptance Criteria

Parameter	Criterion
Calibration curve coefficient of determination (r^2)	≥ 0.990
Acceptance limit for lowest calibration standard processed against curve	75 - 125%
Acceptance limit for remaining calibration standards processed against curve	85 - 115%
Solvent blank samples	< lowest calibration standard
Acceptance limit for lowest CCV	75 - 125%
Acceptance limit for remaining CCVs	85 - 115%
Signal-to-noise ratio for the lowest calibration standard	Minimum of 3:1
Retention time for target compound and IS	± 0.1 min as same compounds in mid-level calibration standard
IS area in samples	50% to 200% area of nearest passing calibration standard or passing CCV, criteria per EPA Method 8000D [8]

The concentration of analyte in samples was interpolated using the analyte area/IS area ratio and the regression equation generated from calibration standards. Samples that quantitated below the lowest calibration standard concentration of the curve were reported as less than the Lower Limit of Quantitation (LLOQ), e.g., <0.10 µg/mL. Samples that quantitated above the highest calibration standard of the curve were diluted using calibrated positive displacement pipettes and reanalyzed. If the internal standard area was outside the acceptance range of 50% - 200%, the sample dilution factor was increased to reduce matrix effects until a passing internal standard area was obtained; the LLOQ was adjusted on a per-sample basis to account for any required dilutions. All data were reported to two significant figures.

C.8 Quantitative Analysis by LC-MS/MS

Extracts of wipes, film coupons, and SPE disks contaminated with VX and fipronil were analyzed using reversed-phase HPLC and multiple reaction monitoring (MRM) mass spectrometry on an AB Sciex 5500 triple quadrupole MS coupled to a Shimadzu 20 XR series LC. VR (Russian VX; CAS # 159939-87-4) was obtained from the U.S. Army Combat Capabilities Development Command (CCDC) Chemical Biological Center as a solution (85.7 µg/mL, lot # RVX/IPA-7145-R&T-DIL-C) and used as the internal standard for quantitation of VX. Labeled fipronil ($^{13}C_4$, $^{15}N_2$; CNLM-9650-1.2, Cambridge Isotope Laboratories, Inc.) was used as the internal standard for quantitation of fipronil. Internal standard was added to calibration standards, controls, and test sample extracts just prior to LC-MS/MS analysis using a solution of VR or labeled fipronil in water as the sample diluent. Table 9 provides the ion transitions used for quantitation of the analytes, and Table 10 and Table 11 provide additional LC-MS/MS method conditions for each analyte.

Table 9. Analyte Ion Transitions

Analyte	Precursor Ion	Product Ion Quantifier
VX	268	128
VR	268	100
Fipronil	435	330
Labeled Fipronil	441	336

The most sensitive transitions for fipronil and fipronil $^{13}C_4$ internal standard are 435 > 330 and 439 > 334, respectively. However, the fipronil molecule contains two chlorine atoms causing its chlorine isotopes to add significant area to the most sensitive fipronil $^{13}C_4$ transition, resulting in varied internal standard areas based on the concentration of fipronil in the sample. For this reason, the second most abundant carbon isotope of fipronil, $^{13}C_4$, was selected as the internal standard transition, 441 > 336.

Table 10. LC-MS/MS Conditions for Analysis of VX

Parameter	Description		
Ionization Mode and Polarity	Electrospray ionization, positive mode		
HPLC Column	Restek Allure pentafluorophenylpropyl (PFPP), 2.1 x 50 mm, 5 µm, part 9169552		
Column Temperature	Ambient		
Mobile Phase	A: 2 millimoles (mM) Formic Acid/2 mM Ammonium Formate in Water		
	B: 2 mM Formic Acid/2 mM Ammonium Formate in Methanol		
Mobile Phase Gradient	**Time (minutes)**	**%B**	**Flow Rate (mL/min)**
	0.0	20	0.5
	1.0	20	0.5
	2.0	100	0.7
	4.0	100	0.7
	4.1	20	0.5
	4.5	20	0.5
Typical Injection Volume	5 µL		
Run Time	4.5 minutes		

Table 11. LC-MS/MS Conditions for Analysis of Fipronil

Parameter	Description		
Ionization Mode and Polarity	Electrospray ionization, negative mode		
HPLC Column	Waters Atlantis dC18, 2.1 x 50 mm, 3 µm, part 186001291		
Column Temperature	Ambient		
Mobile Phase	A: 2 mM Formic Acid/2 mM Ammonium Formate in Water		
	B: 2 mM Formic Acid/2 mM Ammonium Formate in Methanol		
Mobile Phase Gradient	**Time (minutes)**	**%B**	**Flow Rate (mL/min)**
	0.0	15	0.3
	1.0	15	0.3
	3.0	100	0.3
	4.0	100	0.3
	4.1	15	0.3
	5.0	15	0.3

Typical Injection Volume	10 µL
Run Time	5.0 minutes

Neat VX and a purchased fipronil stock (AccuStandard P-738S-A, 100 µg/mL in acetone) were used to prepare calibration standards in both isopropanol and acetone to allow matrix matching of calibration standards with sample extracts. Stock solutions of VX were prepared from Lot Number C070-7-1 or C070-7-2 (refer to Table 1). Table 12 provides a summary of the calibration level concentrations for standards and CCVs. The expiration date for VX and fipronil calibration standards was three months from the date of preparation. The expiration date for VX CCV standards was one month from the date of preparation. The expiration date for fipronil CCV standards was three months from the date of preparation. Calibration standards and CCVs were stored at -20 ± 10 °C. The signal-to-noise ratio of the lowest calibration standard was required to be 3:1 at minimum.

Table 12. LC-MS/MS Conditions for Quantitative Analysis of Fipronil

Calibration Level	VX (ng/mL)	Fipronil (ng/mL)
1	0.010	0.010
2	0.020	0.040
3	0.040	0.20
4	0.20	0.40
5	0.40	1.0
6	1.0	2.0
7	2.0	5.0
Low CCV	0.010	0.010
High CCV	1.0	2.0
IS	0.45	1.8

CCVs prepared by a second analyst were analyzed prior to sample analysis and after no more than every ten samples. Quantifiable samples bracketed by a failing CCV were reanalyzed. Calibration standards and CCVs were matrix-matched to the samples as closely as possible. For example, test samples in IPA prepared for analysis by a 10-fold dilution in water were quantitated using calibration standards and CCVs prepared in 10% IPA. Note that due to this 10-fold dilution performed during sample preparation, the LLOQs for VX and fipronil were 0.10 ng/mL.

A linear or quadratic regression was used to describe the data with $1/x^2$ weighting with the origin excluded. Each calibration standard or CCV was required to calculate back (using the established calibration curve) to within ±15% of the nominal standard concentration. The lowest calibration standard and CCV were required to be within ±25% of the nominal standard concentration. Table 8 provides a summary of the analytical run acceptance criteria for various parameters.

The concentration of analyte in samples was interpolated using the analyte area/IS area ratio and the regression equation generated from calibration standards. Samples that quantitated below the lowest calibration standard concentration, or displayed area counts below the area counts of the lowest concentration on the calibration curve, were reported as less than the LLOQ corrected to account for the sample dilution factor (e.g., <0.10 ng/mL). Samples that quantitated above the highest calibration standard were diluted using calibrated positive displacement pipettes and reanalyzed. If the internal standard area was outside the acceptance range of 50% - 200%, the sample dilution factor was increased to reduce matrix effects until a passing internal standard area was obtained. All data were reported to two significant figures.

C.9 Calculations

Wipe, film coupon, and SPE disk extract concentrations of CWAs and pesticides for all test, control, and blank samples were calculated in units of µg/mL for GC/MS analyses and ng/mL for LC-MS/MS analyses. All calibrations used an internal standard. Instrument software was used to calibrate each instrument. A quadratic regression or weighted quadratic regression was typically used for a calibration fit for all compounds.

Mass recovered from the wipe samples, film coupons, and SPE disks via extraction was determined according to Equation 2:

$$Mass_{Rec} = \frac{Conc_{Ext}}{Conv} \times Vol_{Ext} \qquad (2)$$

where: $Mass_{Rec}$ = CWA or pesticide mass recovered from the wipe/film coupon/SPE disk (µg)

$Conc_{Ext}$ = Wipe, film, or SPE disk extract CWA/pesticide concentration (in units of ng/mL or µg/mL)

Vol_{Ext} = Volume of wipe/film/SPE disk extraction solvent (mL)

Conv = Conversion factor (1000 for LC-MS/MS analyses; 1 for GC/MS analyses)

Total sample mass was determined using the average masses recovered from the wipe sample, paint/sealant film coupon, and SPE disk according to Equation 3:

$$Mass_{Tot} = Mass_{Rec\ (wipe)} + Mass_{Rec\ (film)} + Mass_{Rec\ (SPE)} \qquad (3)$$

where: $Mass_{Tot}$= Average total CWA/pesticide mass recovered (µg)

$Mass_{Rec\ (wipe)}$ = Average CWA/pesticide mass recovered from the wipe sample (µg)

$Mass_{Rec\ (film)}$ = Average CWA/pesticide mass recovered from the film (µg)

$Mass_{Rec\ (SPE)}$ = Average CWA/pesticide mass recovered from the SPE disk (µg)

CWA or pesticide total decontamination efficacy was calculated using the average total mass recovered from the test samples and associated positive controls according to Equation 4:

$$E_T = \left(\frac{Mass_{Tot\ (pos)} - Mass_{Tot(test)}}{Mass_{Tot\ (pos)}}\right) \times 100 \qquad (4)$$

where: E_T = Total decontamination efficacy (%)

$Mass_{Tot\ (test)}$ = Average total CWA/pesticide mass recovered from a test sample (µg)

$Mass_{Tot\ (pos)}$ = Average total CWA/pesticide mass recovered from the associated positive control (µg)

Surface decontamination efficacy was calculated based only on wipe results using Equation 5. For surface decontamination efficacy, the average total mass recovered was replaced with average wipe mass recovered for the test samples and average wipe mass recovered for the positive controls.

$$E_S = \left(\frac{Mass_{Wipe\ (pos)} - Mass_{Wipe(test)}}{Mass_{Wipe\ (pos)}}\right) \times 100 \qquad (5)$$

where: E_S = Surface decontamination efficacy (%)

$Mass_{Wipe\ (test)}$ = Average wipe CWA/pesticide mass recovered from a test sample (µg)

$Mass_{Wipe\ (pos)}$ = Average wipe CWA/pesticide mass recovered from the associated positive control (µg)

For each CWA or pesticide, film type, and decontamination technology (or technology modification) combination, the surface and total decontamination efficacy was reported.

The propagation of error for the surface and total decontamination efficacy was also calculated. Equation 4 and equation 5 can be rewritten as Equation 6:

$$E = \frac{b-a}{b} = 1 - a/b \qquad (6)$$

where: $a = Mass_{Tot\ (test)}$

$b = Mass_{Tot\ (pos)}$

The decontamination efficacy error (ΔE) was calculated using Equation 7:

$$\Delta E = (1 - E)\sqrt{\left(\frac{\Delta a}{a}\right)^2 + \left(\frac{\Delta b}{b}\right)^2} \qquad (7)$$

where: Δa = the standard deviation of a

Δb = the standard deviation of b

In cases where one or more replicates was a nondetect, for either the test sample or positive control, a standard deviation could not reasonably be calculated for those samples, and therefore the efficacy error was not calculated.

D. TESTING APPROACH

D.1 Wipe Sampling Demonstration

The film surface wipe sampling methods developed for CWAs during previous testing [1] were evaluated for use in recovering residual surface HD, VX, malathion, and fipronil contamination from the surface of paint and sealant films during this testing. The wipe sampling method demonstration testing evaluated recovery of HD, VX, malathion, and fipronil from freestanding films of the paint and sealant selected for this project. Freestanding films of the paint and sealant were contaminated with 2 µL of CWA or 12 µL of pesticide solution, and the solutions were allowed to be in contact with the surface of the film coupons for 60 minutes. Following the contact period, film coupons were wiped, extracted, and analyzed as described above.

Table 13 provides the wipe-sampling method demonstration test matrix. The matrix was completed a total of four times, one time each for HD, VX, malathion, and fipronil. Three replicates of paint film and sealant film coupons were evaluated for wipe recovery along with three replicates of stainless steel coupons (2-inch diameter 18-8 stainless steel disk shim) used as a nonpermeable control material. One replicate of each film and one replicate stainless steel coupon were sampled via direct extraction rather than by wipe sampling to assess the mass of CWA/pesticide that evaporates from the surface of the films during the 60-minute contact period (i.e., evaporation controls).

In addition to the test sample film coupons and evaporation controls, a single procedural blank per material type was included. Procedural blanks were not spiked with CWA or pesticide but were wipe-sampled and analyzed alongside the test samples. A single laboratory blank per material type was also included. Laboratory blanks were not spiked with CWA or pesticide and were sampled via direct solvent extraction. Three spike control samples were prepared to confirm the CWA/pesticide application mass.

Table 13. Wipe Sampling Demonstration Matrix

Sample Type	Material	Contaminant Spike Vol.	Contaminant Contact Period	Sampling Method	Replicates
Test	Paint	2 µL or 12 µL	60 minutes	Wipe Sampling	3
Test	Sealant	2 µL or 12 µL	60 minutes	Wipe Sampling	3
Control	Stainless Steel	2 µL or 12 µL	60 minutes	Wipe Sampling	3
Evaporation Control	Paint	2 µL or 12 µL	60 minutes	Extraction	1
Evaporation Control	Sealant	2 µL or 12 µL	60 minutes	Extraction	1
Evaporation Control	Stainless Steel	2 µL or 12 µL	60 minutes	Extraction	1
Procedural Blank	Paint	None	NA	Wipe Sampling	1
Procedural Blank	Sealant	None	NA	Wipe Sampling	1
Procedural Blank	Stainless Steel	None	NA	Wipe Sampling	1
Laboratory Blank	Paint	None	NA	Extraction	1
Laboratory Blank	Sealant	None	NA	Extraction	1
Laboratory Blank	Stainless Steel	None	NA	Extraction	1
Spike Control	None	2 µL or 12 µL	NA	Extraction	3

NA = not applicable

The average recoveries from the spike controls were required to be within 80% to 120% of the theoretical target amount with a replicate RSD of < 30% (n=3). The wipe-sampling method was deemed acceptable for use during subsequent fate and transport and decontamination efficacy testing if the average wipe-sampling recoveries were within the range of 70% to 120% of the average of the spike control results with a replicate RSD < 30% (n=3). Procedural blanks and laboratory blanks should have had less than 50% of the lowest detected amount on the test coupon.

D.2 Solvent Extraction Demonstration

Recovery of HD, VX, malathion, and fipronil via extraction of paint and sealant film coupons and SPE disks in solvent was evaluated. Solvent extraction methods were based on the methods developed and used during previous testing [1]. Solvent extraction method demonstration testing evaluated recovery of HD, VX, malathion, and fipronil from freestanding film coupons of the paint and sealant selected for this project and from SPE disks. Freestanding film coupons of the paint and sealant and SPE disks were contaminated with 2 µL of CWA or 12 µL of pesticide solution, and the solution was allowed to be in contact with the surface of film coupons/within the SPE disks for 60 minutes. Following the contact period, film coupons and SPE disks were extracted and analyzed as described above.

Table 14 provides the solvent extraction method demonstration test matrix. The matrix was completed four times, once each for HD, VX, malathion, and fipronil. Three replicates of paint and sealant films and SPE disks were evaluated for solvent extraction recovery along with three replicates of stainless steel used as a nonpermeable control material.

In addition to the test sample film coupons, SPE disks, and evaporation controls, a single laboratory blank per material type was included. Laboratory blanks were not spiked with CWA or pesticide. Three spike control samples were prepared to confirm the CWA/pesticide application mass.

Table 14. Solvent Extraction Demonstration Matrix

Sample Type	Material	Contaminant Spike Vol.	Contaminant Contact Period	Replicates
Test	Paint	2 µL or 12 µL	60 minutes	3
Test	Sealant	2 µL or 12 µL	60 minutes	3
Test	SPE Disk	2 µL or 12 µL	60 minutes	3
Control	Stainless Steel	2 µL or 12 µL	60 minutes	3
Laboratory Blank	Paint	None	NA	1
Laboratory Blank	Sealant	None	NA	1
Laboratory Blank	SPE Disk	None	NA	1
Laboratory Blank	Stainless Steel	None	NA	1
Spike Control	None	2 µL or 12 µL	NA	3

The average recoveries from the spike controls were required to be within 80% to 120% of the target amount and a replicate RSD of < 30% (n=3). The solvent extraction method was deemed acceptable for use during subsequent fate and transport and decontamination efficacy testing if the average wipe-sampling recoveries were within the range of 70% to 120% of the average of the spike control results with a replicate RSD < 30% (n=3). Laboratory blanks should have had less than 50% of the lowest detected amount on the test coupon.

D.3 Decontaminant Quench Demonstration

During decontamination efficacy testing residual decontaminant (absorbed into wipes and potentially present in film and SPE disk extracts) could continue to decontaminate CWA and pesticides beyond the defined decontaminant dwell periods, create complex sample matrices, and/or cause analytical interference such as false-positive or false-negative results or analyte enhancement or suppression. Effective methods for quenching decontaminants following the decontaminant dwell period were determined to allow efficacy of each tested decontaminant to be evaluated as a function of dwell time. Additionally, assessment of matrix effects was evaluated to ensure the sample matrices did not interfere with analysis.

Prior to decontamination efficacy testing, the method for quenching (i.e., halting) the reactions of three decontaminants were demonstrated so that decontamination efficacy could be measured as solely a function of decontaminant dwell time on the films. During a previous study [9], a 3 M solution of STS in water was found to effectively halt the reaction of various test decontaminants, including decontaminants based on hypochlorite, peroxide, and peracetic acid active ingredients. Procedurally, 5 mL of 3 M STS was included with the 10 mL of solvent used to extract decontaminated samples. This method was evaluated for use during this testing to halt the reactions of test decontaminants so that CWAs/pesticides in extracts of wipes, films, and SPE disks following the decontaminant dwell period were preserved for quantitation.

Table 15 provides the experimental matrix for HD and VX decontaminant quench that was intended to serve two purposes: to evaluate 3 M STS as an appropriate quench solution for neutralizing the decontaminant reaction with HD and VX and to evaluate possible effects from residual decontaminant, the 3 M STS quench agent itself, and/or extracted wipe/film coupon/SPE disk compounds on the analysis of HD and VX and the response of the associated internal standards. Each sample was spiked with a mass of HD that represented 2% of the total mass used for testing or 0.001% of the total VX mass used for testing. Note that each replicate resulted in one wipe sample, one film sample, and one SPE disk sample. This experimental matrix was completed twice, once for HD and once for VX.

Table 15. HD and VX Decontaminant Quench Demonstration Matrix

Sample Type	Decontaminant	Decontaminant Dwell Period	Spike Mass	Replicates
Test	Bleach	60 minutes	HD = 49 µg	3
			VX = 20 ng	3
Test	D7	60 minutes	HD = 49 µg	3
			VX = 20 ng	3
Test	Dahlgren Decon	60 minutes	HD = 49 µg	3
			VX = 20 ng	3
Positive Control	None	NA	HD = 49 µg	3
			VX = 20 ng	3
Procedural Blank	Bleach	60 minutes	NA	1
Procedural Blank	D7	60 minutes	NA	1
Procedural Blank	Dahlgren Decon	60 minutes	NA	1
Laboratory Blank	None	NA	NA	1
Spike Control	None	NA	HD = 49 µg VX = 20 ng	3

Table 16 provides the experimental matrix for malathion and fipronil decontaminant quench that serves the same purposes as the matrix for the HD and VX. Note that a 10-fold dilution of bleach in water and D7 were the only decontaminants selected for pesticide testing. Each sample was spiked with the same mass of malathion or fipronil as was used in testing. Note that each replicate resulted in one wipe sample, one film sample, and one SPE disk sample. This matrix was completed twice, once for malathion and once for fipronil.

Table 16. Malathion and Fipronil Decontaminant Quench Demonstration Matrix

Sample Type	Decontaminant	Decontaminant Dwell Period	Spike Mass	Replicates
Test	10x Diluted Bleach	60 minutes	malathion = 37 µg fipronil = 14 µg	3
Test	D7	60 minutes	malathion = 37 µg fipronil = 14 µg/mL	3
Positive Control	None	NA	malathion = 37 µg fipronil = 14 µg	3
Procedural Blank	10 x Bleach	60 minutes	NA	1
Procedural Blank	D7	60 minutes	NA	1
Laboratory Blank	None	NA	NA	1
Spike Control	None	NA	malathion = 37 µg fipronil = 14 µg	3

Decontaminant quench samples representative of decontamination efficacy testing were prepared by applying decontaminants by spray application as described above to paint film coupons in LVAP assemblies and allowing the decontaminants to contact the films for 60 minutes. Note that only paint film coupons were used for decontaminant quench demonstration. Following the decontaminant contact period, the paint film coupons were wiped, and the wipe samples extracted by sonication. The LVAP assemblies were then disassembled and the paint films and

SPE disks were solvent extracted by sonication. Quench solution (5 mL of 3 M STS) was added to the jars containing the 10 mL of solvent for extraction of the wipes, film coupons, and SPE disks.

Each target analyte was added to the 10 mL of extraction solvent at a known concentration. The final concentrations in solvent were as follows: HD = 4.9 µg/mL, VX = 2.0 ng/mL, malathion = 3.7 µg/mL, and fipronil = 1.4 µg/mL. Aliquots of the extracts were collected from the solvent layer following sonication and analyzed by GC/MS for HD and malathion and LC-MS/MS for VX and fipronil. Following the initial analyses, the HD and VX extracts were stored at -20 ± 10 °C for 3 days (nominal 72 hours) and the malathion and fipronil extracts were stored for 4 days (nominal 96 hours). Following the 3-day or 4-day storage period, the extracts were analyzed again via LC-MS/MS or GC/MS. The initial and reanalysis results were compared to each other, the nominal spike concentration, and to positive controls to determine the effectiveness of the quench method.

The following acceptance criteria needed to be met for the quench demonstration to have been successful:

- Recovery of CWA/pesticide mass was ≥ 70% relative to the positive control for samples containing representative amounts of test decontaminants (quench samples).
- The criteria for internal standard response were satisfied.

Additionally, the results for sample extracts analyzed after 72 hours or 96 hours were compared to the initial analyses to determine if sample extract archives could be stored without degradation.

D.4 Fate and Transport Assessment

Previous fate and transport evaluations [1] used LVAP test methodologies to determine that HD and VX applied to the surface of paint and sealant films would penetrate the permeable films and migrate into underlying porous materials. Fate and transport testing conducted during this project was conducted to replicate earlier testing.

Fate and transport testing was conducted using the modified LVAP test cell assemblies as described in Section C.3. Table 17 provides the CWA fate and transport test matrix; this matrix was performed twice, once using HD with a 24-hour contact time and once using VX with 72-hour contact time. Note that each replicate resulted in one wipe sample, one film coupon sample, and one SPE disk sample.

Table 17. CWA Fate and Transport Matrix

Sample Type	Material	CWA Spike Vol.	Replicates
Test	Paint	2 µL	3
Test	Sealant	2 µL	3
Lab Blank	Paint	NA	1
Lab Blank	Sealant	NA	1
Spike Control	None	2 µL	3

For malathion and fipronil, a contact period of 24 and 72 hours was determined to be evaluated as these data had not previously been collected. Table 18 provides a matrix for fate and transport testing using the two pesticides. This matrix was performed twice, once using malathion and once using fipronil. Note that each replicate resulted in one wipe sample, one film coupon sample, and one SPE disk sample.

Table 18. Pesticide Fate and Transport Matrix

Sample Type	Material	Pesticide Solution Spike Vol.	Contact Period	Replicates
Test	Paint	12 µL	24 hours	3
Test	Paint	12 µL	72 hours	3
Test	Sealant	12 µL	24 hours	3
Test	Sealant	12 µL	72 hours	3
Lab Blank	Paint	NA	24 hours	1
Lab Blank	Paint	NA	72 hours	1
Lab Blank	Sealant	NA	24 hours	1
Lab Blank	Sealant	NA	72 hours	1
Spike Control	None	12 µL	NA	3

Neat CWA was applied to the films as a single 2-µL droplet, and pesticide solutions were applied as a single 12-µL droplet. Following the contact period all films were sampled via wiping. LVAP assemblies were disassembled and paint/sealant film coupons and SPE disks were extracted in solvent. Sample extracts were analyzed by GC/MS for HD and malathion or LC-MS/MS for VX and fipronil to quantify CWAs/pesticides in each LVAP assembly component. Laboratory blanks were included and sampled and analyzed alongside the test samples. Three spike controls were prepared during each test to confirm the mass of CWA/pesticide applied to test samples.

Based on the results of the 24-hour and 72-hour malathion and fipronil wipe, film, and SPE disk samples (see Section D.4), a 72-hour contact time was selected for use with malathion and fipronil decontamination efficacy testing.

D.5 Baseline Decontamination Efficacy Testing

Following fate and transport testing, the decontamination step was incorporated to evaluate baseline efficacy of selected decontaminants for CWAs and pesticides. Test samples were contaminated, decontaminated, sampled, and analyzed for CWA/pesticide. Decontamination efficacy was defined as the percentage of CWA or pesticide remaining in the LVAP test sample assembly components (total from the wipe sample, freestanding film, and SPE disk extracts) compared to the positive control samples. Surface decontamination efficacy was defined as the percentage of CWA or pesticide remaining on the surface (wipe sample) compared to the positive control samples.

Table 19 provides a test matrix for evaluating the baseline efficacy of three decontaminants (bleach, D7, and Dahlgren Decon) against HD and VX applied to the surface of paint and sealant film coupons; this matrix was performed twice, once using HD with a 24-hour contact time and once using VX with 72-hour contact time. Decontaminant dwell time was 60 minutes for all baseline testing. Paint and sealant films were tested in triplicate. Note that each replicate resulted in one wipe sample, one film sample, and one SPE disk sample.

CWA positive control samples were also prepared in triplicate but did not have decontaminant applied. Procedural blank samples were not contaminated with CWA but were decontaminated and processed alongside the test samples. Laboratory blank samples were neither contaminated nor decontaminated and were also processed alongside the test samples. Procedural blanks and laboratory blanks should have had less than 50% of the lowest detected amount on the test coupon. Three spike controls were prepared during each test to confirm the mass of CWA applied to the test samples. The average recoveries from the spike controls were required to be within 80% to 120% of the theoretical target amount with a replicate RSD of < 30% (n=3).

Table 19. CWA Baseline Decontamination Efficacy Matrix

Sample Type	Material	CWA Spike Vol.	Decontaminant	Replicates
Test	Paint	2 µL	Bleach	3
Test	Paint	2 µL	D7	3
Test	Paint	2 µL	Dahlgren Decon	3
Test	Sealant	2 µL	Bleach	3
Test	Sealant	2 µL	D7	3
Test	Sealant	2 µL	Dahlgren Decon	3
Positive Control	Paint	2 µL	None	3
Positive Control	Sealant	2 µL	None	3
Procedural Blank	Paint	NA	Bleach	1
Procedural Blank	Paint	NA	D7	1
Procedural Blank	Paint	NA	Dahlgren Decon	1
Procedural Blank	Sealant	NA	Bleach	1
Procedural Blank	Sealant	NA	D7	1
Procedural Blank	Sealant	NA	Dahlgren Decon	1
Lab Blank	Paint	NA	None	1
Lab Blank	Sealant	NA	None	1
Spike Control	None	2 µL	None	3

Table 20 provides a test matrix for evaluating the baseline efficacy of two decontaminants (10-fold dilution of bleach in water and D7) against malathion and fipronil applied to the surface of paint and sealant films; this matrix was performed twice, once using malathion and once using fipronil, both with a 72-hour pesticide contact time. Decontaminant dwell time was 60 minutes for all baseline testing. Paint and sealant film coupons were tested in triplicate. Note that each replicate resulted in one wipe sample, one film coupon sample, and one SPE disk sample.

Pesticide positive control samples were also prepared in triplicate but did not have decontaminant applied. Procedural blank samples were not contaminated with pesticide but were decontaminated and processed alongside the test samples. Laboratory blank samples were neither contaminated nor decontaminated and were also processed alongside the test samples. Procedural blanks and laboratory blanks should have had less than 50% of the lowest detected amount on the test coupon. Three spike controls were prepared during each test to confirm the mass of pesticide applied to the test samples. The average recoveries from the spike controls were required to be within 80% to 120% of the theoretical target amount with a replicate RSD of < 30% (n=3).

Table 20. Pesticide Baseline Decontamination Efficacy Matrix

Sample Type	Material	Pesticide Solution Spike Vol.	Decontaminant	Replicates
Test	Paint	12 µL	10x Diluted Bleach	3
Test	Paint	12 µL	D7	3
Test	Sealant	12 µL	10x Diluted Bleach	3
Test	Sealant	12 µL	D7	3
Positive Control	Paint	12 µL	None	3
Positive Control	Sealant	12 µL	None	3
Procedural Blank	Paint	NA	10x Diluted Bleach	1
Procedural Blank	Paint	NA	D7	1
Procedural Blank	Sealant	NA	10x Diluted Bleach	1
Procedural Blank	Sealant	NA	D7	1
Lab Blank	Paint	NA	None	1
Lab Blank	Sealant	NA	None	1
Spike Control	None	12 µL	None	3

Following the CWA/pesticide contact period, decontaminants were sprayed onto the film surfaces as described in Section C.4. Following the decontaminant dwell time, film coupons were wiped, and the wipes were solvent-extracted. The LVAP assemblies were then disassembled and paint/sealant film coupons and SPE disks were solvent-extracted. Wipe, film, and SPE disk extracts were analyzed via GC/MS for HD and malathion or LC-MS/MS for VX and fipronil to quantify residual CWA/pesticide.

Baseline efficacy of the decontaminants was determined through comparison of CWA/pesticide masses recovered from decontaminated test samples to the CWA/pesticide masses recovered from positive control samples.

D.6 Modified Decontamination Efficacy Testing - CWAs

Based on the results of testing to determine the baseline performance of the selected decontaminants, modifications to the decontamination approaches were made to improve the measured decontamination efficacies. Also based on the result of baseline testing, a down selection to two decontaminants was made for CWA testing and to one decontaminant for pesticide testing. Aside from the modifications, testing was conducted in a manner identical to baseline decontamination efficacy testing. Efficacy of the modified decontamination approaches was determined through comparison of CWA/pesticide masses recovered from decontaminated test samples to the CWA/pesticide masses recovered from positive controls. Calculated efficacies of the modified decontamination approaches were also compared to the baseline efficacies to determine if any of the modifications made to the decontamination approaches resulted in efficacy improvements.

Modification 1 for HD and VX decontamination evaluated the effect of increasing decontaminant dwell time from 60 minutes to 120 minutes. Table 21 provides a test matrix for evaluating the Modification 1 efficacy of two decontaminants (bleach and D7) against HD applied to the surface of paint and sealant film coupons with a 24-hour contact time. Table 22 provides a test matrix for evaluating the Modification 1 efficacy of two decontaminants (bleach and Dahlgren Decon) against VX applied to the surface of paint and sealant films with a 72-hour contact time. For HD and VX, paint and sealant film coupons were tested in triplicate. Note that each replicate resulted in one wipe sample, one film extraction sample, and one SPE disk sample.

CWA positive control samples were also prepared in triplicate but did not have decontaminant applied. Procedural blank samples were not contaminated with CWA but were decontaminated and processed alongside the test samples. Laboratory blank samples were neither contaminated nor decontaminated and were also processed alongside the test samples. Procedural blanks and laboratory blanks should have had less than 50% of the lowest detected amount on the test coupon. Three spike controls were prepared during each test to confirm the mass of CWA applied to the test samples. The average recoveries from the spike controls were required to be within 80% to 120% of the theoretical target amount with a replicate RSD of < 30% (n=3).

Table 21. HD Modification 1 Decontamination Efficacy Matrix

Sample Type	Material	CWA Spike Vol.	Decontaminant	Replicates
Test	Paint	2 µL	Bleach	3
Test	Paint	2 µL	D7	3
Test	Sealant	2 µL	Bleach	3
Test	Sealant	2 µL	D7	3
Positive Control	Paint	2 µL	None	3
Positive Control	Sealant	2 µL	None	3
Procedural Blank	Paint	NA	Bleach	1
Procedural Blank	Paint	NA	D7	1
Procedural Blank	Sealant	NA	Bleach	1
Procedural Blank	Sealant	NA	D7	1
Lab Blank	Paint	NA	None	1
Lab Blank	Sealant	NA	None	1
Spike Control	None	2 µL	None	3

Table 22. VX Modification 1 Decontamination Efficacy Matrix

Sample Type	Material	CWA Spike Vol.	Decontaminant	Replicates
Test	Paint	2 µL	Bleach	3
Test	Paint	2 µL	Dahlgren Decon	3
Test	Sealant	2 µL	Bleach	3
Test	Sealant	2 µL	Dahlgren Decon	3
Positive Control	Paint	2 µL	None	3
Positive Control	Sealant	2 µL	None	3
Procedural Blank	Paint	NA	Bleach	1
Procedural Blank	Paint	NA	Dahlgren Decon	1
Procedural Blank	Sealant	NA	Bleach	1
Procedural Blank	Sealant	NA	Dahlgren Decon	1
Lab Blank	Paint	NA	None	1
Lab Blank	Sealant	NA	None	1
Spike Control	None	2 µL	None	3

Modification 2 and Modification 3 both evaluated the use of chemical treatments to potentially extract CWA from paint and sealant film coupons prior to the application of decontaminant. The chemicals that were selected for testing were 2-butoxyethanol (CAS # 111-76-2), (Fisher Scientific, Part # AC154330010) and Zep® Foaming Wall Cleaner (FWC) with primary active ingredients per the SDS of 1-butoxypropan-2-ol (CAS # 5131-66-8), 1-5 % and 2-(2-butoxyethoxy)ethanol (CAS # 112-34-5), 1-5 %. These chemicals were selected based on their commercial use as cleansers with the ability to “lift” stains from surfaces. A proof-of-concept testing was initially performed to evaluate the effects of both chemicals on the paint and sealant films. The effects of each chemical on the analysis of HD and VX were also evaluated. Three different solutions of 2-butoxyethanol (BE) in distilled water were evaluated: 30% BE, 10% BE, and 0.5% BE. Zep FWC was purchased as an 18-ounce spray can and used as received (shaken well before each use).

All three BE solutions and the FWC were separately applied to a single paint coupon and a single sealant coupon installed in an LVAP assembly. A calibrated positive displacement pipette was used to apply 1.0 mL of the BE solutions; the 30% and 10% solutions covered 100% of the coupon surface while the 0.5% solution appeared to cover < 90% of the surface due to beading on the surface. The can of FWC was held about 9 inches above the coupon and applied by performing three spray-passes across the coupons. The mass of applied FWC was characterized by spraying the chemical to a Teflon disk (10 replicates) following the same spray procedure, then using a balance to weigh the amount of chemical on the Teflon disk. The average mass applied to the 20 cm^2 surface was 0.30 g with an RSD of 7.9%.

Observations were made and photographs taken at different time points (see Figure 8 and Figure 9) until each chemical was visibly dry on the coupon surface or until 4 hours had passed (see Figure 10 and Figure 11). Each chemical was also applied to stainless steel coupons for visual evaluation of residue after drying; no visible residue was observed for either chemical (photos not shown). As the 30% BE appeared to interact too aggressively with the films and the 0.5% BE did not cover the film surface well, the 10% BE was selected for testing. A residence time of 10 minutes on the paint film and 60 minutes on the sealant film was selected not to damage the film coupons (see highlighted photos in Figure 8 and Figure 9); the 10% BE solution had not dried at either of these time points. A residence time of 5 minutes was selected for the FWC (see highlighted photos in Figure 8 and Figure 9, during which the FWC remained wet.

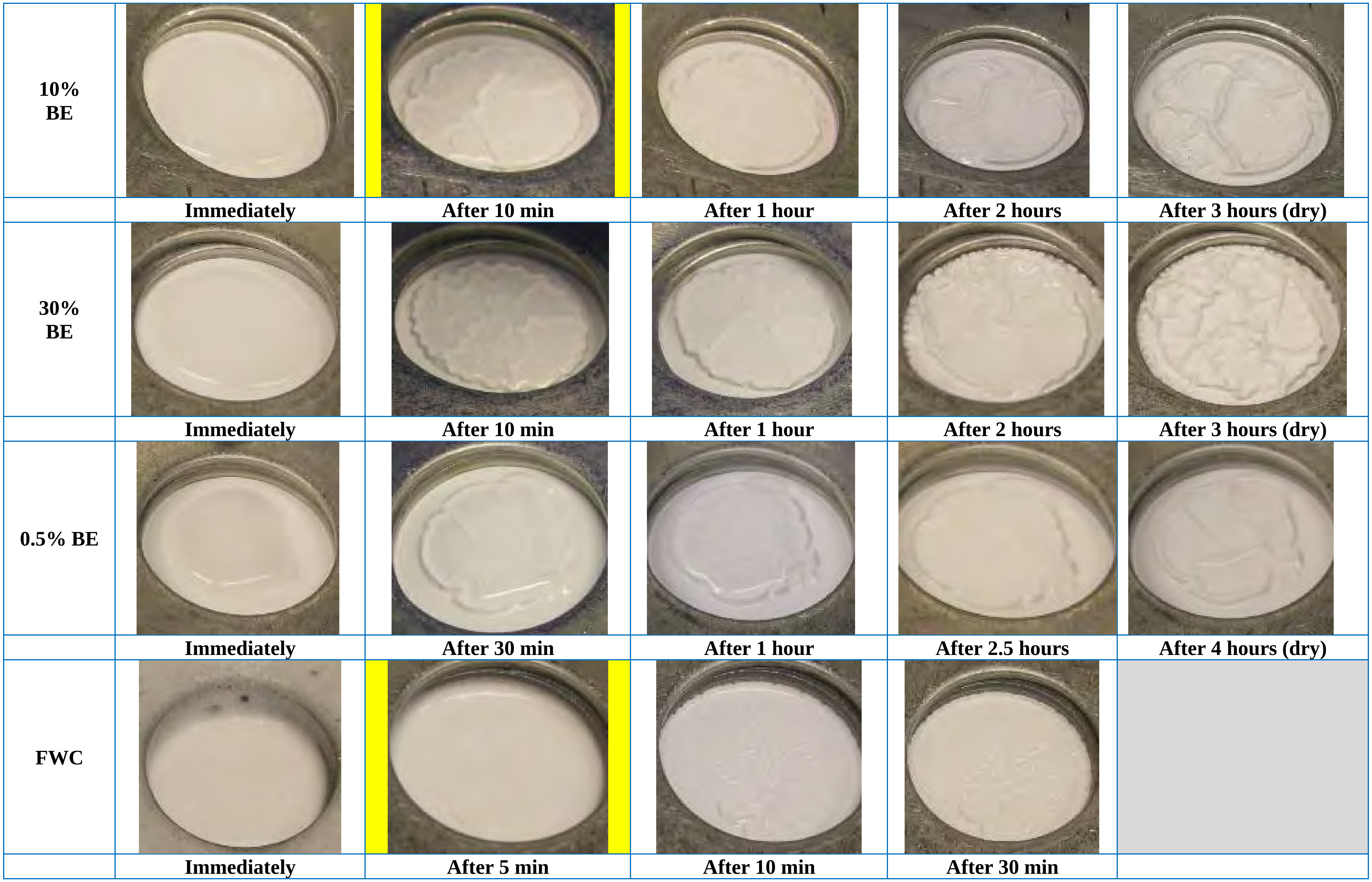


Figure 8. Evaluation of Paint Coupons with Modification 2/3 Chemicals

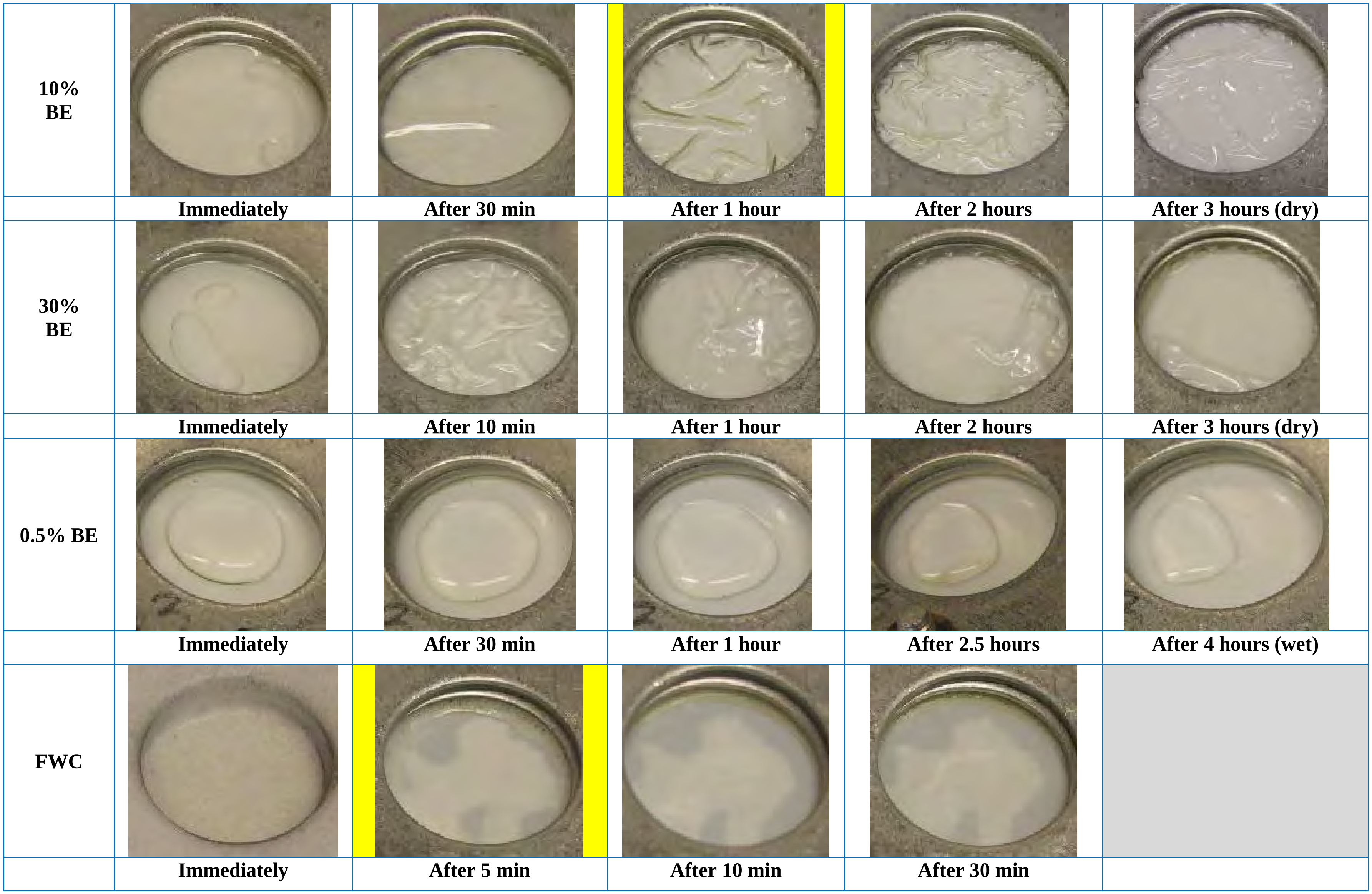


Figure 9. Evaluation of Sealant Coupons with Modification 2/3 Chemicals

10% BE		• Coupon was deformed/wrinkled • Teflon below coupon was wet • Coupon was slightly tacky
30% BE		• Coupon was deformed/wrinkled/stretched • Coupon was shriveling up once removed • Teflon below coupon was wet • Coupon was tacky
0.5% BE		• Coupon was deformed/wrinkled • Dry residue observed on Teflon • Coupon was not tacky
FWC		• Coupon was slightly deformed/wrinkled • Dry residue observed on Teflon • Coupon was slightly tacky

Figure 10. Observations of Paint Coupons After Chemical Dried

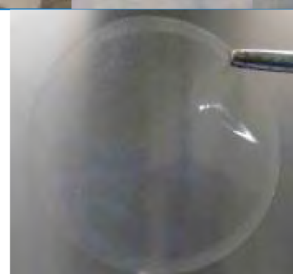
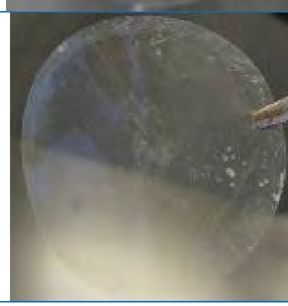
10% BE		• Coupon was deformed/wrinkled • No chemical breakthrough to Teflon • Coupon was slightly tacky
30% BE		• Coupon was deformed/wrinkled • Coupon difficult to remove from LVAP assembly • Coupon was shriveling up once removed • Teflon below coupon was wet • Coupon was tacky
0.5% BE		• Chemical not dry after 4 hours • No visible deformation/deterioration • No chemical breakthrough to Teflon • Coupon was not tacky
FWC		• No visible deformation • Slight residue/discoloration • No chemical breakthrough to Teflon • Coupon was not tacky

Figure 11. Observations of Sealant Coupons After Chemical Dried

A water rinse was then evaluated for removal of each chemical following treatment prior to application of decontaminant. Each chemical (1.0 mL of 10% BE and 0.3 g of FWC spray) were applied to paint and sealant coupons installed in LVAP assemblies and allowed to remain on the surface for the selected residence times; tests were performed in triplicate. Rinsing consisted of 3 x 2.0 mL applications of distilled water applied to the coupon with a positive displacement pipette. Each water application was allowed to sit for 5 minutes prior to removal with a Pasteur pipette; all three rinses were combined in a glass vial and inspected for evidence of dissolved paint/sealant. No visible dissolved paint/sealant was observed in any of the water rinse samples, however, the water rinse for FWC on paint was slightly foamy.

An aliquot of the combined rinsate was postspiked with VX at a concentration of 2.0 ng/mL and analyzed. To isolate HD from the combined rinsate a 1.0 mL aliquot was postspiked with HD at concentration of 1.0 µg/mL and extracted 1:1 with *n*-hexane, which resulted in a gel-like emulsion for all samples. A 1.0-mL aliquot was then postspiked with HD at concentration of 2.5 µg/mL and extracted 1:5 with hexane, which resulted in cloudy samples but no emulsions. The HD samples were then centrifuged at 1,000 rpm for 5 minutes to form two layers. The top organic layer was analyzed for HD. Table 23 shows the recovery of HD and VX for each chemical applied to each film type; all recoveries were > 90%. Unspiked rinsate was also analyzed for HD and VX. Results were all below the detection limit: <0.50 µg/mL for the 1:5 HD extraction and <0.10 ng/mL for VX.

Table 23. Post-Spike CWA Recovery from Water Rinse

Chemical	Material	Average HD Recovery	HD RSD	Average VX Recovery	VX RSD
10% BE	Paint	97%	0.12%	97%	3.5%
	Sealant	125%	2.8%	101%	1.9%
FWC	Paint	98%	0.80%	95%	4.9%
	Sealant	98%	1.9%	94%	0.65%

Based on these results, no negative or positive interferences for HD or VX were expected during the analysis of water used to rinse the two test chemicals from paint and sealant coupons.

After rinsing was complete, the paint and sealant coupons were removed from the LVAP assemblies and inspected to immediately determine if any residual chemical remained on the coupons and if the coupons were negatively impacted by the chemicals. No obvious residue was observed on any of the coupons, however, the paint coupons that had FWC applied were tacky. Coupons were inspected a second time after 30 minutes, at which point all coupons were dry and the paint/ FWC coupons were no longer tacky. Representative coupons are shown in Figure 12.

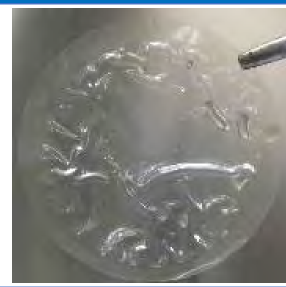
Chemical	Coupons After Rinsing	
	Paint	Sealant
10% BE		
FWC		

Figure 12. Representative Coupons After Water Rinse

Prior to conducting CWA Modification 2 and 3 testing, a study was performed to evaluate use of 10% BE (designated as “Chemical A” or “Chem A” during testing) and FWC (Chem B) to remove HD and VX without the addition of decontaminants, as shown in Table 24. This matrix was performed twice, once using HD with a 24-hour contact time and once using VX with 72-hour contact time. Following the CWA contact time, 1.0 mL of Chem A was applied with a residence time of 10 minutes on the paint film and 60 minutes on the sealant film. Chem B was sprayed onto the coupons with a residence time of 5 minutes. Following the chemical treatment residence time, each coupon was rinsed with 3 x 2.0 mL of water. The water rinses were aggregated and analyzed for HD or VX as described above. Following the water rinse, films were wiped, and the wipe solvent was extracted. The LVAP assemblies were then disassembled and paint/sealant films and SPE disks were solvent extracted. Paint and sealant films were tested in triplicate. Note that each replicate resulted in one wipe sample, one film coupon sample, and one SPE disk sample.

CWA positive control samples were also prepared in triplicate but did not have decontaminant applied; two sets of positive controls were prepared: one without chemical treatment and one with chemical treatment. Procedural blank samples were not contaminated with CWA but were exposed to Chem A or Chem B and processed alongside the test samples. Laboratory blank samples were neither contaminated nor exposed to chemicals and were also processed alongside the test samples. Three spike controls were prepared during each test to confirm the mass of CWA applied to the test samples.

Table 24. CWA Modification 2 Rinse Matrix

Sample Type	Material	CWA Spike Vol.	Chemical	Chemical Residence (Minutes)	Replicates
Test	Paint	2 µL	Chem A	10	3
Test	Paint	2 µL	Chem B	5	3
Test	Sealant	2 µL	Chem A	60	3
Test	Sealant	2 µL	Chem B	5	3
Positive Control	Paint	2 µL	None	10	3
Positive Control	Sealant	2 µL	None	60	3
Positive Control	Paint	2 µL	Chem B	5	3
Positive Control	Sealant	2 µL	Chem B	5	3
Procedural Blank	Paint	NA	Chem A	10	1
Procedural Blank	Paint	NA	Chem B	5	1
Procedural Blank	Sealant	NA	Chem A	60	1
Procedural Blank	Sealant	NA	Chem B	5	1
Lab Blank	Paint	NA	None	NA	1
Lab Blank	Sealant	NA	None	NA	1
Spike Control	None	2 µL	None	NA	3

For Modification 2, the effect of Chem A and Chem B chemical treatment on HD and VX contaminated films prior to bleach application was evaluated as shown in the Table 25 test matrix. This matrix was performed twice, once using HD with a 24-hour contact time and once using VX with 72-hour contact time. Following the CWA contact time, 1.0 mL of Chem A was applied with a residence time of 10 minutes on the paint film and 60 minutes on the sealant film. Chem B was sprayed onto the coupons with a residence time of 5 minutes. Following the chemical treatment residence time, each coupon was rinsed with 3 x 2.0 mL of water. The water rinses were aggregated and analyzed for HD or VX as described above. Bleach was then sprayed onto the coupons with a 60-minute dwell time prior to sample processing. Paint and sealant film coupons were tested in triplicate. Note that each replicate resulted in one wipe sample, one film coupon sample, and one SPE disk sample.

CWA positive control samples were also prepared in triplicate but did not have decontaminant applied; two sets of positive controls were prepared: one without chemical treatment and one with chemical treatment. Procedural blank samples were not contaminated with CWA but were decontaminated and processed alongside the test samples. Laboratory blank samples were neither contaminated nor decontaminated and were also processed alongside the test samples. Procedural blanks and laboratory blanks should have had less than 50% of the lowest detected amount on the test coupon. Three spike controls were prepared during each test to confirm the mass of CWA applied to the test samples. The average recoveries from the spike controls were required to be within 80% to 120% of the theoretical target amount with a replicate RSD of < 30% (n=3).

Table 25. CWA Modification 2 Decontamination Efficacy Matrix

Sample Type	Material	CWA Spike Volume	Decontaminant	Chemical	Chemical Residence (Minutes)	Replicates
Test	Paint	2 µL	Bleach	Chem A	10	3
Test	Paint	2 µL	Bleach	Chem B	5	3
Test	Sealant	2 µL	Bleach	Chem A	60	3
Test	Sealant	2 µL	Bleach	Chem B	5	3
Positive Control	Paint	2 µL	None	None	NA	3
Positive Control	Sealant	2 µL	None	None	NA	3
Positive Control	Paint	2 µL	None	Chem A	10	3
Positive Control	Sealant	2 µL	None	Chem A	60	3
Positive Control	Paint	2 µL	None	Chem B	5	3
Positive Control	Sealant	2 µL	None	Chem B	5	3
Procedural Blank	Paint	NA	Bleach	Chem A	10	1
Procedural Blank	Paint	NA	Bleach	Chem B	5	1
Procedural Blank	Sealant	NA	Bleach	Chem A	60	1
Procedural Blank	Sealant	NA	Bleach	Chem B	5	1
Lab Blank	Paint	NA	None	None	NA	1
Lab Blank	Sealant	NA	None	None	NA	1
Spike Control	None	2 µL	None	None	NA	3

For Modification 3, the effect of Chem A (10% BE) chemical treatment on HD- and VX-contaminated films prior to Dahlgren Decon application was evaluated, as shown in the Table 26 test matrix. This matrix was performed twice, once using HD with a 24-hour contact time and once using VX with 72-hour contact time. Following the CWA contact time, 1.0 mL of Chem A was applied with a residence time of 10 minutes on the paint film coupon and 60 minutes on the sealant film. Following the chemical treatment residence time, each coupon was rinsed with 3 x 2.0 mL of water. The water rinses were aggregated and analyzed for HD and VX as described above. Dahlgren Decon was then sprayed onto the coupons with a 60-minute dwell time prior to sample processing. Paint and sealant films were tested in triplicate. Note that each replicate resulted in one wipe sample, one film coupon sample, and one SPE disk sample.

CWA positive control samples were also prepared in triplicate but did not have decontaminant applied; two sets of positive controls were prepared: one without chemical treatment and one with chemical treatment. Procedural blank samples were not contaminated with CWA but were decontaminated and processed alongside the test samples. Laboratory blank samples were neither contaminated nor decontaminated and were also processed alongside the test samples. Procedural blanks and laboratory blanks should have had less than 50% of the lowest detected amount on the test coupon. Three spike controls were prepared during each test to confirm the mass of CWA applied to the test samples. The average recoveries from the spike controls were required to be within 80% to 120% of the theoretical target amount with a replicate RSD of < 30% (n=3).

Table 26. CWA Modification 3 Decontamination Efficacy Matrix

Sample Type	Material	CWA Spike Vol.	Decontaminant	Chemical	Chemical Residence (Minutes)	Replicates
Test	Paint	2 µL	Dahlgren Decon	Chem A	10	3
Test	Sealant	2 µL	Dahlgren Decon	Chem A	60	3
Positive Control	Paint	2 µL	None	None	NA	3
Positive Control	Sealant	2 µL	None	None	NA	3
Positive Control	Paint	2 µL	None	Chem A	10	3
Positive Control	Sealant	2 µL	None	Chem A	60	3
Procedural Blank	Paint	NA	Dahlgren Decon	Chem A	10	1
Procedural Blank	Sealant	NA	Dahlgren Decon	Chem A	60	1
Lab Blank	Paint	NA	None	None	NA	1
Lab Blank	Sealant	NA	None	None	NA	1
Spike Control	None	2 µL	None	None	NA	3

D.7 Modified Decontamination Efficacy Testing - Pesticides

Table 27 provides the matrix for decontamination efficacy testing to evaluate modifications to decontamination technologies to improve the initially measured efficacies against two pesticides. Use of 10x diluted bleach and D7 evaluated during baseline testing was downselected to just D7 for decontamination approach modifications. The pesticide Modification 1 and 2 test matrix was performed twice, once with malathion and once with fipronil, with both pesticides having a 72-hour contact time on the test coupons. The results from the modifications to the decontamination approach as evaluated for the CWAs (see Section D.6) assisted in the determination of the modifications to the decontamination approaches for the two pesticides.

Two modifications to the decontamination technology were evaluated. Modification 1 involved application of D7 with a 60-minute dwell time, followed by 3 x 2.0 mL water rinses, and then a second application of D7 with a 60-minute dwell time. The water rinses were aggregated and analyzed for malathion or fipronil in a manner similar to water rinse analysis performed for HD and VX. Modification 2 extended the D7 dwell time from 60 minutes to 120 minutes. However, at the 60-minute time point, samples were visually evaluated for dryness to determine if additional D7 should be applied. No samples required D7 reapplication during testing. Paint and sealant films were tested in triplicate. Note that each replicate resulted in one wipe sample, one film coupon sample, and one SPE disk sample.

Pesticide positive control samples were also prepared in triplicate but did not have decontaminant applied. Procedural blank samples were not contaminated with pesticide but were decontaminated and processed alongside the test samples. Laboratory blank samples were neither contaminated nor decontaminated and were also processed alongside the test samples. Procedural blanks and laboratory blanks should have had less than 50% of the lowest detected amount on the test coupon. Three spike controls were prepared during each test to confirm the mass of pesticide applied to the test samples. The average recoveries from the spike controls were required to be within 80% to 120% of the theoretical target amount with a replicate RSD of < 30% (n=3).

Table 27. Pesticide Modification 1 and 2 Decontamination Efficacy Matrix

Sample Type	Material	Pesticide Solution Spike Volume	Decontaminant	Replicates
Test	Paint	12 µL	D7	3
Test	Sealant	12 µL	D7	3
Positive Control	Paint	12 µL	None	3
Positive Control	Sealant	12 µL	None	3
Procedural Blank	Paint	NA	D7	1
Procedural Blank	Sealant	NA	D7	1
Lab Blank	Paint	NA	None	1
Lab Blank	Sealant	NA	None	1
Spike Control	None	12 µL	None	3

For all modification testing, following the CWA/pesticide contact period, decontaminants were sprayed onto the film surfaces as described in Section C.4. Following the decontaminant dwell time, films were wiped, and the wipes were solvent-extracted. The LVAP assemblies were then disassembled and paint/sealant film coupons and SPE disks were solvent-extracted. Wipe, film, and SPE disk extracts were analyzed via GC/MS for HD and malathion or LC-MS/MS for VX and fipronil to quantify residual CWA/pesticide.

E. RESULTS

Results for the wipe sampling, solvent extraction, and decontaminant quench demonstrations are shown in Sections E.1 – E.3. Fate and transport results can be found in Section E.4 while results from the baseline decontamination tests are presented in Sections E.5 (CWAs HD and VX) and E.6 (pesticides malathion and fipronil). Finally, Sections E.7 and E.8 display results for modifications to the baseline decontamination approach for the two CWAs and two pesticides, respectively. Results are provided, as applicable, for spike controls (chemical spiked into extraction solution); for stainless steel coupons (chemical applied to stainless steel and extracted); for paint or sealant coupon (chemical applied to surface of paint/sealant coupon; then surface wiped followed by extraction of the paint or sealant coupon); for chemical recovered in a water rinse; and for the SPE disk below the paint or sealant coupon. Positive control results are associated with results for the surface wipe sampled paint/sealant to which a chemical was applied but was not decontaminated.

E.1 Wipe Sampling Demonstration Results

Wipe sampling testing was completed per Section D.1. The results of HD and VX wipe sampling studies are shown in Table 28; standard deviations (SDs) and RSDs are also provided. The wipe-sampling method was deemed acceptable for use if the average wipe-sampling recoveries were within the range of 70% to 120% of the average of the spike control results with a replicate RSD < 30%. These criteria were met except for the VX recovery from sealant which was different by 1%. As the stainless-steel wipe recovery for VX was within acceptance limits, the wipe-sampling method was deemed acceptable for all CWAs.

Table 28. CWA Wipe Sampling Results

Sample Type	HD Average Mass Recovered ± SD (µg) (n=3)	HD Recovery (vs SC)	HD Recovery RSD	VX Average Mass Recovered ± SD (µg) (n=3)	VX Recovery (vs SC)	VX Recovery RSD
Spike Control	2,600±110	NA	4.1%	1,800±56	NA	3.0%
Paint	1,900±150	76%	7.5%	1,300±240	70%	19%
Sealant	2,000±140	78%	7.2%	1,300±76	69%	6.0%
Stainless Steel	2,600±40	102%	1.6%	1,800±92	95%	5.3%

All CWA wipe quality control data were also acceptable: HD and VX spike controls were within 20% of the theoretical spike mass; recoveries for HD evaporation controls were as follows: paint 90%, sealant 102%, and stainless steel 98%; recoveries for VX evaporation controls were as follows: paint 100%, sealant 95%, and stainless steel 100%; all HD and VX procedural blanks and laboratory blanks were nondetects.

The results of the malathion and fipronil wipe sampling studies are shown in Table 29; SDs and RSDs are also provided. This test was the second malathion wipe trial; the first trial had highly variable spike control recoveries (326% RSD). The results from the first trial (and the first malathion solvent extraction trial, see below) drove the decision to stir the malathion solution prior to and during spiking as described in Section C.1. Wipe acceptance criteria were met except for the malathion recovery from paint and sealant. As the stainless-steel wipe recovery for malathion was within acceptance limits, the wipe sampling method was deemed acceptable for all pesticides.

Table 29. Pesticide Wipe Sampling Results

Sample Type	Malathion Average Mass Recovered ± SD (µg) (n=3)	Malathion Recovery (vs SC)	Malathion Recovery RSD	Fipronil Average Mass Recovered ± SD (µg) (n=3)	Fipronil Recovery (vs SC)	Fipronil Recovery RSD
Spike Control	33±4.4	NA	13%	14±1.0	NA	7.3%
Paint	9.2±1.4	28%	15%	13±1.8	91%	15%
Sealant	18±2.0	56%	11%	16±1.9	115%	12%
Stainless Steel	30±2.3	90%	7.9%	16±1.3	118%	7.9%

Malathion and fipronil spike controls for the second trial were within 20% of the theoretical spike mass. Recoveries for malathion evaporation controls were as follows: paint 56%, sealant 75%, and stainless steel 90%. The low malathion recovery for the paint evaporation controls might reflect poor extraction efficiency; see extraction results in section E.1. Recoveries for fipronil evaporation controls were as follows: paint 112%, sealant 112%, and stainless steel 118%. All malathion and fipronil procedural blanks and laboratory blanks were nondetects.

E.2 Solvent Extraction Demonstration Results

Solvent extraction testing was completed per Section D.2 for extraction of the target chemical from paint and sealant film coupons and SPE disk. The results of HD and VX solvent extraction studies are shown in Table 30; SDs and RSDs are also provided. The solvent extraction method was deemed acceptable for use if the average wipe-sampling recoveries were within the range of 70% to 120% of the average of the spike control results with a replicate RSD < 30%. These criteria were met for both HD and VX. All CWA extraction quality control data were also acceptable: HD and VX spike controls were within 20% of the theoretical spike mass, and all HD and VX laboratory blanks were nondetects.

Table 30. CWA Extraction Sampling Results

Sample Type	HD Average Mass Recovered ± SD (µg) (n=3)	HD Recovery (vs SC)	HD Recovery RSD	VX Average Mass Recovered ± SD (µg) (n=3)	VX Recovery (vs SC)	VX Recovery RSD
Spike Control	2,500±51	NA	2.1%	1,900±80	NA	4.3%
Paint	2,400±230	97%	9.5%	1,700±160	92%	9.1%
Sealant	2,400±32	98%	1.3%	1,700±32	92%	1.9%
SPE	2,500±180	103%	7.1%	2,000±230	108%	11%
Stainless Steel	2,500±59	102%	2.4%	1,600±100	86%	6.3%

The results of the malathion and fipronil solvent extraction studies are shown in Table 31; SDs and RSDs are also provided. This testwas the second malathion solvent extraction trial; the first trial had highly variable spike control recoveries (205% RSD). While the spike control RSD was acceptable for the second malathion extraction trial (18%), the average malathion spike control recovery was only 60% of theoretical. Fipronil spike controls were within 20% of theoretical spike mass. All malathion and fipronil laboratory blanks were nondetects. The recovery criteria for fipronil were met for all sample types, however, low recovery was observed for malathion from paint and sealant, with high RSDs. The decision was made to continue with testing despite the low recoveries and RSDs for malathion.

Table 31. Pesticide Extraction Sampling Results

Sample Type	Malathion Average Mass Recovered ± SD (µg) (n=3)	Malathion Recovery (vs SC)	Malathion Recovery RSD	Fipronil Average Mass Recovered ± SD (µg) (n=3)	Fipronil Recovery (vs SC)	Fipronil Recovery RSD
Spike Control	22±4.0	NA	18%	13±0.23	NA	1.8%
Paint	12±5.4	55%	44%	12±0.21	99%	1.6%
Sealant	12±5.9	53%	50%	13±0.50	101%	3.9%
SPE	19±4.6	85%	24%	14±0.33	109%	2.4%
Stainless Steel	24±6.9	108%	28%	11±2.0	91%	17%

E.3 Decontaminant Quench Demonstration Results

Decontaminant quench testing was performed for CWA per Section D.3. The quench method was deemed acceptable for use if the average recovery of CWA in the quench samples was ≥ 70% relative to the average positive control results. Average HD spike control recovery on Day 0 was 102% versus theoretical and on Day 3 was 88%, with all positive control recoveries greater than 70% compared to the spike controls for both days of analysis, with one exception. The HD SPE positive control exhibited only 39% recovery compared to the spike control on Day 0; this recovery improved to 71% on Day 3. The SPE sample extracts required a 10-fold sample dilution to get the internal standard within acceptance range, which indicated some type of interference from the SPE disk in combination with quench and in the absence of decontaminant. Average VX spike control recovery on Day 0 was 98% versus theoretical and on Day 3 was 95%, with all positive control recoveries greater than 80% compared to the spike controls for both days of analysis. HD and VX process and laboratory blanks were all nondetects.

Table 32 shows the results of the CWA quench study. Average HD recovery compared to the positive controls was greater than 80% for all decontaminants with all materials for all Day 0 and most Day 3 analysis time points, with the D7 SPE average recovery on Day 3 being 69%. The average VX recovery compared to the positive controls was greater than 90% for all decontaminants with for all materials at both the Day 0 and Day 3 analysis time points. Based on these results, we determined that the quench approach was sufficient for both decontaminants in the presence of HD and VX. Also, a 3-day hold time was acceptable if needed for analysis of both CWAs (in the presence of any products resulting from quenching of the decontaminants, including excess quantities of 3 M STS).

Table 32. CWA Quench Recovery

Sample Type	Analysis Time Point	Material	HD Average Mass Recovered ± SD (µg) (n=3)	HD Recovery (vs PC)	VX Average Mass Recovered ± SD (µg) (n=3)	VX Recovery (vs PC)
Positive Control (PC)	Day 0	Wipe	45±1.1	NA	17±0.80	NA
		Coupon	50±1.4	NA	17±0.19	NA
		SPE	20±0.11	NA	19±0.29	NA
Bleach	Day 0	Wipe	46±1.2	102%	16±0.16	92%
		Coupon	48±2.0	97%	17±0.28	101%
		SPE	23±2.4	115%	19±0.59	102%
Dahlgren Decon	Day 0	Wipe	46±0.55	102%	18±0.58	104%
		Coupon	49±1.2	97%	18±0.29	108%
		SPE	22±3.8	113%	20±2.6	110%
D7	Day 0	Wipe	46±2.1	102%	17±1.2	96%
		Coupon	51±1.4	103%	17±0.61	102%
		SPE	16±5.3	81%	18±0.94	98%
Positive Control	Day 3	Wipe	34±1.2	NA	17±0.42	NA
		Coupon	38±1.7	NA	16±0.40	NA
		SPE	31±1.8	NA	18±0.25	NA
Bleach	Day 3	Wipe	36±1.1	106%	16±0.14	94%
		Coupon	39±1.5	102%	16±0.59	99%
		SPE	31±1.7	101%	19±1.1	103%
Dahlgren Decon	Day3	Wipe	35±0.72	103%	17±0.34	104%
		Coupon	37±1.7	99%	18±0.49	113%
		SPE	33±2.3	105%	19±0.22	103%
D7	Day3	Wipe	35±1.2	102%	17±1.3	100%
		Coupon	38±2.0	101%	17±0.91	102%
		SPE	21±17	69%	18±0.49	98%

Decontaminant quench testing was performed for pesticides per Section D.3. The quench method was deemed acceptable for use if the average recovery of pesticide in the quench samples was ≥ 70% relative to the average positive control results. Average malathion spike control recovery on Day 0 was 87% versus theoretical, and on Day 4 was 83%, with all positive control recoveries greater than 90% compared to the spike controls for both days of analysis. Average fipronil spike control recovery on Day 0 was 105% versus theoretical, and on Day 4 was 103%, with all positive control recoveries greater than 85% compared to the spike controls for both days of analysis. Malathion and fipronil process and laboratory blanks were all nondetects.

Table 33 shows the results of the pesticide quench study. Average malathion recovery compared to the positive controls was greater than 80% for both 10x diluted bleach and D7 with all materials at both the Day 0 and Day 4 analysis time points. Similarly, the average fipronil recovery compared to the positive controls was greater than 85% for both 10x diluted bleach and D7 with all materials at both the Day 0 and Day 4 analysis time points. Based on these results, we determined that the quench approach was sufficient for both decontaminants in the presence of malathion and fipronil. Also, a 4-day hold time was acceptable if needed for analysis of both pesticides (in the presence of any products resulting from quenching of the decontaminants, including excess quantities of 3 M STS).

Table 33. Pesticide Quench Recovery

Sample Type	Analysis Time Point	Material	Malathion Average Mass Recovered ± SD (µg) (n=3)	Malathion Recovery (vs PC)	Fipronil Average Mass Recovered ± SD (µg) (n=3)	Fipronil Recovery (vs PC)
Positive Control	Day 0	Wipe	36±5.7	NA	13±1.4	NA
		Coupon	32±6.4	NA	13±1.5	NA
		SPE	30±9.6	NA	14±0.78	NA
10x Diluted Bleach	Day 0	Wipe	30±3.7	85%	12±0.80	88%
		Coupon	26±4.2	83%	13±1.3	97%
		SPE	30±5.7	87%	15±1.8	102%
D7	Day 0	Wipe	34±11	97%	13±0.78	95%
		Coupon	29±3.1	92%	14±2.6	102%
		SPE	29±5.4	99%	15±0.87	102%
Positive Control	Day 4	Wipe	35±5.1	NA	13±1.3	NA
		Coupon	31±5.9	NA	13±1.3	NA
		SPE	32±11	NA	13±0.63	NA
10x Diluted Bleach	Day 4	Wipe	29±3.8	81%	12±1.0	90%
		Coupon	26±4.9	83%	13±0.92	99%
		SPE	33±9.2	103%	14±1.3	106%
D7	Day 4	Wipe	32±9.4	91%	13±0.94	100%
		Coupon	28±2.5	92%	14±2.6	103%
		SPE	30±5.7	92%	15±0.87	110%

E.4 Fate and Transport Assessment

HD and VX fate and transport testing was performed per Section D.4. Both HD and VX primarily beaded on paint film coupons or formed a pancake shape on sealant film coupons when spiked. Both HD and VX caused a blistering of all test films after the 24- or 72-hour agent contact period. Representative photographs of VX interactions on films are shown in Figure 13, and HD interactions on films are shown in Figure 14. Note that color differences in the photos were due to limitations of photography and should not be taken to represent actual changes in film coloration.

			
Droplet Beaded after Spiking on Paint	Droplet Blistered after 72-hour Contact on Paint	Droplet Pancake Shape after Spiking on Sealant	Droplet Blistered after 72-hour Contact on Sealant

Figure 13. VX Interaction on Paint and Sealant Films

			
Droplet Beaded after Spiking on Paint	Droplet Blistered after 24-hour Contact on Paint	Droplet Pancake Shape after Spiking on Sealant	Droplet Blistered after 24-hour Contact on Sealant

Figure 14. HD Interaction on Paint and Sealant Film Coupons

Average HD spike control mass was 1,800 µg (72% of theoretical) with a 60% RSD; the mass of the first spike control replicate was 20% of the mass of the other two replicates, indicating a possibly mis-spiked sample. The low spike control sample was the first prepared sample in the trial. It is possible that an air bubble in the syringe caused this error. No other samples in the trial resulted in apparent low spike levels. Note that all laboratory blanks were nondetects for this testing. The results of the fate and transport are shown in Table 34 for paint and sealant coupons. For both the paint and sealant coupons, most of the HD permeated through the coupon to the SPE disk, with less than 1% of all HD collected from the surface on wipes. The total HD amount recovered is comparable to the spiked amount, which suggests that HD does not degrade appreciable while the evaporation is significantly reduced due to the competing permeation into the paint or sealant layer.

Average VX spike control mass was 1,800 µg (95% of theoretical) with a 1.8% RSD. While there were very low level VX detections for some laboratory blank samples, all laboratory blanks met the quality control criteria of less than 50% of the lowest detected amount on the test coupon. The results of the fate and transport study are shown in Table 34 for paint and sealant coupons. For both the paint and sealant coupons, most of the VX was found within the coupon. The total VX amount recovered from the paint sample is comparable to the spiked amount while only half of the VX was recovered from the sealant material. Considering the low volatility of VX, it is plausible that VX degrades upon interaction with the sealant rather than being lost due to evaporation. However, it is also possible that VX adheres more strongly to the sealant layer over time (72 hours) leading to poorer extraction efficiencies. The conducted research cannot decouple these two interpretations of the lower total VX recovery from the sealant sample.

Table 34. CWA Fate and Transport Results

Analyte	Sample Type	Average Mass Recovered ± SD (µg) [% of Total]			
		Wipe	Coupon	SPE	Total
HD	Paint	14±2.9 [0.7%]	520±320 [26%]	1,500±390 [75%]	2,000±130
	Sealant	1.2[1] [0.06%]	380±91 [20%]	1,500±370 [79%]	1,900±270
VX	Paint	440±201 [26%]	1,000±247 [59%]	290±177 [17%]	1,700±68
	Sealant	69±5.2 [7.5%]	650±87 [71%]	200±234 [22%]	920±155

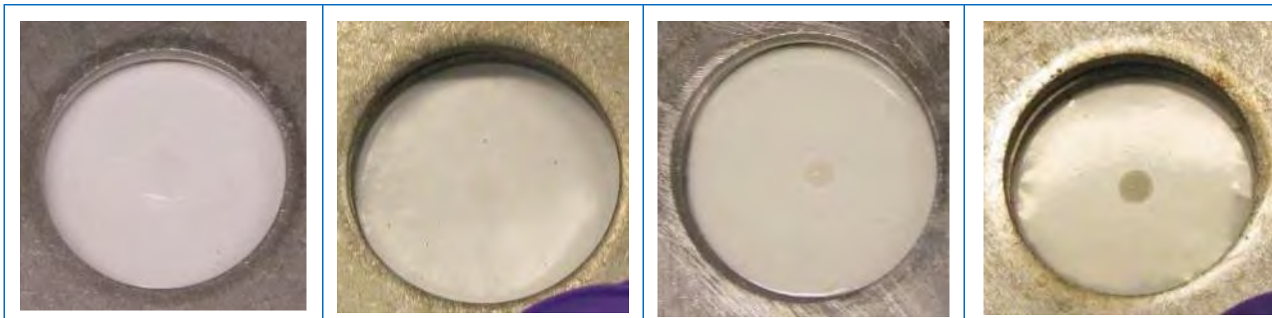
[1] One replicate detection (1.3 µg) averaged with two nondetect replicates set equal to quantitation limit (1.1 µg)

Based on the observed results, the decision was made to conduct all decontamination testing with a 24-hour HD contact time and 72-hour VX contact time as performed previously.

Malathion and fipronil fate and transport testing was performed per Section D.4. Both malathion and fipronil beaded or formed a pancake shape on the paint and sealant film coupons when spiked. The malathion solution was dry with a visible spot or blister where the droplet was applied on test film coupons after the 24- or 72-hour contact period. The fipronil solution was dry with a visible spot where the droplet was applied on all test films after the 24- or 72-hour contact period. Representative photographs of the malathion solution interaction on film coupons are shown in Figure 15, and fipronil solution interactions on film coupons are shown in Figure 16.

Droplet Beaded after Spiking on Paint	Droplet Blistered after 72-hour Contact on Paint	Droplet Beaded after Spiking on Sealant	Dry Visible Spot after 72-hour Contact on Sealant

Figure 15. Malathion Interactions on Paint and Sealant Film Coupons



Droplet Pancake Shape after Spiking on Paint	Dry Visible Spot after 72-hour Contact on Paint	Droplet Beaded after Spiking on Sealant	Dry, Visible Spot after 72-hour Contact on Sealant

Figure 16. Fipronil Interactions on Paint and Sealant Films

Average malathion spike control mass was 31 µg (85% of theoretical) with a 13% RSD. All laboratory blanks were nondetects. The results of the fate and transport comparison of a 24-hour malathion contact time to a 72-hour contact time are shown in Table 35 for paint and sealant. A *t*-test was performed to compare the 24-hour and 72-hour contact times; the *t*-test was two-tailed for heteroscedastic (i.e., different variances) data. Similar behavior was observed for malathion on the paint films However, significantly less malathion was recovered from the sealant wipe after 72 hours. A 72-hour contact time was selected for malathion, representing a potentially conservative scenario in the field while still providing sufficient target chemical for surface analysis.

Table 35. Malathion Fate and Transport Results

Sample Type	Material	Average Mass Recovered ± SD (µg) [% of Total]		*t*-Test *P* Value	Significant Difference
		24-Hour	72-Hour		
Paint	Wipe	2.3±0.35 [12%]	1.6±0.33 [8%]	0.081	No
	Coupon	18±4.5 [90%]	18±6.6 [90%]	0.99	No
	SPE	<1.0 [<5%]	<1. 0 [<5%]	NA	
	Total	20±4.8	20±7.0	0.91	No
Sealant	Wipe	9.9±0.91 [58%]	2.8±0.45 [23%]	0.0015	Yes
	Coupon	7.5±2.3 [44%]	8.8±1.8 [73%]	0.46	No
	SPE	<1.0 [<6%]	<1.0 [<8%]	NA	
	Total	17±1.8	12±2.2	0.028	Yes

Figure 17 shows the total recovered malathion for the paint and sealant 24-hour and 72-hour contact times with error bars of one standard deviation.

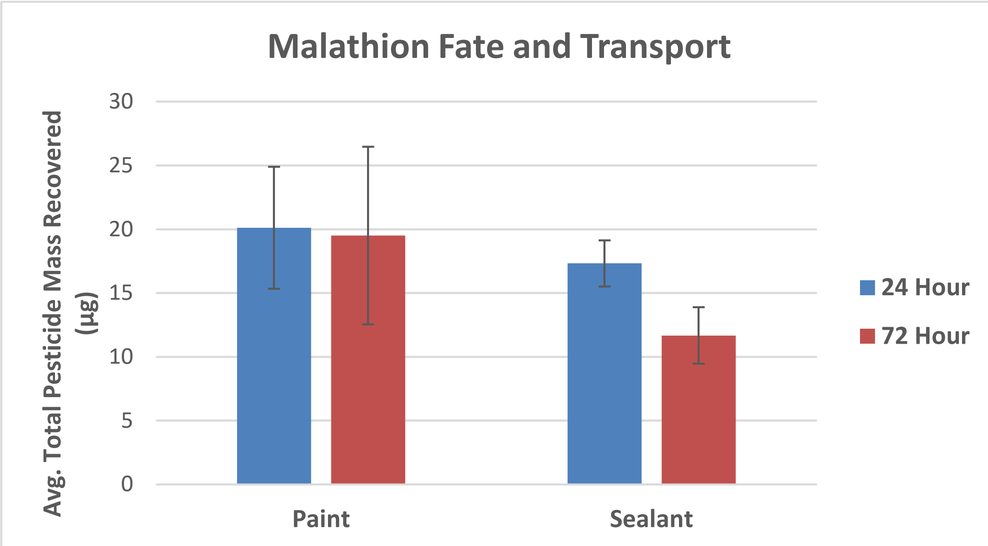


Figure 17. Total Recovered Malathion for Fate and Transport Testing

Average fipronil spike control mass was 13 µg (92% of theoretical) with a 1.4% RSD. All laboratory blanks were nondetects. The results of the fate and transport comparison of a 24-hour fipronil contact time to a 72-hour contact time are shown in Table 27 for paint and sealant coupons. A Student's *t*-test was performed to compare the 24-hour and 72-hour contact times; the *t*-test was two-tailed for heteroscedastic data. Similar behavior was observed for fipronil on the sealant film coupons. However, significantly less fipronil was recovered from the paint coupon wipe after 72 hours. A 72-hour contact time was selected for fipronil representing a potentially conservative scenario in the field while still providing sufficient target chemical for surface analysis.

Table 36. Fipronil Fate and Transport Results

Sample Type	Material	Average Mass Recovered ± SD (µg) (n=3)		*t*-Test *P* Value	Significant Difference
		24-Hour	72-Hour		
Paint	Wipe	10±0.43	7.2±0.51	0.0019	Yes
	Coupon	3.0±0.77	4.7±0.0076	0.057	No
	SPE	<0.001	<0.001	NA	
	Total	13±0.84	12±0.55	0.12	No
Sealant	Wipe	9.7±2.2	9.4±0.46	0.85	No
	Coupon	1.7±0.64	2.3±0.25	0.25	No
	SPE	0.0019[1]	<0.001	NA	
	Total	11±1.6	12±0.28	0.77	No

[1] One replicate detection (0.005 µg) averaged with two nondetect replicates equal to quantitation limit (0.001 µg)

Figure 18 shows the total recovered fipronil for the paint and sealant coupon 24-hour and 72-hour contact times with error bars of one standard deviation.

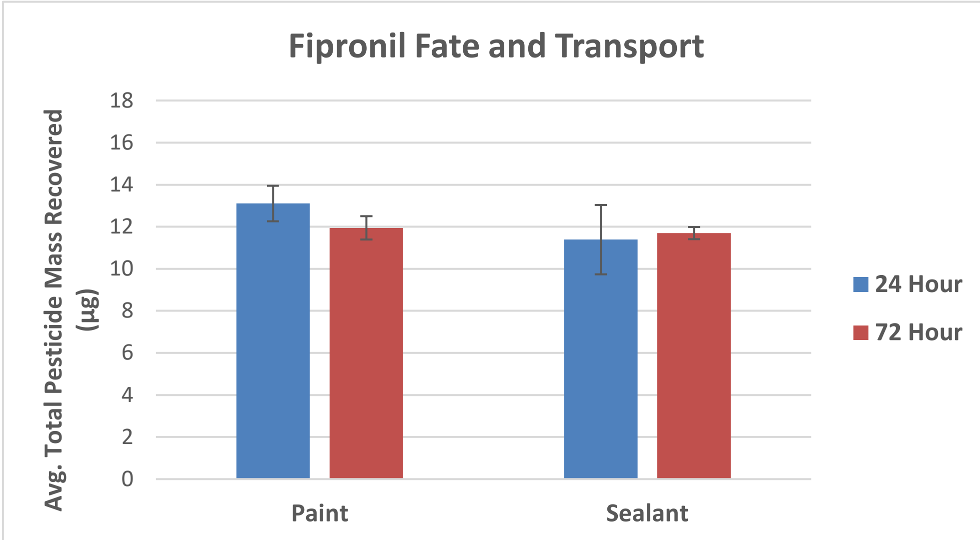


Figure 18. Total Recovered Fipronil for Fate and Transport Testing

E.5 Baseline Decontamination - CWAs

During CWA testing, decontaminants were sprayed onto the surfaces of paint and sealant film coupons to coat the entire exposed surface with decontaminant. The bleach wetted the surface of the paint film coupons and beaded on the surface of the sealant film coupons. The Dahlgren Decon and D7 decontaminants both foamed when applied to the surface of the film coupons. During baseline and Mod 1 testing, decontaminants appeared mostly dry and crusty after the 60-minute dwell period. During Mod 2 and Mod 3 testing, decontaminants appeared wet. The difference in observed decontaminant wetness over time may have been related to slightly higher laboratory RH during Mod 2 and 3 testing (refer to Appendix B). Representative photographs of the decontamination observations on the films are shown in Figure 19 and Figure 20.

			
Bleach Applied to Paint	Bleach Applied to Sealant	Dahlgren Applied to Paint	D7 Applied to Paint

Figure 19. Decontaminant Observations when Applied to Film Coupons

	
Dry, Crusty Decontaminant (Dahlgren on Paint)	Wet Decontaminant (Bleach on Sealant)

Figure 20. Decontaminant Observations after 60-Minute Dwell Time

During pesticide testing, the 10x diluted bleach wetted the surface of the paint film coupons and beaded on the surface of the sealant film coupons. The D7 decontaminant foamed when applied to the surface of the film coupons. These observations were consistent with the decontamination application observations from chemical agent testing, and representative photos are shown in Figure 19. The 10x diluted bleach looked wet after the 60-minute dwell period, and the D7 decontaminant looked wet and foamy after the 60-minute or 120-minute dwell periods. Representative photographs of the decontaminant observations after the dwell time are shown in Figure 21.

	
Wet Foamy D7 on Sealant	Wet 10x Bleach on Sealant

Figure 21. Sealant Film Coupon Decontaminant Observations after Dwell Time

After the 60-minute dwell period for 10x diluted bleach, paint films looked wrinkled, and sealant films looked unchanged. After the 60-minute or 120-minute dwell periods for D7, paint films looked wrinkled, and sealant films looked unchanged. For the trials using fipronil, a yellow spot was present where the pesticide was applied after the 10x diluted bleach and D7 dwell periods. Representative photographs of the paint film observations after decontamination are shown in Figure 22.

	
Wrinkled Paint Coupon after 60-min 10x Diluted Bleach Dwell Period	Fipronil Yellow Spot on Paint Coupon after 60-min D7 Dwell Period

Figure 22. Paint Film Decontaminant Observations after Dwell Time

The CWA baseline testing included two trials, one for HD and one for VX. Each trial included two test materials (paint and sealant film coupons) and three decontaminants (bleach, Dahlgren Decon, and D7) with a 60-minute decontaminant dwell time. All CWA spike control results (shown in Table 37) met the acceptance criteria described in Section D.5.

Table 37. Baseline Decontaminant Efficacy Testing – CWA Average Spike Control Results

CWA	Average Mass Recovered (µg)	Percent Recovery (vs Theoretical)*	RSD
HD	2,400	93%	2.8%
VX	2,100	113%	5.5%
* See Table 1 for purity and mass contamination targets of 2,540 µg for HD and 2,016 µg for VX based on density			

All HD and VX laboratory blank results and a majority of the procedural blank results met the acceptance criteria described in Section D.5. One procedural blank had a low-level result for HD (slightly above the detection limit); no impact to the data was expected. Chromatographic peak splitting was observed for all HD SPE laboratory and procedural blanks. Hence, samples were diluted 2:1 to mitigate the matrix effect for these blanks. Internal standard responses for all HD and VX results met the acceptance criteria described in Section C.7 and C.8.

The average HD mass recoveries for each sample component for the positive controls and decontaminants, as well as total mass (combined recoveries of the wipe, coupon, and SPE) are provided in Table 38 for paint film coupons and Table 39 for sealant film coupons. Decontaminant efficacy for just the wipe samples collected from the coupon surface (surface decontamination efficacy) was calculated using Equation 5. Total decontamination efficacy against all test components (wipe, extracted coupon, and SPE) was calculated by comparing the summed total of the average mass recovered for all components for each decontaminant test to the summed total of the average massed recovered for all components for the Positive Controls using Equation 4. The surface decontamination efficacy and total decontamination efficacy error, calculated using Equation 7, is also provided as a ± percentage. Note that results for all measured target analyte masses are considered accurate to two significant figures. Reported values, including total average mass recovered and decontaminant efficacy, are calculated prior to rounding. Small differences in presented data are due to rounding.

If replicate results for the positive controls and/or decontaminants were below the detection limit, the standard deviation, and associated percent efficacy and/or error were not calculated. In these cases, a standard deviation is not provided. For bleach and Dahlgren paint testing, all replicate wipe results were below the quantitation limit. For calculation purposes, the quantitation limit (1.1 µg) was used, and wipe efficacy results were expressed with > symbol. For Dahlgren and D7 paint testing the average mass recovered for the decontaminant samples was greater than the average total mass recovered for positive controls; therefore, the total efficacy is reported as 0%.

Table 38. Average HD Mass Recovered for Paint Film Coupons - Baseline

Sample Type	Average Mass Recovered ± SD (µg)				Decontamination Efficacy	
	Wipe	Coupon	SPE	Total	Surface	Total
Positive Control	6.1±2.8	320±130	880±120	1,200±250	NA	NA
Bleach	<1.1	110±44	1,000±200	1,100±230	>82%	6.1±27%
Dahlgren	<1.1	310±110	1,000±110	1,300±20	>82%	0%
D7	2.2±0.68	240±80	1,000±69	1,300±72	64±20%	0%
NA = Not Applicable						

Table 39. Average HD Mass Recovered for Sealant Film Coupons - Baseline

Sample Type	Average Mass Recovered ± SD (µg)				Decontamination Efficacy	
	Wipe	Coupon	SPE	Total	Surface	Total
Positive Control	<1.1	190±74	1,100±57	1,300±38	NA	NA
Bleach	<1.1	190±17	1,100±106	1,300±92	NA	0%
Dahlgren	<1.1	210±19	1,100±110	1,300±110	NA	0%
D7	3.0±0.55	270±62	1,000±12	1,300±65	0%	0%
NA = Not Applicable						

For bleach, Dahlgren, and D7 sealant testing, the average total mass recovered for the decontaminant samples was greater than the average total mass recovered for positive controls. Therefore, the total decontamination efficacy is reported as 0%. Similarly, the average total mass recovered for the D7 wipe samples was greater than the average wipe mass recovered for positive controls; therefore, the surface decontamination efficacy is reported as 0%.

Some observations from the results in Table 38 and Table 39 include that minimal HD remained on the surface of film coupons following the contact period and that most of the HD (> 70%) penetrated through all film coupons to the SPE disk. There was minimal difference between positive controls and decontaminant samples for either coupon type. Total HD decontamination efficacy was less than 7% for both film types, although surface decontamination efficacy indicated that at least 60% of surface HD was decontaminated for decontaminant types. See

Appendix D for statistical interpretation of the HD baseline results and discussion of whether decontaminants had a significant effect on HD levels. See also Section E.7 for a summary of statistical observations.

Figure 23 summarizes the paint and sealant HD average total mass recoveries including error bars equal to one standard deviation.

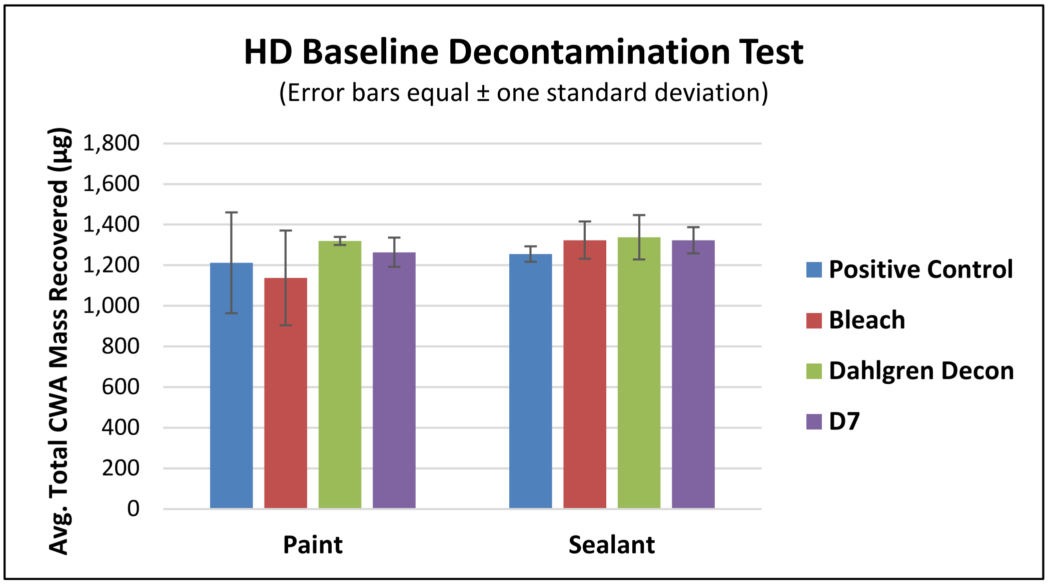


Figure 23. HD Baseline Decontaminant Test, Average Total Mass Recoveries

The average VX mass recoveries for each sample component for the positive controls and decontaminants, as well as total mass (combined recoveries of the wipe, coupon, and SPE) are provided in Table 40 for paint film coupons and Table 41 for sealant film coupons. The average total mass recovered for the bleach paint wipe samples was greater than the average wipe mass recovered for positive controls; therefore, the surface decontamination efficacy was reported as 0%. For the paint samples, no decontaminants appeared to reduce the level of VX compared to the positive control; approximately the same mass of VX remained on the surface of all coupons, and approximately equal masses of VX remained within all coupons and penetrated to the SPE disk. Total VX decontamination on paint range from only 4% to 16%, with surface decontamination ranging from 0% to 50%.

Table 40. Average VX Mass Recovered for Paint Films - Baseline

Sample Type	Average Mass Recovered ± SD (µg)				Decontamination Efficacy	
	Wipe	Coupon	SPE	Total	Surface	Total
Positive Control	92±30	290±24	230±93	620±94	NA	NA
Bleach	140±45	230±86	150±27	520±14	0%	16±13%
Dahlgren	46±11	230±32	320±29	590±69	50±20%	4.3±18%
D7	70±11	190±23	330±80	580±46	24±27%	6.0±16%

The average total mass recovered for the D7 sealant wipe samples was greater than the average wipe mass recovered for positive controls; therefore, the surface decontamination efficacy was reported as 0%. For the sealant samples, bleach and Dahlgren Decon both appeared to reduce the mass of VX on the surface relative to the positive controls and the majority of VX (> 90%) was within the coupon. Total VX decontamination efficacy was better on sealant than paint, with 50% efficacy measured for bleach. Surface decontamination ranged from a low of 0% for D7 up to 96% and 97% for bleach and Dahlgren Decon, respectively.

Table 41. Average VX Mass Recovered for Sealant Films - Baseline

Sample Type	Average Mass Recovered ± SD (µg)				Decontamination Efficacy	
	Wipe	Coupon	SPE	Total	Surface	Total
Positive Control	31±4.0	820±49	8.7±14	860±65	NA	NA
Bleach	1.1±2.0	420±101	9.9±17	430±107	96±6.4%	50±13%
Dahlgren	0.97±0.76	730±72	23±27	750±52	97±2.5%	12±8.9%
D7	42±2.0	570±39	15±14	630±50	0%	27±8.0%

See Appendix D for statistical interpretation of the VX baseline results and discussion of whether decontaminants had a significant effect on VX levels. See also Section E.7 for a summary of statistical observations.

Figure 24 summarizes the paint and sealant VX average total mass recoveries including error bars equal to one standard deviation.

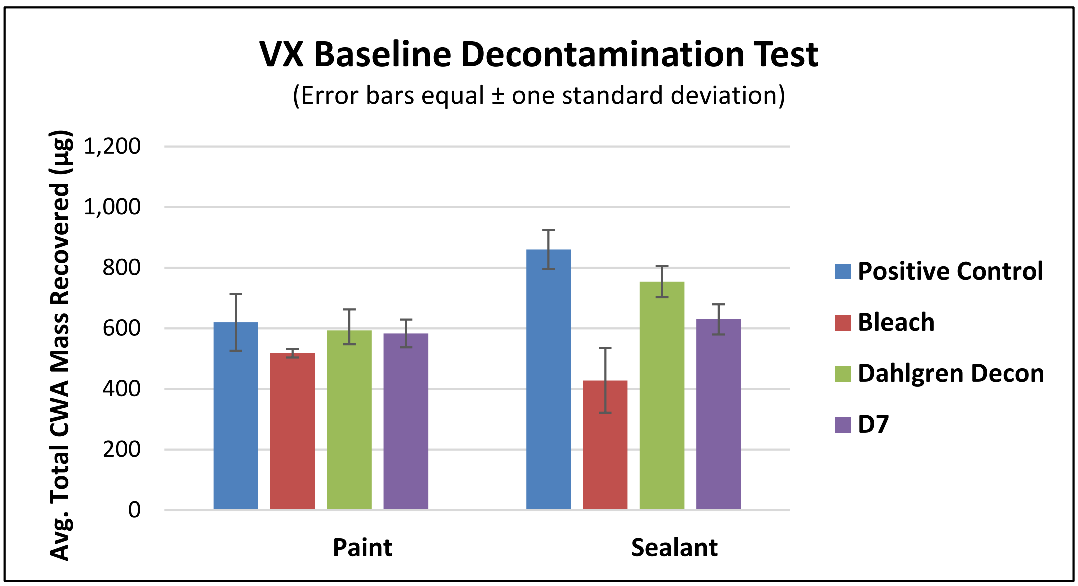


Figure 24. VX Baseline Decontaminant Test, Average Total Mass Recoveries

E.6 ***Baseline Decontamination - Pesticides***

The baseline testing for pesticides included two trials, one for malathion and one for fipronil. Each trial included two test materials (paint and sealant film coupons) and two decontaminants (10x diluted bleach and D7) with a 60-minute decontaminant dwell time.

Pesticide spike control results are shown in Table 42. The malathion spike controls did not meet the acceptance criteria and had an average of 51% recovery and 38% RSD. No reason for the low recovery and high RSD could be ascertained other than possibly nonrepresentative samples being collected from the stirred malathion solution. All the fipronil spike controls met the acceptance criteria described in Section D.5.

Table 42. Pesticide Average Spike Control Results - Baseline

Pesticide	Average Mass Recovered (µg)	Percent Recovery (vs Theoretical)	RSD
Malathion	19	51%	38%
Fipronil	13	89%	1.1%

One laboratory blank wipe had a low-level result for fipronil (slightly above the detection limit), no impact to the data was expected. All other laboratory blank results and procedural blank results met the acceptance criteria for both malathion and fipronil described in Section D.5. Internal standard response for all malathion and fipronil baseline testing results met the acceptance criteria described in Section C.7 and C.8. Most of the wipe and coupon procedural blanks required a 100-fold dilution (rather than the typical 10-fold) to ensure IS response was acceptable.

The average malathion mass recoveries for each sample component for the positive controls and decontaminants, as well as total mass (combined recoveries of the wipe, coupon, and SPE) are provided in Table 43 for paint film coupons and Table 44 for sealant film coupons. Note that the D7 paint wipe samples needed to be diluted 10-fold to get the internal standard within the acceptance range. Because all three wipe samples were nondetects at this dilution level, the wipe results are reported as < 11 µg due to the elevated quantitation limit. The average total mass recovered for the 10x diluted bleach and D7 paint wipe samples was greater than the average wipe mass recovered for positive controls; therefore, the surface decontamination efficacy was reported as 0%. For the paint samples, 10x diluted bleach and D7 coupons were not different from the positive controls. Total malathion decontamination efficacy was less than 7% for paints.

Table 43. Average Malathion Mass Recovered for Paint Film Coupons - Baseline

Sample Type	Average Mass Recovered ± SD (µg)				Decontamination Efficacy	
	Wipe	Coupon	SPE	Total	Surface	Total
Positive Control	<1.1	12±4.6	<1.0	12±4.6	NA	NA
10x Diluted Bleach	1.1[1]	10±1.0	<1.0	12±1.7	0%	6.8±37%
D7	<11	12±3.7	<1.0	12±3.7	0%	5.5±46%

[1]SD could not be calculated; only one replicate mass above the quantitation limit

Note that the D7 sealant wipe samples needed to be diluted 5-fold to get the internal standard within the acceptance range. Because all three wipe samples were nondetects at this dilution level, the wipe results are reported as < 5.5 µg due to the elevated quantitation limit. For 10x diluted bleach sealant testing, all replicate wipe, coupon, and SPE results were below the quantitation limit. For calculation purposes, the quantitation limit (1.1 µg) was used, and surface decontamination efficacy results and 3.1 µg were used for the Total Decontamination Efficacy results. Both 10x diluted bleach efficacy results were expressed with > symbol. The average mass recovered for the D7 sealant wipe samples and D7 sealant total was greater than the average wipe mass recovered for the corresponding positive controls; therefore, the surface decontamination efficacy and Total Decontamination Efficacy was reported as 0%. Total malathion decontamination efficacy may be above 28% for sealant using 10x diluted bleach.

Table 44. Average Malathion Mass Recovered for Sealant Films - Baseline

Sample Type	Average Mass Recovered ± SD (µg)				Decontamination Efficacy	
	Wipe	Coupon	SPE	Total	Surface	Total
Positive Control	3.2±1.6	1.1[1]	<1.0	4.3±0.93	NA	NA
10x Diluted Bleach	<1.1	<1.0	<1.0	<3.1	>66 %	>28 %
D7	<5.5	5.0±0.32	<1.0	5.0±0.32	0%	0%

[1]SD could not be calculated; only one replicate mass above the quantitation limit

See Appendix E for statistical interpretation of the malathion baseline results and discussion of whether decontaminants had a significant effect on malathion levels. See also Section E.8 for a summary of statistical observations.

In addition to malathion analysis, samples were analyzed for malaoxon, a malathion degradation product. Several wipe and coupon samples using 10x diluted bleach had malaoxon present; results are shown in Table 45. No other malathion samples contained measurable malaoxon above the quantitation limit.

Table 45. Malaoxon Mass Recovered - Baseline

Sample Type	Film	Mass Recovered (µg)	
		Wipe	Coupon
10x Diluted Bleach	Paint Rep 1	1.1	1.2
	Paint Rep 2	Not Detected	1.2
	Paint Rep 3	1.2	1.2
	Sealant Rep 2	1.2	1.0

Figure 25 summarizes the paint and sealant malathion average total malathion mass recoveries including error bars equal to one standard deviation. Note that for 10x diluted bleach on sealant, the quantitation limit was 3.1 ng.

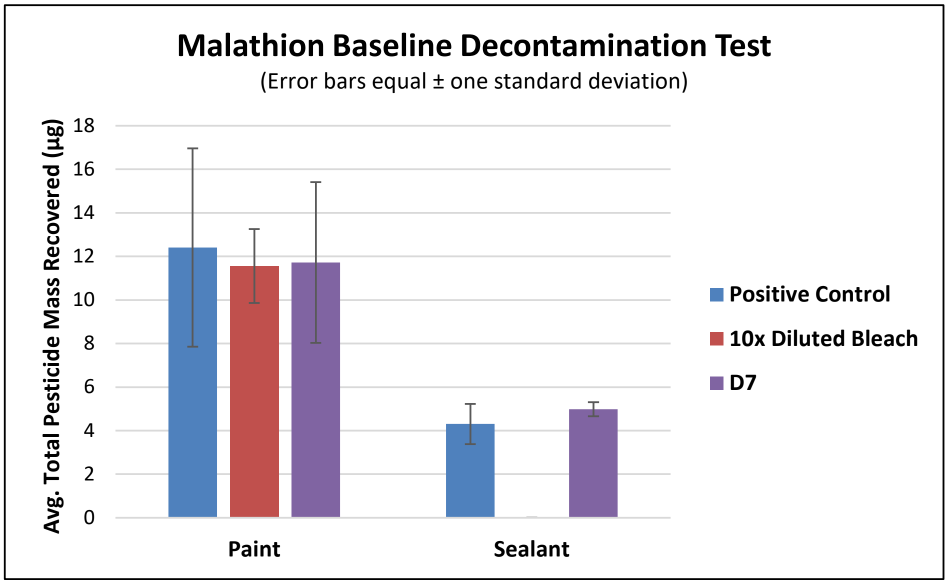


Figure 25. Malathion Baseline Decontaminant Test, Average Total Mass Recoveries

The average fipronil mass recoveries for each sample component for the positive controls and decontaminants, as well as total mass (combined recoveries of the wipe, coupon, and SPE) are provided in Table 46 for paint films and Table 47 for sealant films. Total fipronil decontamination efficacy was much better for fipronil than for malathion, with greater than 90% efficacy with D7 on both films and greater than 99% efficacy for 10x diluted bleach on sealant.

Table 46. Average Fipronil Mass Recovered for Paint Film Coupons - Baseline

Sample Type	Average Mass Recovered ± SD (µg)				Decontamination Efficacy	
	Wipe	Coupon	SPE	Total	Surface	Total
Positive Control	8.0±0.62	2.8±0.51	<0.001	11±0.39	NA	NA
10x Diluted Bleach	4.2±1.1	2.8±0.25	<0.001	6.9±1.3	48±14%	36±13%
D7	0.057±0.025	0.69±0.040	<0.001	0.75±0.022	99.3±0.31%	93±0.33%

Table 47. Average Fipronil Mass Recovered for Sealant Film Coupons - Baseline

Sample Type	Average Mass Recovered ± SD (µg)				Decontamination Efficacy	
	Wipe	Coupon	SPE	Total	Surface	Total
Positive Control	9.6±0.27	1.5±0.16	<0.001	11±0.14	NA	NA
10x Diluted Bleach	0.068±0.055	0.037±0.012	<0.001	0.10±0.061	99.3±0.57%	99.1±0.55%
D7	0.83±0.29	0.15±0.041	<0.001	0.98±0.33	91±3.0%	91±3.0%

See Appendix E for statistical interpretation of the fipronil baseline results and discussion of whether decontaminants had a significant effect on fipronil levels. See also Section E.8 for a summary of statistical observations.

Figure 26 summarizes the paint and sealant fipronil average total mass recoveries including error bars equal to one standard deviation.

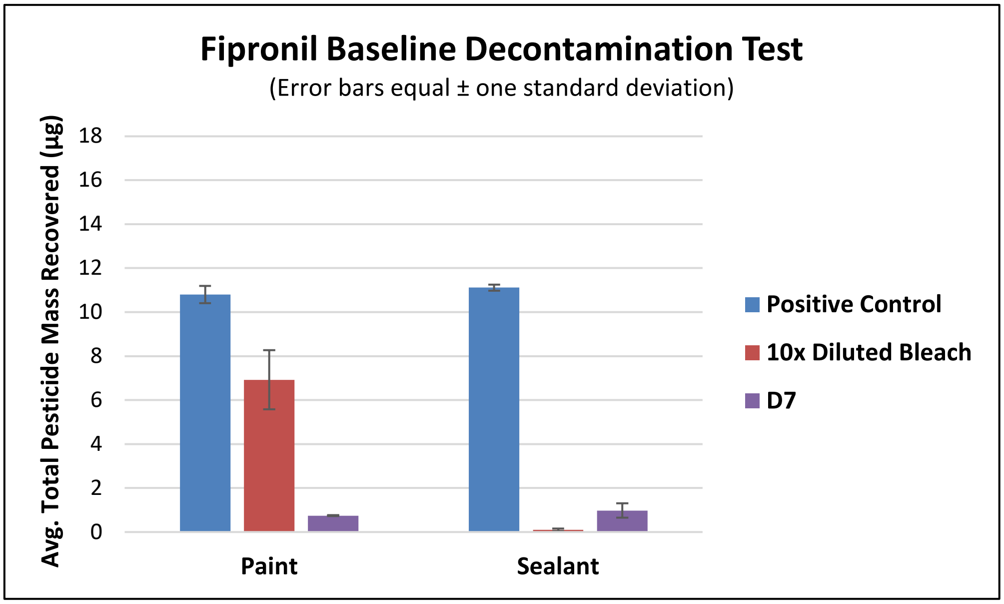


Figure 26. Fipronil Baseline Decontaminant Test, Average Total Mass Recoveries

E.7 CWA Modification Decontamination Testing

CWA modification testing included a total of ten trials, five each for HD and VX. Each trial included two test materials (paint and sealant film coupons) and each of the five decontaminant modification tests described below:

- Modification 1 – two decontaminants tested (bleach and D7 for HD, bleach and Dahlgren for VX). Each decontaminant was applied twice with a 60-minute dwell time following each application.
- Modification 2 (Evaluation) – Evaluation of Chemical A (10% butoxyethanol) and Chemical B (Zep Foaming Wall Cleaner) application followed by triple water rinse. No decontaminants added during these trials.
- Modification 2 (Chem A) - Chemical A application and triple water rinse followed by a single application of bleach with a 60-minute dwell time.
- Modification 2 (Chem B) - Chemical B application and triple water rinse followed by a single application of bleach with a 60-minute dwell time.
- Modification 3 (Chem A) - Chemical A application and triple water rinse followed by a single application of Dahlgren Decon with a 60-minute dwell time.

During Mod 2 and Mod 3 testing, Chem A and Chem B were applied to the films before decontamination. The paint film coupons looked wrinkled after Chem A and Chem B application. The agent blister on sealant film coupons looked more pronounced after Chem A application; no change was observed on the sealant films after Chem B application.

Representative photographs of the Chem A and Chem B interactions with the films are shown in Figure 27.

Paint, Chem A	Paint, Chem B	Sealant, Chem A

Figure 27. Chem A and B Observations

All HD and VX spike control results (shown in Table 48) met the acceptance criteria described in Section D.6. All HD and VX laboratory blank results, and most of the procedural blank results met the acceptance criteria described in Section D.6. Several procedural blanks in the Modification 2 Chem A and Chem B tests had low-level results for VX; no impact to the data was expected. Internal standard response for all CWA modification testing results met the acceptance criteria described in Sections C.7 and C.8. Peak splitting was observed for Modification 1 HD SPE laboratory and procedural blanks; samples were diluted 2:1 to mitigate the matrix effect. For Dahlgren Decon procedural blanks (Modification 1 and Modification 3 tests), samples required a 100-fold or 1000-fold dilution as opposed to typical 10-fold dilution to get internal standard response within acceptance criteria.

Table 48. Average CWA Spike Control Results – Modification Testing

CWA	Test	Average Mass Recovered (µg)	Percent Recovery (vs Theoretical)	RSD
HD	Mod 1	2,200	87%	5.3%
	Mod 2 - Evaluation	2,700	106%	6.4%
	Mod 2 – Chem A	2,600	103%	2.4%
	Mod 2 – Chem B	2,700	105%	1.1%
	Mod 3 – Chem A	2,800	113%	2.2%
VX	Mod 1	1,900	99%	2.1%
	Mod 2 - Evaluation	1,900	101%	7.0%
	Mod 2 – Chem A	1,900	103%	9.0%
	Mod 2 – Chem B	1,800	95%	4.1%
	Mod 3 – Chem A	1,800	96%	4.0%

The average HD mass recoveries for each sample component for the positive controls and decontaminants, as well as total mass (combined recoveries of the rinse, wipe, coupon, and SPE) are provided in Table 49 for paint film coupons and Table 50 for sealant film coupons. The efficacy calculations for Modification 2 and Modification 3 are calculated against the Positive Control without addition of Chem A or B (not the Positive with Chem A or Positive with Chem B). For all modification paint tests, all replicate wipe results were below the quantitation limit. For calculation purposes, the quantitation limit (1.1 µg) was used, and surface decontamination efficacy results were expressed with the > symbol. All test modifications resulted in improved total HD decontamination efficacy for paint film coupons compared to baseline testing, although efficacies still ranged from only 24 to 35%. As all wipe samples were below the quantitation limit, the surface decontamination efficacy for all tests was generally high (> 85%).

Table 49. Average HD Mass Recovered for Paint Films – Modification Testing

Test	Sample Type	Average Mass Recovered ± SD (µg)					Decontamination Efficacy	
		Rinse	Wipe	Coupon	SPE	Total	Surface	Total
Mod 1	Positive Control	NA	10±1.7	470±49	1,100±170	1,600±130	NA	NA
	Bleach	NA	<1.1	47±16	1,200±14	1,200±30	>89 %	24±6.3%
	D7	NA	<1.1	82±42	1,100±140	1,200±120	>89 %	25±9.2%
Mod 2 Eval	Positive Control	4.7±0.56	NA	170±36	1,400±110	1,600±120	NA	NA
	Chem A (no decontaminant)	12±2.3	NA	210±44	1,400±12	1,700±58	NA	NA
	Chem B (no decontaminant)	11±1.1	NA	220±105	1,400±150	1,600±110	NA	NA
Mod 2 Chem A	Positive Control	NA	11±0.083	620±106	1,100±76	1,700±67	NA	NA
	Positive with Chem A	16±3.5	4.5±2.1	310±120	1,200±190	1,500±72	NA	NA
	Bleach with Chem A	14±7.0	<1.1	74±42	1,000±70	1,100±83	>90 %	35±5.6%
Mod 2 Chem B	Positive Control	NA	12±3.8	490±80	1,100±44	1,600±40	NA	NA
	Positive w/ Chem B	15±2.9	5.2±1.3	380±95	1,100±120	1,500±66	NA	NA
	Bleach w/ Chem B	16±1.7	<1.1	88±31	1,000±55	1,100±86	>91 %	32±5.5%
Mod 3 Chem A	Positive Control	NA	8.6±3.8	420±100	1,100±83	1,500±18	NA	NA
	Positive w/ Chem A	16±4.0	5.8±1.3	370±96	1,100±160	1,500±200	NA	NA
	Dahlgren w/ Chem A	13±1.9	<1.1	130±70	1,100±94	1,200±57	>87 %	21±3.8%

NA = not applicable

For Modification 1 sealant testing, the average mass recovered for the D7 wipe samples was greater than the average wipe mass recovered for positive controls; therefore, the surface decontamination efficacy was reported as 0%. Also, the average total mass recovered for the bleach and D7 samples was greater than the average mass recovered for the positive controls, with the Total decontamination efficacy reported as 0%. Modifications 2 and 3 resulted in slight improvements for total HD decontamination efficacy compared to baseline, ranging from 6 to 14% efficacy.

See Appendix D for detailed statistical interpretation of the HD Modification 1, 2, and 3 results and discussion of whether decontaminants had a significant effect on HD levels. The statistical HD evaluations for baseline and modification testing resulted in the following:

- Bleach Modification 1 paint coupon wipe samples resulted in lower HD values (lower HD recovery) compared to Modification 2 with Chem A and Modification 2 with Chem B. Note that bleach Modification 1 was not different from Baseline for HD paint coupon wipes.
- Bleach Modification 1 sealant coupon wipe samples resulted in lower HD values compared to Modification 2 with Chem A and Modification 2 with Chem B. Note that bleach Mod 1 was not different from Baseline for HD sealant coupon wipes.
- Mod 1 with D7 typically did not result in improved HD decontamination compared to Baseline for any material/sample type.
- Most Modification 1 comparisons of bleach to D7 did not have significantly different HD recoveries across all material and sample types.
- Most Modification 2 comparisons were not significantly different. Where differences did exist, there was no clear pattern as to whether Chem A or Chem B provided improved decontamination results
- Most Chemical A comparisons were not significantly different. Where differences did exist, there was no clear pattern as to whether bleach or Dahlgren Decon provided improved decontamination

Table 50. Average HD Mass Recovered for Sealant Films – Modification Testing

Test	Sample Type	Average Mass Recovered ± SD (µg)					Decontamination Efficacy	
		Rinse	Wipe	Coupon	SPE	Total	Surface	Total
Mod 1	Positive Control	NA	<1.1	250±43	1,100±49	1,400±91	NA	NA
	Bleach	NA	<1.1	130±16	1,300±170	1,400±170	ND	0%
	D7	NA	3.8±0.42	180±29	1,200±120	1,400±96	0%	0%
Mod 2 Eval	Positive Control	<3.0	NA	220±15	1,300±100	1,500±110	NA	NA
	Chem A (no decontaminant)	8.7±1.1	NA	160±36	1,400±64	1,500±43	NA	NA
	Chem B (no decontaminant)	9.9±1.5	NA	270±36	1,200±270	1,500±240	NA	NA
Mod 2 Chem A	Positive Control	NA	<1.1	210±27	1,100±57	1,300±81	NA	NA
	Positive with/ Chem A	9.4±0.97	<1.1	210±39	1,100±47	1,400±9.7	NA	NA
	Bleach with Chem A	8.0±0.23	<1.1	68±42	1,100±83	1,200±113	ND	6.7±11%
Mod 2 Chem B	Positive Control	NA	<1.1	250±52	1,200±10	1,400±58	NA	NA
	Positive with Chem B	13±1.4	<1.1	200±38	1,000±45	1,300±42	NA	NA
	Bleach with Chem B	11±0.39	<1.1	130±12	1,100±59	1,200±51	ND	14±5.0%
Mod 3 Chem A	Positive Control	NA	<1.1	276±37	1,100±43	1,400±11	NA	NA
	Positive with Chem A	8.2±1.9	<1.1	200±49	1,100±91	1,300±42	NA	NA
	Dahlgren with Chem A	9.4±0.78	<1.1	160±28	1,100±120	1,300±110	ND	6.3±7.5%
NA = not applicable ND = not determined								

Figure 28 through Figure 32 summarize the Modification 1, 2, and 3 paint and sealant coupon HD average total mass recoveries including error bars equal to one standard deviation for each of the modification tests.

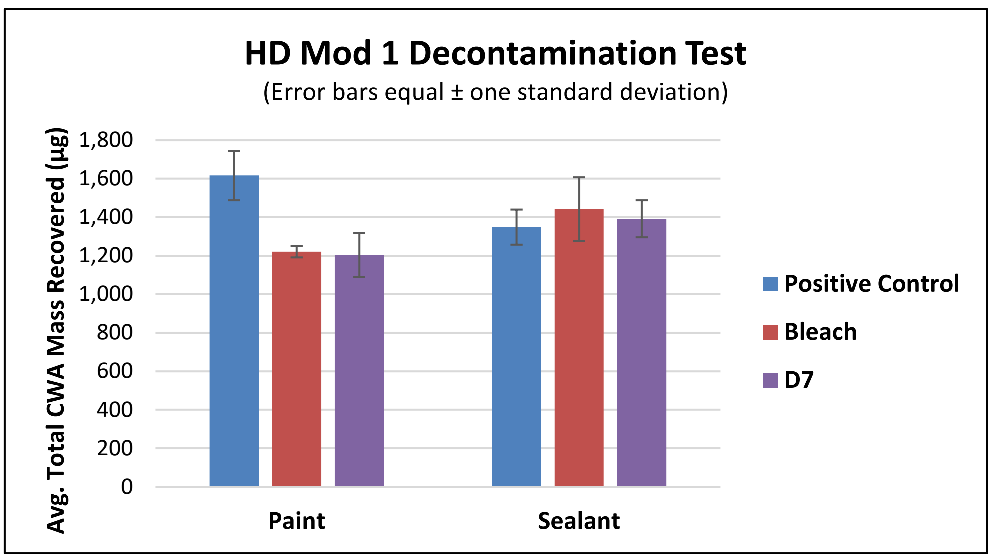


Figure 28. HD Mod 1 Decontaminant Test, Average Total Mass Recoveries

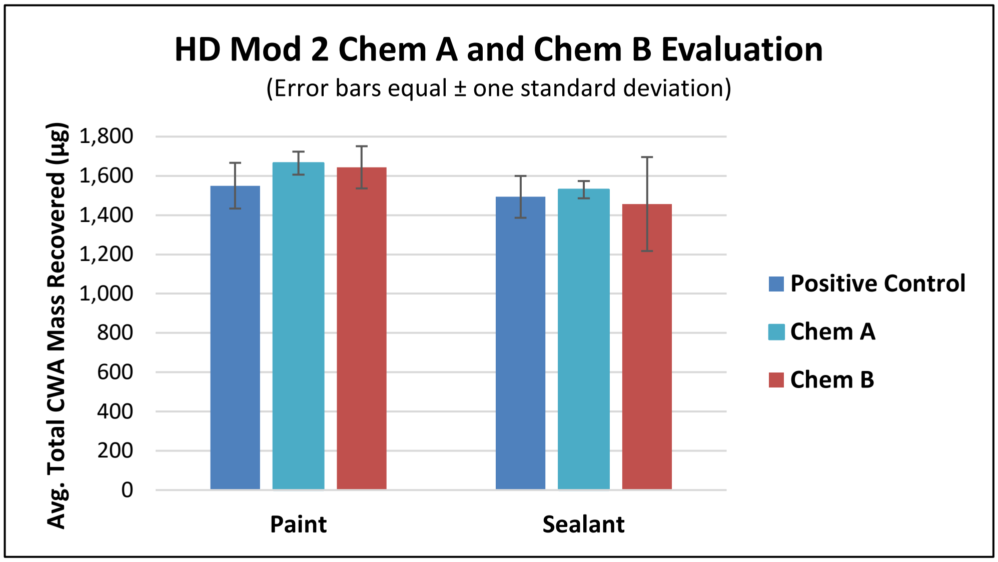


Figure 29. HD Mod 2 Chem A and Chem B Evaluation, Average Total Mass Recoveries

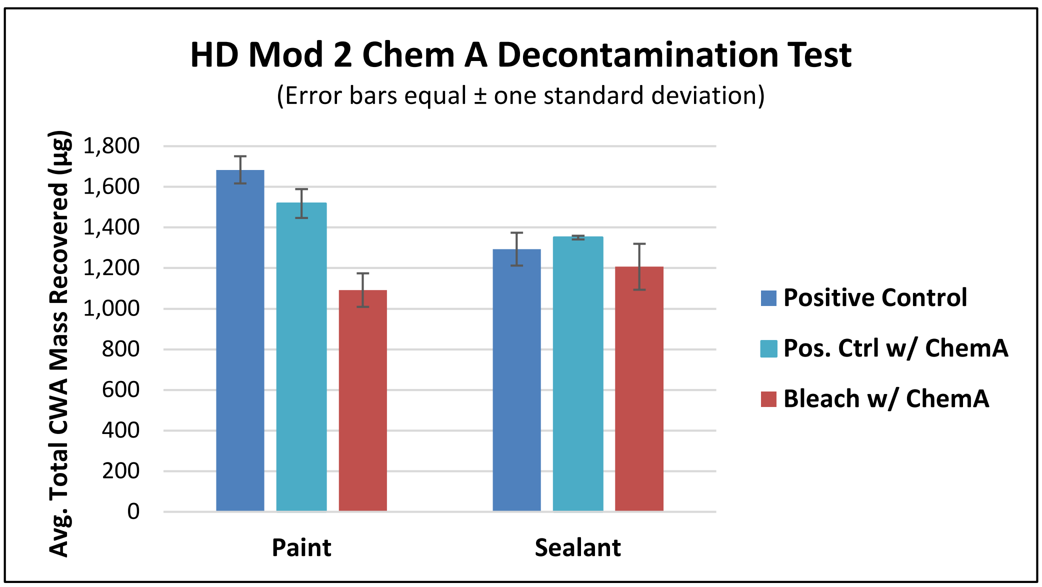


Figure 30. HD Mod 2 Chem A Decontaminant Test, Average Total Mass Recoveries

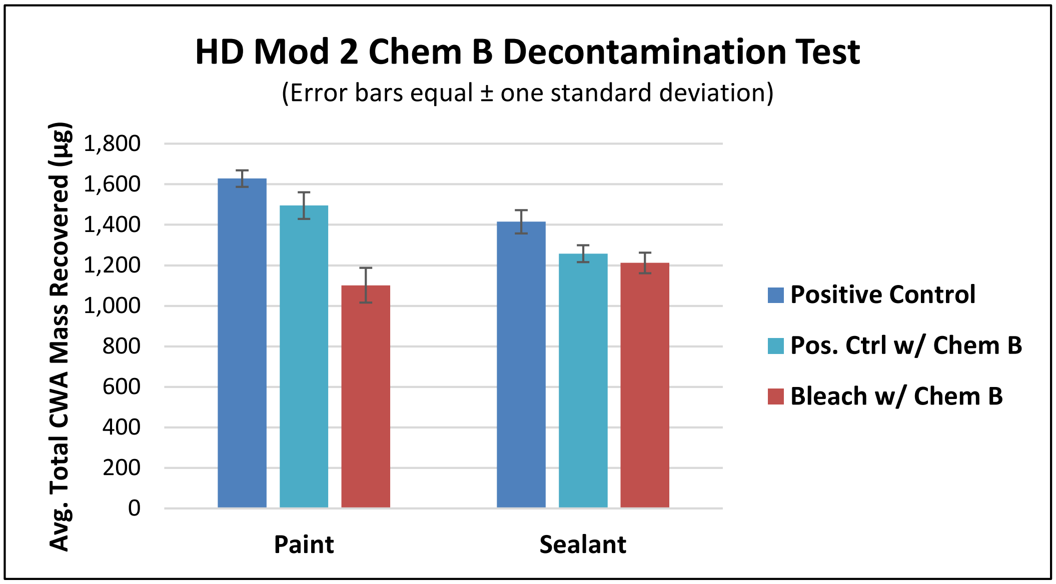


Figure 31. HD Mod 2 Chem B Decontaminant Test, Average Total Mass Recoveries

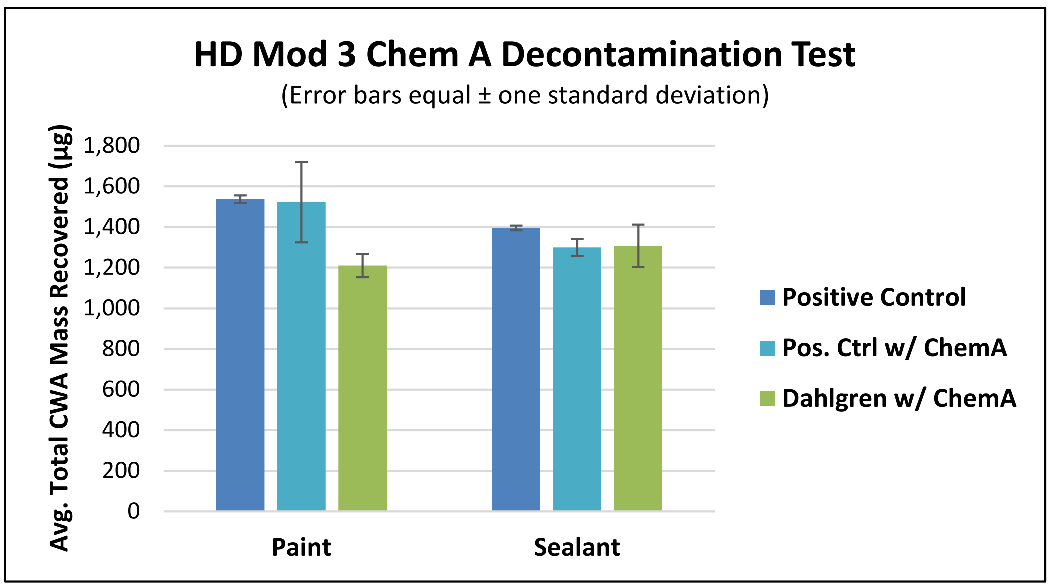


Figure 32. HD Mod 3 Chem A Decontaminant Test, Average Total Mass Recoveries

The average VX mass recoveries for each sample component for the positive controls and decontaminants, as well as total mass (combined recoveries of the rinse, wipe, coupon, and SPE) are provided in Table 51 for paint film coupons and Table 52 for sealant film coupons. The efficacy calculations for Modification 2 and Modification 3 are calculated against the true Positive Control (not the Positive with Chem A or Positive with Chem B). For Modification 3, all paint replicate wipe results were below the quantitation limit. For calculation purposes, the quantitation limit (0.1 µg) was used, and surface decontamination efficacy results were expressed with the > symbol. Also, for paint Modification 3, the average total mass recovered for the Dahlgren Decon samples was greater than the average mass recovered for the positive controls, with the total decontamination efficacy reported as 0%. Doubling bleach dwell time on paint for Modification 1 resulted in approximately doubled VX total decontamination efficacy compared to baseline testing (39% vs 16%). Modification 2 also provided improved total efficacy compared to the baseline for Dahlgren Decon.

Table 51. Average VX Mass Recovered for Paint Films – Modification Testing

Test	Sample Type	Average Mass Recovered ± SD (µg)					Decontamination Efficacy	
		Rinse	Wipe	Coupon	SPE	Total	Surface	Total
Mod 1	Positive Control	NA	88±14	260±40	280±38	620±15	NA	NA
	Bleach	NA	15±6.9	160±36	210±62	380±40	83±8.2%	39±6.6%
	Dahlgren	NA	18±8.6	200±39	270±53	490±22	79±10%	21±4.1%
Mod 2 Eval	Positive Control	260±49	NA	220±20	510±87	990±67	NA	NA
	Chem A (no decontaminant)	280±74	NA	240±56	430±50	960±37	NA	NA
	Chem B (no decontaminant)	390±60	NA	220±24	320±136	930±80	NA	NA
Mod 2 Chem A	Positive Control	NA	180±83	370±95	390±180	940±86	NA	NA
	Positive with Chem A	330±39	29±16	270±38	350±58	980±7.5	NA	NA
	Bleach with Chem A	350±29	0.52±0.59	120±97	250±10	730±120	99.7±0.35%	23±15%
Mod 2 Chem B	Positive Control	NA	110±18	200±18	230±29	540±6.5	NA	NA
	Positive with Chem B	140±9.9	7.5±0.72	150±42	170±98	460±47	NA	NA
	Bleach with Chem B	180±31	0.082±0.073	84±8.0	209±31	470±40	99.9±0.068 %	13±7.6%
Mod 3 Chem A	Positive Control	NA	100±5.3	150±18	220±26	460±23	NA	NA
	Positive with Chem A	260±35	5.8±1.6	110±10	200±64	580±33	NA	NA
	Dahlgren with Chem A	250±3.5	<0.10	80±43	230±38	560±8.3	>99.9	0%

NA = not applicable

For sealant Modification 3, the average total mass recovered for the Dahlgren Decon samples was greater than the average mass recovered for the positive controls, with the total decontamination efficacy reported as 0%. Modification 1 resulted in similar total VX decontamination efficacy for bleach but approximately doubled efficacy for Dahlgren Decon compared to baseline (26% vs 12%). Modification 2 did not provide apparent improvement in efficacy for Dahlgren Decon compared to the baseline. See Appendix D for detailed statistical interpretation of the VX Modification 1, 2, and 3 results and discussion of whether decontaminants had a significant effect on VX levels. Statistical VX evaluations of baseline and modification testing resulted in the following:

- Bleach Modification 1 paint coupon wipe resulted in lower VX values than Baseline, Modification 2 with Chem A, and Modification 2 with Chem B.
- Bleach Modification 1 sealant coupon wipe samples resulted in lower VX values compared to Modification 2 with Chem A and Modification 2 with Chem B. Note that bleach Modification 1 was not statistically different from Baseline for VX sealant wipes.
- Modification 3 with Dahlgren typically did not result in improved VX decontamination compared to Baseline for any material/sample type.

- Most Modification 1 comparisons of bleach to Dahlgren Decon did not have statistically significant different VX recoveries across all material and sample types.
- Most Modification 2 comparisons were not significantly different. Where differences did exist, there was no clear pattern as to whether Chem A or Chem B provided improved decontamination results
- Most Chemical A comparisons were not significantly different. Where differences did exist, there was no clear pattern as to whether bleach or Dahlgren Decon provided improved decontamination

Table 52. Average VX Mass Recovered for Sealant Film Coupons – Modification Testing

Test	Sample Type	Average Mass Recovered ± SD (µg)					Decontamination Efficacy	
		Rinse	Wipe	Coupon	SPE	Total	Surface	Total
Mod 1	Positive Control	NA	35±5.3	890±27	1.7±2.3	930±24	NA	NA
	Bleach	NA	0.0049± 0.0023	510±89	20[1]	530±98	99.99 ±0.0068%	43±11%
	Dahlgren	NA	0.37±0.28	680±23	5.6±7.3	690±29	99.0 ±0.79%	26±3.6%
Mod 2 Eval	Positive Control	470±21	NA	370±71	140±145	980±89	NA	NA
	Chem A (no decontaminant)	410±110	NA	410±35	3.0±3.9	820±94	NA	NA
	Chem B (no decontaminant)	390±18	NA	500±30	0.56±0.41	900±41	NA	NA
Mod 2 Chem A	Positive Control	NA	29±8.6	550±100	110±190	690±96	NA	NA
	Positive with Chem A	400±50	11±7.7	200±18	21±30	630±29	NA	NA
	Bleach with Chem A	360±120	0.0070± 0.0038	250±32	2.2±3.6	610±108	99.98± 0.015%	11±20%
Mod 2 Chem B	Positive Control	NA	32±3.9	720±47	0.58±0.75	750±51	NA	NA
	Positive with Chem B	300±70	17±0.52	340±39	1.0±2.0	660±100	NA	NA
	Bleach with Chem B	290±34	0.091± 0.093	330±6.0	0.041± 0.018	620±28	99.7± 0.29%	17±6.8%
Mod 3 Chem A	Positive Control	NA	38±3.8	650±41	0.80±0.65	680±44	NA	NA
	Positive w/ Chem A	500±47	4.3±2.1	180±42	100±86	780±4.3	NA	NA
	Dahlgren w/ Chem A	520±13	0.26[1]	250±15	3.3±2.5	780±8.7	99.3	0%
NA = not applicable								

[1]SD could not be calculated; only one replicate mass above the quantitation limit

Figure 33 through Figure 37 summarizes the Modification 1, 2, and 3 paint and sealant film coupon VX average total mass recoveries, including error bars equal to one standard deviation for each of the modification tests.

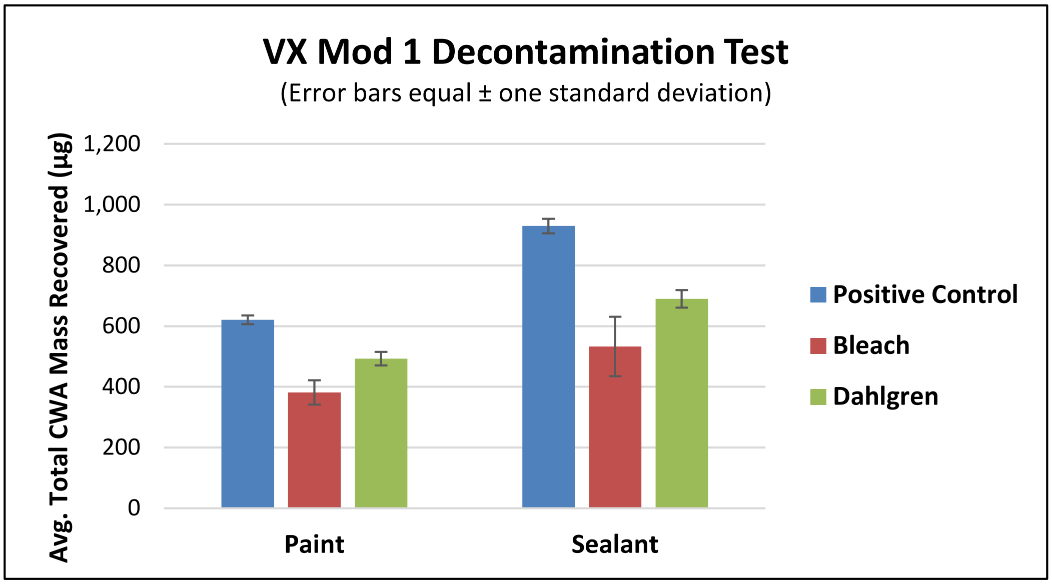


Figure 33. VX Mod 1 Decontaminant Test, Average Total Mass Recoveries

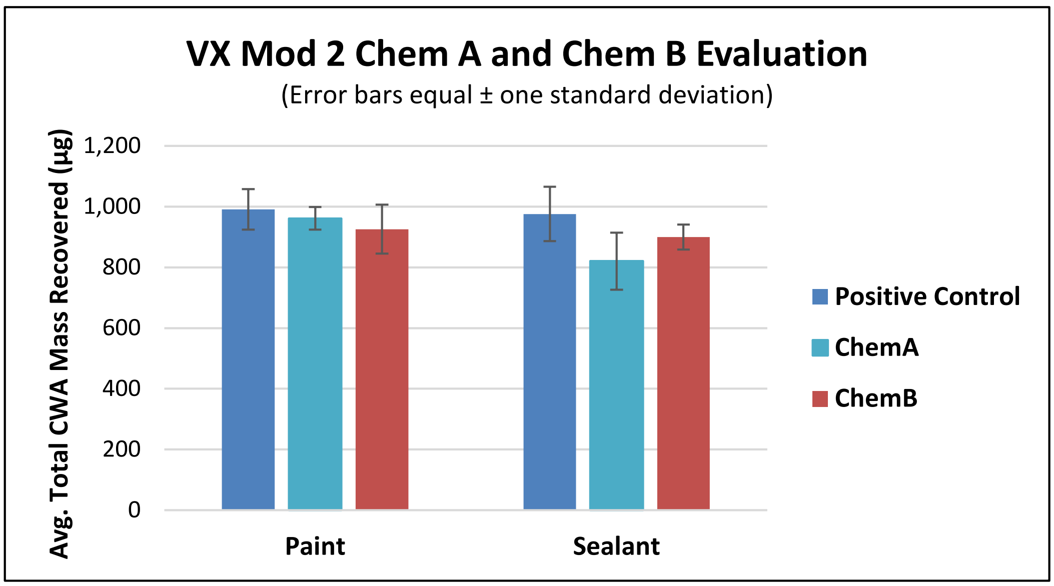


Figure 34. VX Mod 2 Chem A and Chem B Evaluation, Average Total Mass Recoveries

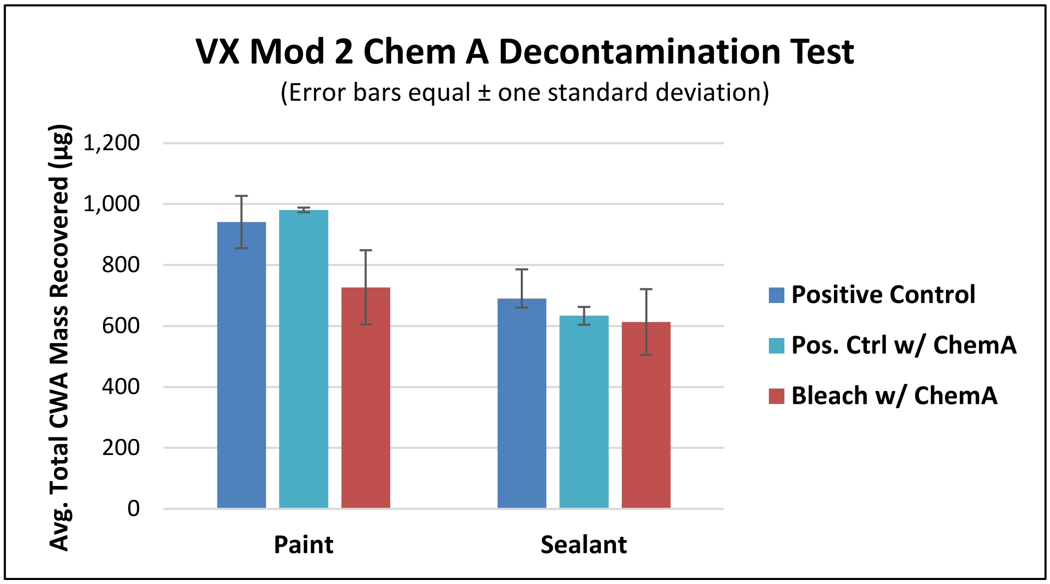


Figure 35. VX Mod 2 Chem A Decontaminant Test, Average Total Mass Recoveries

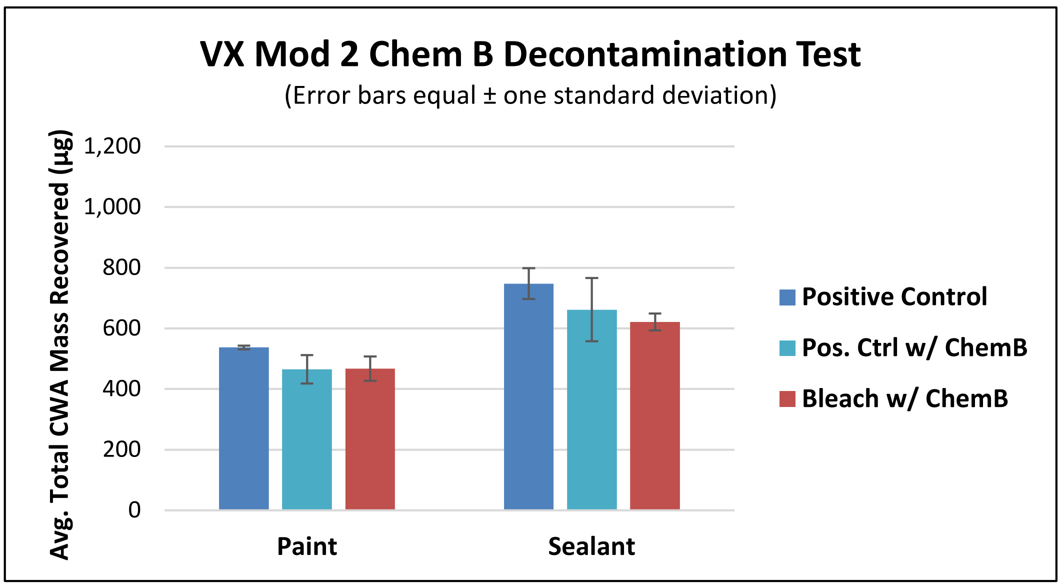


Figure 36. VX Mod 2 Chem B Decontaminant Test, Average Total Mass Recoveries

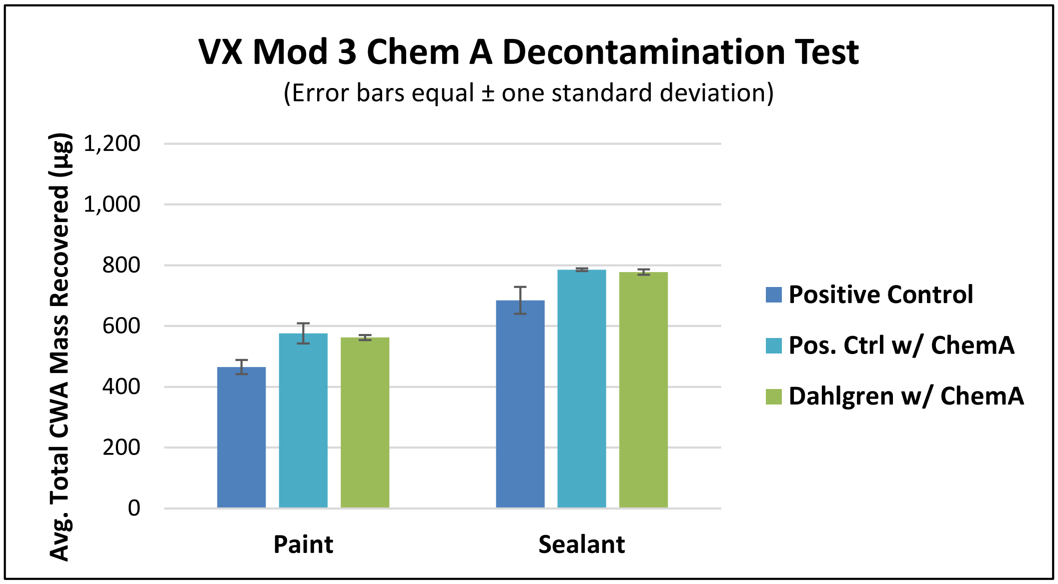


Figure 37. VX Mod 3 Chem A Decontaminant Test, Average Total Mass Recoveries

E.8 Pesticide Modification Decontamination Testing

Pesticide modification testing included a total of two trials, one each for malathion and fipronil. Each trial included two test materials (paint and sealant film coupons), one decontaminant (D7), and two decontaminant modifications as described below:

- Modification 1 – two applications of D7, each with a 60-minute dwell time. A triple water rinse was performed after the first application of D7.
- Modification 2 – one application of D7 with a 120-minute dwell time.

All pesticide spike control results (shown in Table 53) met the acceptance criteria described in Section D.6. All malathion and fipronil laboratory blank results and a majority of the procedural blank results met the acceptance criteria described in Section D.6. One procedural blank wipe sample had a low-level result for fipronil; no impact to the data was observed. Internal standard response for all fipronil and malathion results met the acceptance criteria described in Sections C.7 and C.8. Most of the wipe and coupon procedural blanks required a 100-fold dilution (rather than the typical 10-fold) to ensure internal standard response was acceptable.

Table 53. Pesticide Average Spike Control Results – Modification Testing

Pesticide	Test	Average Mass Recovered (µg)	Percent Recovery (vs Theoretical)	RSD
Malathion	Mod 1/Mod 2	32	85%	12%
Fipronil	Mod 1/Mod 2	13	90%	0.88%

The average malathion mass recoveries for each sample component for the positive controls and decontaminants, as well as total mass (combined recoveries of the rinse, wipe, coupon, and SPE) are provided in Table 54 for paint films and Table 55 for sealant films. For paint Modification 1 and 2, the average total mass recovered for the decontaminated samples was greater than the average mass recovered for the positive controls, with the total decontamination efficacy reported as 0%, which was below Baseline efficacy.

Table 54. Average Malathion Mass Recovered for Paint Films – Modification Testing

Sample Type	Average Mass Recovered ± SD (µg)					Decontamination Efficacy	
	Rinse	Wipe	Coupon	SPE	Total	Surface	Total
Positive Control	NA	<1.1	13±7.7	<1.0	13±7.7	NA	NA
Mod 1	<5.0	<1.1	17±3.1	<1.0	17±3.1	ND	0%
Mod 2	NA	<1.1	14±7.2	<1.0	14±7.2	ND	0%
NA = not applicable ND = not determined							

For Modification 1, all sealant coupon replicate wipe results were below the quantitation limit. For calculation purposes, the quantitation limit (1.1 µg) was used, and surface decontamination efficacy results were expressed with the > symbol. Modification 1 and 2 both resulted in improved total malathion decontamination efficacy compared to the baseline result, which was 0% for D7. In addition to malathion analysis, samples were also analyzed for malaoxon. No samples had any measurable malaoxon above the quantitation limit.

Table 55. Average Malathion Mass Recovered for Sealant Films – Modification Testing

Sample Type	Average Mass Recovered ± SD (µg)					Decontamination Efficacy	
	Rinse	Wipe	Coupon	SPE	Total	Surface	Total
Positive Control	NA	5.2±3.4	7.6±6.8	<1.0	13±4.5	NA	NA
Mod 1	<5.0	<1.1	7.3±0.99	<1.0	7.3±0.99	>79 %	43±22%
Mod 2	NA	1.3[1]	9.7±1.8	<1.0	11±2.4	74 %	14±36%
NA = not applicable							

[1]SD could not be calculated; only one replicate mass above the quantitation limit

See Appendix E for statistical interpretation of the malathion Modification 1 and 2 results and discussion of whether decontaminants had a significant effect on malathion levels. Statistical malathion evaluations of baseline and modification testing resulted in the following:

- For Baseline comparisons, 10x diluted bleach resulted in lower malathion values only for sealant coupons.
- For D7, Baseline sealant coupon samples resulted in lower malathion values compared to Modification 1 or Modification 2. For malathion, there were no statistical differences for Baseline, Modification 1, or Modification 2 paint coupon sample recoveries.

Figure 38 summarizes the Modification 1 and 2 paint and sealant malathion average total mass recoveries including error bars equal to one standard deviation for the modification test.

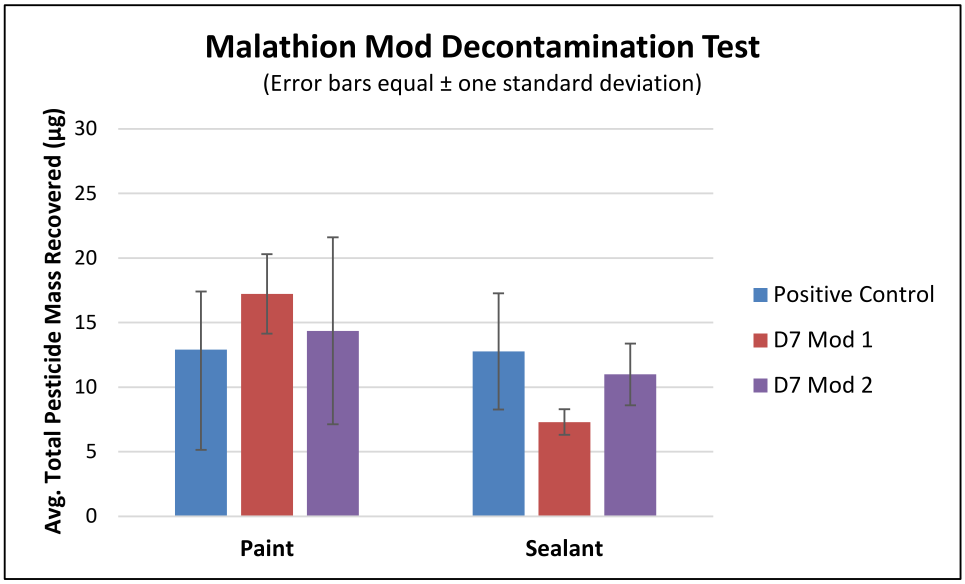


Figure 38. Malathion Modification Decontaminant Test, Average Total Mass Recoveries

The average fipronil mass recoveries for each sample component for the positive controls and decontaminants, as well as total mass (combined recoveries of the rinse, wipe, coupon, and SPE) are provided in Table 56 for paint film coupons and Table 57 for sealant film coupons. Modification 1 for paint coupons resulted in improved total fipronil decontamination efficacy compared to Baseline (98% vs. 36%). All other modification results for fipronil were similar to Baseline results.

Table 56. Average Fipronil Mass Recovered for Paint Film Coupons – Modification Testing

Sample Type	Average Mass Recovered ± SD (µg)					Decontamination Efficacy	
	Rinse	Wipe	Coupon	SPE	Total	Surface	Total
Positive Control	NA	7.4±1.1	3.6±0.81	<0.001	11±0.42	NA	NA
Mod 1	<0.01	0.079±0.10	0.15±0.019	<0.001	0.23±0.033	99±1.3%	98±0.31%
Mod 2	NA	0.12±0.076	0.79±0.14	<0.001	0.91±0.22	98±1.1%	92±2.0%
NA = not applicable							

Table 57. Average Fipronil Mass Recovered for Sealant Film Coupons – Modification Testing

Sample Type	Average Mass Recovered ± SD (µg)					Decontamination Efficacy	
	Rinse	Wipe	Coupon	SPE	Total	Surface	Total
Positive Control	NA	10±0.35	1.4±0.52	<0.001	11±0.22	NA	NA
Mod 1	0.20±0.15	0.17±0.072	0.015±0.0094	<0.01	0.38±0.21	98±0.72%	97±1.9%
Mod 2	NA	1.4±0.50	0.52±0.11	<0.001	1.9±0.59	86±5.0%	83±5.2%
NA = not applicable							

See Appendix E for statistical interpretation of the fipronil Modification 1 and 2 results and discussion of whether decontaminants had a significant effect on fipronil levels. Statistical fipronil evaluations for Baseline and modification testing resulted in the following:

- For D7 comparisons, Modification 1 paint and sealant coupon samples resulted in statistically significant lower fipronil values (lower fipronil recovery) compared to baseline and Modification 2.
- For Baseline comparisons, D7 resulted in statistically significant lower fipronil values for both paint wipes and paint coupons compared to 10x diluted bleach. D7 also resulted in statistically significant lower fipronil values for sealant coupons compared to 10x diluted bleach.

Figure 39 summarizes the Modification 1 and 2 paint and sealant fipronil average total mass recoveries including error bars equal to one standard deviation for the modification test.

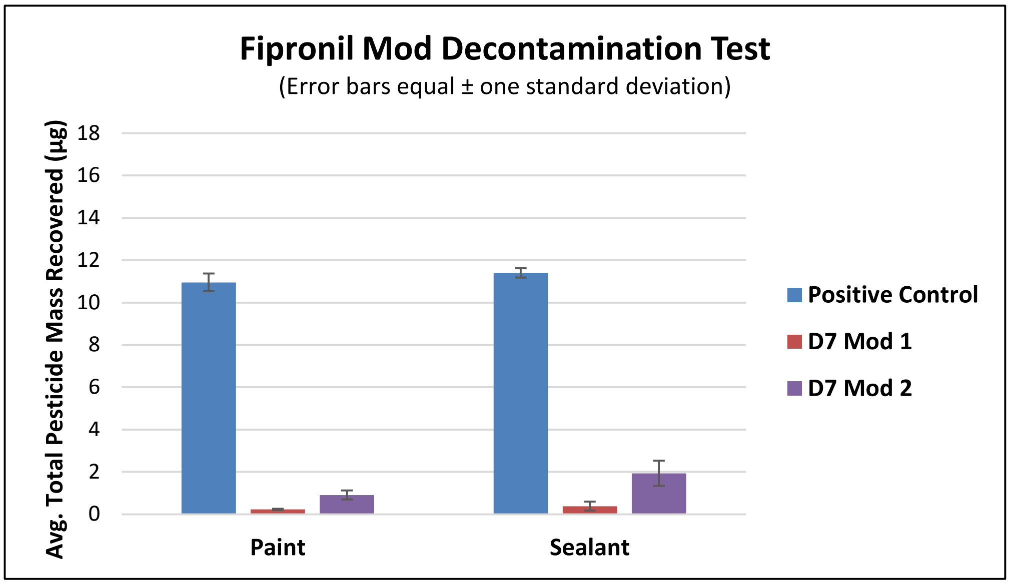


Figure 39. Fipronil Modification Decontaminant Test, Average Total Mass Recoveries

F. QUALITY ASSURANCE/QUALITY CONTROL

Data quality indicators, quality control elements, and quality assurance audits described in the sections below provide the requirements for determining the adequacy of data generated during this project. Methods were considered acceptable and the data valid if the data quality indicators for the test measurements were met, and the performance evaluation, TSA, and data quality audits demonstrated acceptable results.

F.1 Data Quality Indicators

Data quality indicators and results are provided in Table 58. Most of the data quality indicator results were acceptable per the QAPP for *Decontamination Options for Chemical Warfare Agents Permeated into Surface Layers* (May 18, 2020), as amended, including checks of the measurement methods for time, temperature, RH, volume, mass, pH, and CWA and pesticide levels in blank samples and spike controls.

Table 58. Data Quality Indicators and Results

Parameter	Measurement Method	Data Quality Indicator	Results
Time (seconds)	Timer/data logger	Compare to time provided at NIST.time.gov once before testing; agree ±2 seconds/hour.	No difference was observed between the timers and NIST.time.gov after one hour.
Temperature (°C)	National Institute of Standards and Testing (NIST*)-traceable thermometer	Compare against calibrated thermometer once before testing; agree ±1 °C through 60 min.	The HOBO UX100 datalogger remained within 0.12 °C of the calibrated reference through 60 min.
Relative Humidity (%)	NIST-traceable hygrometer	Compare against calibrated hygrometer once before testing; agree ±10% through 60 min.	The HOBO UX100 datalogger remained within 0.50% of the calibrated reference through 60 min.
Volume (µL)	Syringe with repeating dispenser or calibrated pipette (CWA/pesticide application)	Syringe and pipette were checked for accuracy and repeatability once before use by determining the mass of water delivered onto a calibrated balance. The syringe and pipette were acceptable if the average mass of five replicate droplets was ±10% of expected (percent error).	The syringe used for CWA application and pipette used for pesticide application were checked. Percent error was calculated using the following equation: % Error = $\frac{\lvert Expected\ Weight - Mean\ Weight \rvert}{Expected\ Weight}\ x\ 100$ Percent error results for each are provided below: • 100 µL syringe: 5.8% (2-µL droplet) • 3-25 µL pipette: 2.6% (13-µL droplet)
Volume (µL)	Calibrated pipettes (LC-MS/MS sample dilution)	Pipettes were checked for accuracy and repeatability before use by the manufacturer. The pipette was acceptable if the percent error was ±10% of expected.	Pipettes used for LC-MS/MS sample dilution were checked. Systematic error for each is provided below: • MR-10 at 5 µL: 0.39% • MR-50 at 35 µL: 1.3% • MR-250 at 100 µL: 0.42% • MR-1000 at 500 µL: -0.13%
Weight (g)	Oil mister/sprayer (decontaminant delivery)	Checks of each sprayer were performed before each test by weighing the amount of delivered decontaminant. Spray application was acceptable if the RSD of three replicates was ≤ 15%. The balance was within calibration and checked daily with weights bracketing the mass of sprayed decontaminant.	Three replicate spray applications of each decontaminant were delivered to Teflon disks before each trial. All RSDs met the stated criteria. All balance calibration checks met acceptance criteria of ± 2.0% of nominal mass

Parameter	Measurement Method	Data Quality Indicator	Results
pH	Calibrated pH meter	Meter was checked for accuracy prior to each use using buffer solutions at: • pH 4 (910104, Fisher Scientific) • pH 7 (1552-16, Fisher Scientific) • pH 10 (1602-16, Fisher Scientific) • pH 12.5 (1618-16, Fisher Scientific) Check value must be within ± 0.1 pH units of the buffer value.	pH meter was checked before each use using the specified buffer solutions and was within tolerance during all checks. All buffer solutions were within the expiration date.
CWA/pesticide in LB** sample extracts	Extraction, LC/MS/MS or GC/MS	LBs should have had less than 50% of the lowest detected amount on the test coupon or 1% of the amount on the positive controls, whichever was lower.	No CWA/pesticide outside the stated criteria was measured in any of the LBs throughout testing.
CWA/pesticide in PB*** sample extracts	Extraction, LC/MS/MS or GC/MS	PBs should have had less than 50% of the lowest detected amount on the test coupon or 1% of the amount on the positive controls, whichever was lower.	Most of the PBs met the stated criteria, several replicates had low-level CWA/pesticide detected (see also discussion in Section E.5 and F.6): • VX Mod 2 Chem A – 0.0024 µg detected in sealant wipe sample • VX Mod 2 Chem B – 0.010 µg detected in sealant wipe sample • Fipronil Mod – 0.014 µg detected in paint wipe sample
CWA/pesticide in SCs	LC/MS/MS or GC/MS	The mean of the SCs included with each test should have been within 80% to 120% of the target amount and had an RSD of < 30% between replicates.	Mean and RSD of the following SC sets were outside tolerance: • HD FandT – 72% avg SC recovery, 60% RSD (see also discussion in Section E.4) • Malathion Baseline – 51% avg SC recovery, 38% RSD (see also discussion in Section E.6)

*: NIST: National Institute of Standards and Testing
**: LB: Laboratory blank
***: PB: Procedural blank

F.2 Quality Control Elements

Data accuracy was ensured by the calibration of all instruments. Instrumentation used during this project was maintained and operated according to the quality requirements and documentation described in the approved QAPP and associated standard operating procedures (SOPs). Except for the GC/MS and LC-MS/MS, all instruments utilized during the project were calibrated as stipulated by the manufacturer or, at a minimum, annually. The GC/MS and LC-MS/MS were calibrated according to the approved QAPP and associated SOPs. Table 59 provides calibration frequency for instruments that were used during this project.

Table 59. Instrument Calibration Frequency

Instrument	Frequency
Timer	Prior to testing, performed by the manufacturer. After the manufacturer-provided calibration expired, use of the expired unit was discontinued and the unit was discarded. A new manufacturer-calibrated unit was obtained for use.
Calibrated UX100 HOBO Thermometer/Hygrometer	Prior to testing, performed by the manufacturer. After the manufacturer-provided calibration expired, use of the expired unit was discontinued and the unit was discarded. A new manufacturer-calibrated unit was obtained for use.
Calibrated Pipettes	Prior to testing by the vendor and annually thereafter. Calibration/accuracy was also verified as described in Table 58.
Balances	Prior to testing by the vendor and annually thereafter. Calibration/accuracy was also verified as described in Table 58.
pH Meter	Calibration/accuracy was verified prior to each use as described in Table 58.
GC/MS	Calibrated prior to analysis of each set of test samples (calibration curve) and a calibration verification standard was analyzed after every five samples and at the end of a set of samples (see detailed discussion in Section C.7).
LC-MS/MS	Calibrated prior to analysis of each set of test samples (calibration curve) and a calibration verification standard was analyzed after every ten samples and at the end of a set of samples (see detailed discussion in Section C.8).

At all times during the project, protocols required by Battelle's HMRC were followed in the movement and use of CWA within the test facility. Chain of Custody (CoC) forms were used to ensure that test samples generated during the work were traceable throughout all phases of testing. Test measurements and information were recorded on Test Parameter Control Sheets (TPCSs) or in a laboratory record book (LRB). Monitoring of test conditions, parameters, and times was performed by technical staff familiar with the QAPP and testing and was documented on the TPCS. The results of each test set were provided to the client electronically in the form of Microsoft Excel™ files. Each Excel file included the CoC, GC/MS or LC-MS/MS analytical results, final results for each sample showing all calculations, and a summary of the results. Each sample was traceable from the CoC, to the analytical results, to the final results.

F.3 Quality Assurance Audits

Performance evaluation audits were essentially conducted continuously and addressed those reference measurements that factored into the data used in quantitative analysis during the evaluation, including volume, mass, and time measurements and GC/MS or LC-MS/MS calibration and performance; see results provided in Table 58. The volume of CWA and pesticides dispensed correlated directly to the mass of CWA and pesticides on the coupons. Daily calibration of the GC/MS and LC-MS/MS, CCVs, and internal standard recovery provided confidence that the analysis system was providing accurate data.

While temperature and RH were measured and recorded for all testing using a calibrated device, these parameters were not controlled; therefore, no performance evaluation audit could be performed. See Appendix B for a summary table of measured temperature and RH at the start of each test.

A Battelle Quality Assurance (QA) Officer performed a TSA at the HMRC facility in West Jefferson, Ohio, for this testing on January 18, 2021, and on April 12, 2021. The purpose of the TSA was to ensure that testing was performed in accordance with the QAPP. The QA Officer reviewed the investigation methods, compared test procedures to those specified in the QAPP, and reviewed data acquisition and handling procedures. The Battelle QA Officer did not identify any findings that required corrective action.

A data quality audit provided validation of the data, including verification of the completeness of the data, compliance with the acceptance criteria in the QAPP, recalculation checks, and tracing of the data from instrument outputs through the final report. One hundred percent (100%) of the data was reviewed prior to use in calculations or any data manipulation, and review was completed before the data were provided to QA for the data quality audit.

The QA Officer, operating independently of the laboratory testing effort, audited at least 10% of the data generated during testing. Data were traced from initial acquisition through reduction and to final reporting. All calculations were checked.

Through the data quality audit, the TSA, and review of reports, the QA Officer ensured that data generated during testing were valid, meeting the requirements of the QAPP.

F.4 QAPP

Two amendments to the QAPP were prepared during the project:

- Amendment 1 (dated January 11, 2021) identified which three decontaminants were selected for CWA decontamination testing; identified the two decontaminants that were downselected for use in CWA decontamination modification testing; and included the titration approach for determining the percent peracetic acid concentration in Dahlgren Decon.
- Amendment 2 (dated March 22, 2021) included the use of 10% butoxyethanol in water and Zep Foaming Wall Cleaner for Modification 2 and 3 CWA testing; identified the use of 10x bleach and D7 decontaminants for pesticide testing; established 24-hour and 72-hour pesticide contact time tests for fate and transport testing; noted that all freestanding paint and film coupons used in testing had been 4.5 cm instead of 5 cm and that 4.5 cm coupons would continue to be used; stated that malathion and fipronil would be purchased as commercially available materials for use in testing; and added malaoxon as a target degradation product for malathion testing.

No deviations to the QAPP or QAPP amendments occurred.

G. SUMMARY

The purpose of this project was to evaluate the efficacy of various liquid decontaminants to degrade CWA and pesticide on the surface of coating layers, as well as CWAs and pesticides that permeated past the coatings and into underlying porous materials. Decontaminants were initially tested to determine which would be efficacious in decontamination of CWAs and pesticides from the two freestanding coating layers, paint and sealant. The decontaminants that were initially used for efficacy testing of the CWAs HD and VX included: bleach (nominal hypochlorite concentration of 5% active ingredient), Dahlgren Decon (peracetic acid as active ingredient), and Decon7 (D7) (H_2O_2 active ingredient). The decontaminants that were initially used for efficacy testing of the pesticides (malathion and fipronil) included: 10x diluted bleach (nominal hypochlorite concentration of 0.5%) and D7 (H_2O_2 active ingredient). A 2-µL volume of CWA or a 12-µL volume of aqueous pesticide solution was applied to the surface of replicate paint and sealant film coupons installed in LVAP assemblies and allowed to make contact for 24 hours (HD only) or for 72 hours (VX and both pesticides). Decontaminants were then applied via spray to the surface of the films and allowed to dwell for 60 minutes. Following the decontaminant dwell period, the surfaces of the film coupons were wipe-sampled and the film coupons and SPE disks (underlying porous material) were solvent-extracted. HD and malathion in wipe samples and extracts of film coupons and SPE disks were quantified using gas chromatography/mass spectrometry (GC/MS) analysis. VX and fipronil were quantified in wipe and LVAP component extracts using liquid chromatography-tandem mass spectrometry (LC-MS/MS) analysis.

Based on the initial Baseline decontamination results, the decontaminants were down-selected for additional testing to evaluate efficacy of decontamination modifications. For CWAs, two decontaminants were included in the additional efficacy testing, with three decontamination approach modifications were evaluated, as shown in Table 60. For pesticides, one decontaminant was included in the additional efficacy testing, with two decontamination approach modifications evaluated, as shown in Table 61.

Table 60. Decontaminant Downselection and Modification for CWA

Modification	CWA	Decontaminant	Modification Description
1	HD	Bleach	120-minute decontamination dwell time
		Dahlgren	
	VX	Bleach	
		D7	
2	HD	Bleach	Application of 2-butoxyethanol (Chemical A) or Zep Foaming Wall Cleaner (Chemical B), followed by a water rinse, and 60-minute decontamination dwell time
	VX	Bleach	
3	HD	Dahlgren	Application of Chemical A, followed by a water rinse, and 60-minute decontamination dwell time
	VX	Dahlgren	

Table 61. Decontaminant Downselection and Modification for Pesticides

Modification	Pesticide	Decontaminant	Modification Description
1	Malathion	D7	60-minute decontamination dwell time, followed by a water rinse, and second 60-minute decontamination dwell time
	Fipronil	Bleach	
2	Malathion	D7	120-minute decontamination dwell time
	Fipronil	D7	

The average total (combined efficacy of wipe sampling, film coupon extraction, and SPE disk extraction) decontamination efficacies measured during CWA testing were low (≤ 50% efficacy) across all the Baseline and modification testing for both paint and sealant films. The highest decontamination efficacy measured for HD-contaminated paint films was demonstrated during Modification 2 testing of bleach with Chemical A (average 35% efficacy) and for HD contaminated sealant films, was demonstrated during Modification 2 testing of bleach with Chemical B (average 14% efficacy). The highest decontamination efficacy measured for VX-contaminated paint film coupons was demonstrated during Modification 1 testing with bleach (average 39% efficacy), and for VX-contaminated sealant films was demonstrated during Baseline testing with bleach (average 50% efficacy). Figure 40 and Figure 41summarize the average total percent decontamination efficacy measured for each test condition during Baseline and modification testing for CWAs. While occasional improvements in the total recoveries were noticed for some of the modifications that included the use of Chemical A or B, the general trend appears to be that these chemicals do not help in the extraction of CWAs from the paint or sealant followed by degradation at the surface with the selected decontaminant based on total decontamination efficacy. Degradation of CWAs as present in the SPE disk (representing a porous material under the film) was not observed for any of the tested modifications. All HD paint and sealant wipe recoveries were significantly different compared to the positive controls that occurred for the Baseline and Modification 1 decontamination tests while all the HD paint and sealant coupon differences occurred for Modification 1, Modification 2, and Modification 3. In all cases, the difference was due to the test samples being less than the positive controls. All the VX paint wipe differences occurred for Modification 1 and all of the VX sealant wipe and coupon extraction differences occurred for Baseline and Modification 1. In all cases, the difference was due to the test samples being less than the positive controls. Almost no significant differences were observed between test samples and positive controls for SPE disks extracted for HD or VX.

The average total decontamination efficacy measured during pesticide testing with malathion was low across all the Baseline and modification testing for paint films, with the highest efficacy demonstrated during Baseline testing using 10x diluted bleach (average 6.8% efficacy). The average total decontamination efficacy measured during pesticide testing with malathion were highest during Baseline testing for sealant films using 10x diluted bleach (average 77% efficacy). The average total decontamination efficacy measured during pesticide testing with fipronil was mostly high across all the Baseline and modification testing for both paint and sealant films. The highest decontamination efficacy measured for fipronil-contaminated paint films was demonstrated during Modification 1 testing with D7 (average 98% efficacy), and for fipronil contaminated sealant films was demonstrated during Baseline testing with 10x diluted bleach (average 99% efficacy). Figure 43 and Figure 42 summarize the average total percent decontamination efficacies measured for each test condition during Baseline and modification testing for pesticides.

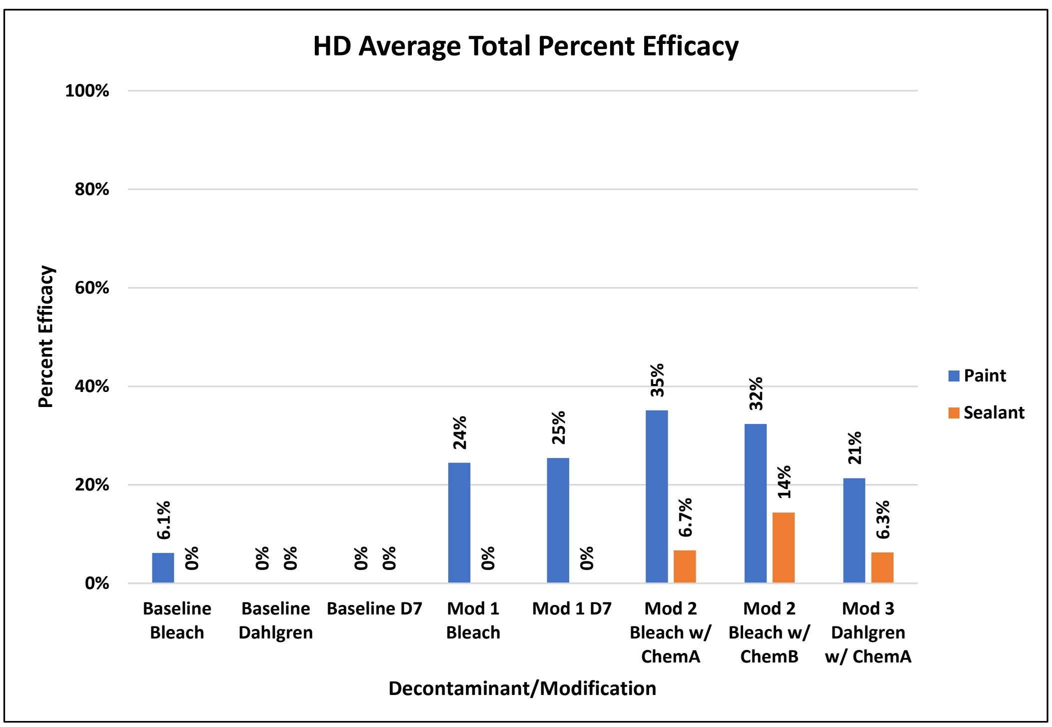


Figure 40. HD Average Total Percent Efficacy

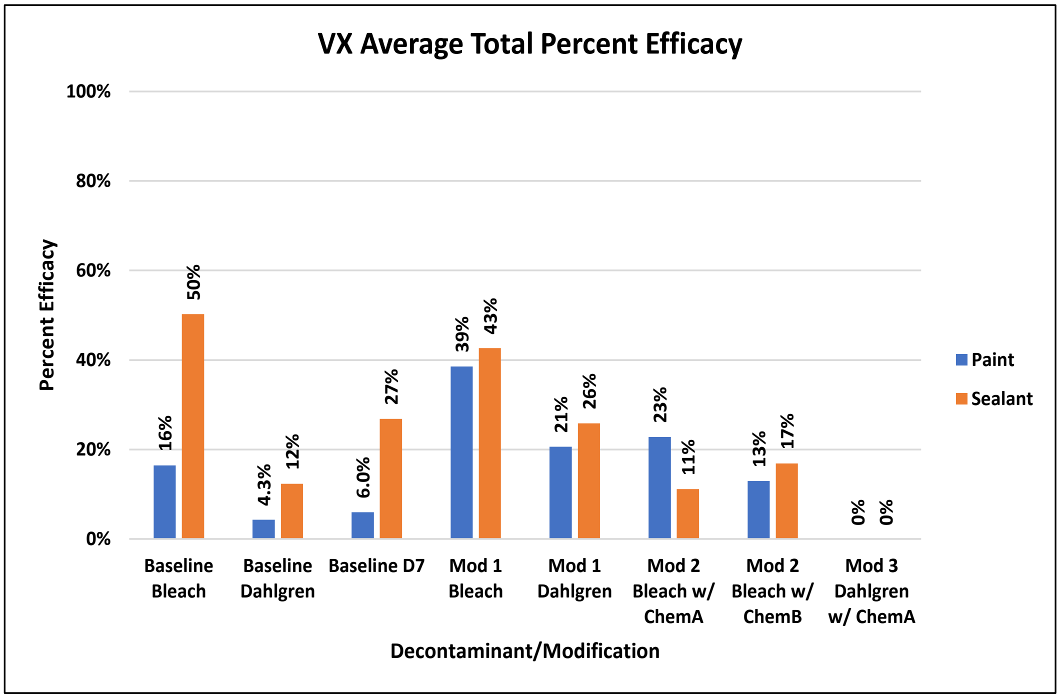


Figure 41. VX Average Total Percent Efficacy

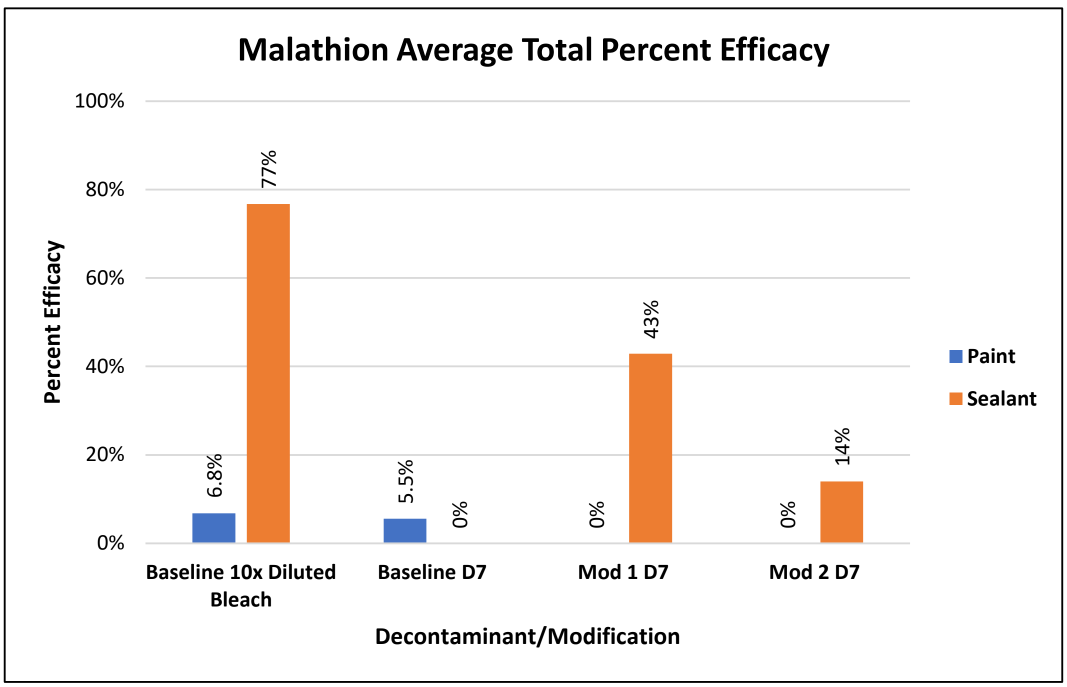


Figure 42. Malathion Average Total Percent Efficacy

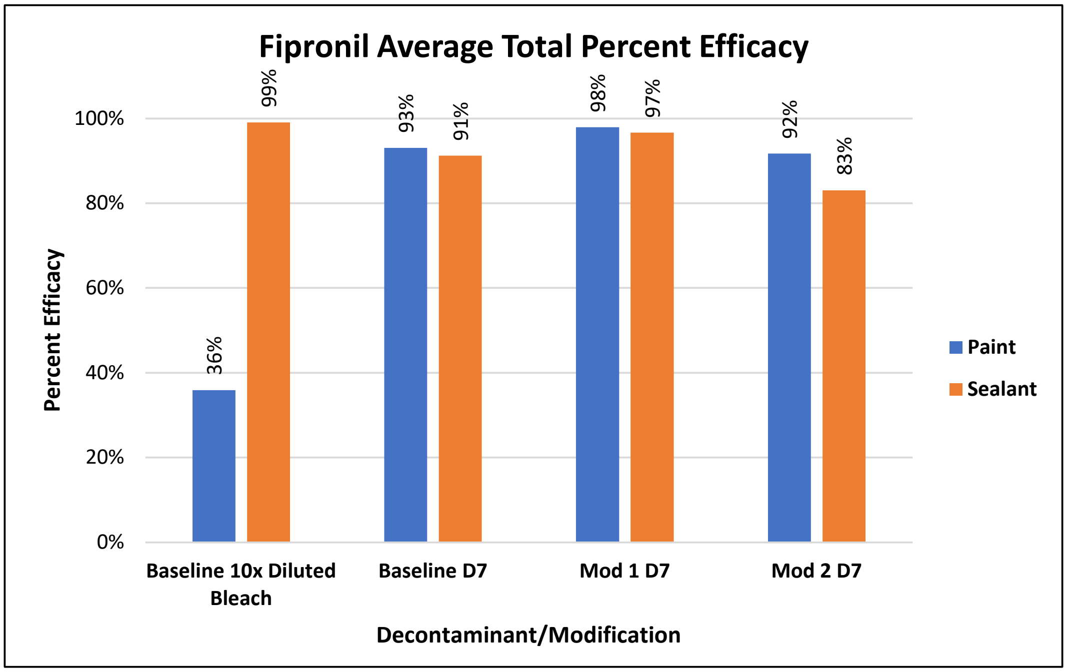


Figure 43. Fipronil Average Total Percent Efficacy

H. REFERENCES

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APPENDIX A

DECONTAMINANT ACTIVITY DATA

Bleach Concentration and pH Results

Trial	Decontaminant Lot #	Concentration (Hypochlorite)	pH
Quench (HD/VX)	20248	5.5%	12.26
Baseline (HD)	20265	5.8%	12.18
Baseline (VX)	20258	5.8%	12.32
Mod 1 (HD)	20258	5.5%	12.34
Mod 1 (VX)	20260	5.8%	12.31
Mod 2 Chem A (HD)	21060	6.8%	12.32
Mod 2 Chem A (VX)	21060	6.4%	12.27
Mod 2 Chem B (HD)	21029	6.1%	12.31
Mod 2 Chem B (VX)	21060	6.2%	12.30

Dahlgren Decon Concentration and pH Results

Trial	Decontaminant Lot #	Concentration (Peracetic Acid)	pH
Quench (HD/VX)	8/2020	6.6%	6.92
Baseline (HD)	8/2020	7.9%	6.71
Baseline (VX)	8/2020	11.0%	6.69
Mod 1 (VX)	8/2020	7.8%	6.97
Mod 3 Chem A (HD)	16621-17421-16221	6.3%	6.84
Mod 3 Chem A (VX)	16621-17421-16221	8.3%	6.86

D7 Concentration and pH Results

Trial	Decontaminant Lot #	Concentration (Hydrogen Peroxide)	pH
Quench (HD/VX)	06-09-20-01M	4.3%	9.66
Baseline (HD)	06-09-20-01M	4.5%	9.58
Baseline (VX)	06-09-20-01M	4.9%	9.67
Mod 1 (HD)	06-09-20-01M	5.8%	9.71
Quench (Pesticides)	06-03-20-01M	1.2%	10.08
Baseline (Fipronil)	06-03-20-01M	4.5%	9.69
Baseline (Malathion)	06-03-20-01M	3.9%	9.66
Mod 1 (Fipronil)	06-09-20-01M	4.6%	9.51
Mod 1 (Malathion)	06-03-20-01M	4.5%	9.72

10x Diluted Bleach Concentration and pH Results

Trial	Decontaminant Lot #	Concentration (Hypochlorite)	pH
Quench (Pesticides)	55444-86-7	0.63%	11.54
Baseline (Fipronil)	55444-91-2	0.67%	11.38
Baseline (Malathion)	55444-94-2	0.63%	11.44

APPENDIX B

LABORATORY ENVIRONMENTAL CONDITIONS

Laboratory Environmental Conditions at Start of Test

Trial	Laboratory Environmental Conditions at Test Start	
	Temp (°F)	RH (%)
Wipe MD* (HD)	68.3	59.5
Wipe MD (VX)	68.4	59.3
Extraction MD (HD)	67.5	59.1
Extraction MD (VX)	68.5	59.1
Fate and Transport (HD)	67.6	58.9
Fate and Transport (VX)	66.8	62.4
Quench (HD)	68.3	17.0
Quench (VX)	68.3	17.0
Baseline (HD)	70.3	15.0
Baseline (VX)	69.2	22.0
Mod 1 (HD)	71.2	15.0
Mod 1 (VX)	70.4	24.6
Mod 2 Evaluation (HD)	72.7	15.0
Mod 2 Evaluation (VX)	71.7	36.2
Mod 2 Chem A (HD)	67.1	37.3
Mod 2 Chem A (VX)	69.5	50.3
Mod 2 Chem B (HD)	70.3	22.4
Mod 2 Chem B (VX)	70.9	17.6
Mod 3 Chem A (HD)	69.4	53.6
Mod 3 Chem A (VX)	70.3	33.6
Wipe MD (Fipronil)	69.5	24.3
Wipe MD (Malathion)	69.2	48.2
Wipe MD 2 (Malathion)	67.9	62.7
Extraction MD (Fipronil)	68.5	51.5
Extraction MD (Malathion)	69.2	54.4
Extraction MD 2 (Malathion)	68.4	57.7
Fate and Transport (Fipronil)	68.0	61.5
Fate and Transport (Malathion)	68.4	58.6
Quench (Fipronil)	71.5	52.3
Quench (Malathion)	69.5	55.2
Baseline (Fipronil)	68.7	55.7
Baseline (Malathion)	67.4	60.6
Mod 1 (Fipronil)	67.1	61.7
Mod 1 (Malathion)	69.8	38.3

*MD = method development

APPENDIX C

PERACETIC ACID TITRATION

Step	Action	Color
1	Set up a 300-mL Erlenmeyer flask in an ice bath on a stir plate.	
2	Insert a glass thermometer into the Erlenmeyer flask. *(Temperatures of solution were required not to exceed 5 °C during the titration)*	
3	Add 100 mL of 10% sulfuric acid to the Erlenmeyer flask.	
4	Add 2 drops of ferroin solution to the Erlenmeyer flask.	Orange
5	Add one drop of cerium (IV) sulfate solution to the Erlenmeyer flask.	Blue/nearly colorless
6	Weigh 0.3 g ± 0.05 g of prepared Dahlgren Decon and add to the Erlenmeyer flask.	Orange
7	Titrate dropwise with cerium (IV) sulfate solution.	Until changes back to blue/nearly colorless
8	Add 10 mL of potassium iodide solution and 5 mL starch solution to the Erlenmeyer flask.	Opaque brown
9	Immediately titrate dropwise with sodium thiosulfate solution (1 drop = 50 µL titrant).	Until changes to clear light orange

Reagent	Vendor	Catalpg Number
Sulfuric Acid, 10% (v/v) Aqueous Solution, Ricca Chemical	Fisher Scientific	8150-16
Honeywell Fluka™ Ferroin Indicator Solution, 0.025 M, Honeywell™ Fluka™	Fisher Scientific	60-046-934
Aqua Solutions Starch Solution1% 500 mL	Fisher Scientific	NC9195165
Sodium thiosulfate solution, Volumetric, Reag. Ph. Eur., 0.1 M $Na_2S_2O_3$ (0.1 N), Honeywell Fluka™	Fisher Scientific	60-026-21
Potassium Iodide Solution (10% w/v), Fisher Chemical™	Fisher Scientific	SP242-500
Cerium(IV) sulfate solution, 0.1M $Ce(SO_4)_2$, Honeywell™	Millipore Sigma	1090921003

Equipment used for the titration included the following:

- Glass burette, 25 mL with 0.1 mL increments
- Balance (Mettler Toledo, model # PG6002-S)
- Stir Plate (Fisherbrand Isotemp, catalog # SP88857200)
- Refrigerated/Heated Bath Circulator (Fisherbrand Isotemp, model # 6200 R28)
- Glass Thermometer (Thermco, -20 to 150 °C, Thomas Scientific catalog # 1221C73)

Calculation of percent peracetic acid concentration was performed using the below equation:

$$\%PAA = \frac{\left((mL\ Na_2S_2O_3)\times(Normality\ of\ titrant)\times\left(\frac{38.025\ eq}{mol}\right)\times(100\%)\right)}{\left((g\ Dahlgren\ Decon)\times\left(\frac{1000\ mL}{L}\right)\right)}$$

APPENDIX D

CWA STATISTICAL ANALYSIS

The objective of this statistical analysis was to compare total HD and VX mass recovered from each of two different materials (paint and sealant) and on each of three different sample types (surface wipes, extracted coupons, and extracted SPE disks) using eight different combinations of one of three decontaminants applied under one of four testing methods (see Table D1). Three replicates were tested for each condition. Table D2 summarizes the study design.

Additional evaluation was performed using positive control tests. The positive control tests excluded application of the target decontaminant but may include the preapplication of a chemical with a water rinse. The decontamination test conditions were compared to their analogous positive control tests to determine if statistically significant decontamination occurred. Positive controls were also compared between testing methods to evaluate whether the differences between methods might be attributable to external factors besides the decontamination process.

Table D1. Description of Testing Methods and Decontaminant Combinations

Testing Method	Dwell Time	Preapplication of Chemical with Water Rinse?	Decontaminant Application
Baseline	0 minutes	No	Bleach, D7, or Dahlgren Decon
Mod 1	120 minutes	No	Bleach, D7 (HD only), or Dahlgren Decon (VX only)
Mod 2	0 minutes	Yes (Chem A or B)	Bleach
Mod 3	0 minutes	Yes (Chem A only)	Dahlgren Decon

Observations below the method quantification limit (MQL) were set equal to the MQL, which was 1.1 µg for HD wipe masses and ranged from 0.001 µg to 0.11 µg for VX, dependent on sample type and required sample dilutions. Table D3 displays the percentage of observations below the MQL in each test condition, as well as the overall percentage of < MQL observations within each agent/material/sample type analysis. Many substitutions at the MQL value likely biases the estimates high and using a single substitution value artificially reduces the variance associated with the estimates. The reduction in variance may make the estimates more likely to be significantly different from other estimates when a real difference is not present.

For Mod 2 and Mod 3 where a preapplication of a chemical with a water rinse was performed, the mass recovery from the rinse data was added to the mass recovery from the wipe data to obtain the total mass recovered for each replicate. The counts of data below the MQL in the wipe conditions (see Table D3) reflect whether either the rinse or the wipe extraction mass was < MQL; samples with both rinse and wipe values below the MQL and samples with only one of the rinse or wipe values below the MQL were counted similarly.

Table D2. Study Design for Testing Method and Decontaminant Comparison

Agent	Material	Testing Method	Decontaminant	Number of Replicates		
				Wipe Samples	Extracted Coupon Samples	SPE Disk Samples
HD	Paint	Baseline	Bleach	3	3	3
HD	Paint	Baseline	Dahlgren Decon	3	3	3
HD	Paint	Baseline	D7	3	3	3
HD	Paint	Mod 1	Bleach	3	3	3
HD	Paint	Mod 1	D7	3	3	3
HD	Paint	Mod 2	Bleach + Chem A	3	3	3
HD	Paint	Mod 2	Bleach + Chem B	3	3	3
HD	Paint	Mod 3	Dahlgren Decon + Chem A	3	3	3
HD	Sealant	Baseline	Bleach	3	3	3
HD	Sealant	Baseline	Dahlgren Decon	3	3	3
HD	Sealant	Baseline	D7	3	3	3
HD	Sealant	Mod 1	Bleach	3	3	3
HD	Sealant	Mod 1	D7	3	3	3
HD	Sealant	Mod 2	Bleach + Chem A	3	3	3
HD	Sealant	Mod 2	Bleach + Chem B	3	3	3
HD	Sealant	Mod 3	Dahlgren Decon + Chem A	3	3	3
VX	Paint	Baseline	Bleach	3	3	3
VX	Paint	Baseline	Dahlgren Decon	3	3	3
VX	Paint	Baseline	D7	3	3	3
VX	Paint	Mod 1	Bleach	3	3	3
VX	Paint	Mod 1	Dahlgren Decon	3	3	3
VX	Paint	Mod 2	Bleach + Chem A	3	3	3
VX	Paint	Mod 2	Bleach + Chem B	3	3	3
VX	Paint	Mod 3	Dahlgren Decon + Chem A	3	3	3
VX	Sealant	Baseline	Bleach	3	3	3
VX	Sealant	Baseline	Dahlgren Decon	3	3	3
VX	Sealant	Baseline	D7	3	3	3
VX	Sealant	Mod 1	Bleach	3	3	3
VX	Sealant	Mod 1	Dahlgren Decon	3	3	3
VX	Sealant	Mod 2	Bleach + Chem A	3	3	3
VX	Sealant	Mod 2	Bleach + Chem B	3	3	3
VX	Sealant	Mod 3	Dahlgren Decon + Chem A	3	3	3

Table D3. Percent of Observations < MQL in Each Test Condition

Agent	Material	Testing Method	Decontaminant	Percent of Replicates		
				Wipe Samples	Samples	SPE Disk Samples
HD	Paint	Baseline	Bleach	100%	0%	0%
HD	Paint	Baseline	Dahlgren	100%	0%	0%
HD	Paint	Baseline	D7	0%	0%	0%
HD	Paint	Mod 1	Bleach	100%	0%	0%
HD	Paint	Mod 1	D7	100%	0%	0%
HD	Paint	Mod 2	Bleach + Chem A	100%	0%	0%
HD	Paint	Mod 2	Bleach + Chem B	100%	0%	0%
HD	Paint	Mod 3	Dahlgren Decon + Chem A	100%	0%	0%
HD	Paint	**Total**		87.5%	0%	0%
HD	Sealant	Baseline	Bleach	100%	0%	0%
HD	Sealant	Baseline	Dahlgren Decon	100%	0%	0%
HD	Sealant	Baseline	D7	0%	0%	0%
HD	Sealant	Mod 1	Bleach	100%	0%	0%
HD	Sealant	Mod 1	D7	0%	0%	0%
HD	Sealant	Mod 2	Bleach + Chem A	100%	0%	0%
HD	Sealant	Mod 2	Bleach + Chem B	100%	0%	0%
HD	Sealant	Mod 3	Dahlgren Decon + Chem A	100%	0%	0%
HD	Sealant	**Total**		75%	0%	0%
VX	Paint	Baseline	Bleach	0%	0%	0%
VX	Paint	Baseline	Dahlgren Decon	0%	0%	0%
VX	Paint	Baseline	D7	0%	0%	0%
VX	Paint	Mod 1	Bleach	0%	0%	0%
VX	Paint	Mod 1	Dahlgren Decon	0%	0%	0%
VX	Paint	Mod 2	Bleach + Chem A	0%	0%	0%
VX	Paint	Mod 2	Bleach + Chem B	0%	0%	0%
VX	Paint	Mod 3	Dahlgren Decon + Chem A	100%	0%	0%
VX	Paint	**Total**		12.5%	0%	0%
VX	Sealant	Baseline	Bleach	0%	0%	0%
VX	Sealant	Baseline	Dahlgren Decon	0%	0%	0%
VX	Sealant	Baseline	D7	0%	0%	0%
VX	Sealant	Mod 1	Bleach	0%	0%	66.7%
VX	Sealant	Mod 1	Dahlgren Decon	0%	0%	0%
VX	Sealant	Mod 2	Bleach + Chem A	0%	0%	0%
VX	Sealant	Mod 2	Bleach + Chem B	0%	0%	0%
VX	Sealant	Mod 3	Dahlgren Decon + Chem A	66.7%	0%	0%
VX	Sealant	**Total**		8.33%	0%	8.33%

CWA Analysis of Variance (ANOVA)

Comparison of Test Sample Results

A fixed effects ANOVA model was fitted to the total mass recovery data over all testing method and decontaminant combinations separately for each agent, material, and sample type condition. The models contained an effect for the combination of testing method and decontaminant and a residual error term. No random effect of trial was fitted due to only one trial being run for all replicates of each agent and material condition.

The assumptions of normally distributed errors with approximately equal variances were better met with untransformed data than with natural logarithm-transformed data, so data were left untransformed for the analysis. For some models, however, Levene's test [D1] still indicated that the Homogeneity of Variances assumption was not satisfied ($p < 0.05$). For the wipe samples for HD on paint and sealant, the unequal variances were believed to be driven by the large proportion of values below the MQL (see Table D3). For these models, residual variances estimates were first calculated from only the values above the MQL, and then were used for all conditions in the full model over all data. This approach assumes that, if the true masses for the < MQL samples could have been measured, then the variance in the corresponding data would have been similar to the variance of the samples measured above the MQL.

The models were fitted using SAS (version 9.4, 64-bit). The form of the model is presented in Equation D1.

$$Mass\ (\mu g) = \beta_0 + \beta_{ij} + \varepsilon_{ijk}$$

Equation D1

where:

- β_0 = intercept or overall mean total mass collected.
- β_{ij} = the fixed effect for the i^{th} testing method and j^{th} decontaminant.
- ε_{ijk} = random error for the k^{th} replicate from the i^{th} testing method, and j^{th} decontaminant. The random error is assumed to be $N(0, \sigma_\epsilon^2)$.

Using the model fitted to all test sample total mass recovery data for a given agent, material, and sample type, arithmetic means and 95% confidence intervals were calculated for the total mass recovered for each testing method and decontaminant combination. Pairwise comparisons were conducted to test for significant differences between pairs of testing method/decontaminant. There were 28 possible pairwise comparisons between the eight testing method/decontaminant combinations for each agent/material/sample type condition, but not all such comparisons were of interest. Instead, the pairwise comparisons performed were restricted to include:

1. The eight comparisons between testing methods with a shared decontaminant (e.g., the Baseline method with a bleach decontaminant vs Mod 1 with bleach).

2. The six comparisons between different decontaminants using the same testing method (e.g., the Baseline method with bleach vs Baseline with D7).

These pairwise comparisons amounted to 14 total comparisons for each of the 12 agent/material/sample type conditions, or 168 total comparisons. See also Table D4 and Table D5 for a summary of the comparisons performed for HD on paint and sealant and VX on paint and sealant, respectively. Conditions of the same color within a column were compared to each other, which resulted in seven HD comparisons for paint and seven for sealant, and six VX comparisons for paint and six for sealant.

Table D4. HD Paint and Sealant Comparison Sets

Test	Comparison Set 1	Comparison Set 2	Comparison Set 3	Sample Type		
Baseline	Bleach	Bleach	Bleach	Wipe	Extracted Coupon	SPE
	Dahlgren Decon	Dahlgren Decon	Dahlgren Decon	Wipe	Extracted Coupon	SPE
	D7	D7	D7	Wipe	Extracted Coupon	SPE
Mod 1	Bleach	Bleach	Bleach	Wipe	Extracted Coupon	SPE
	D7	D7	D7	Wipe	Extracted Coupon	SPE
Mod 2	Bleach/Chem A	Bleach/Chem A	Bleach/Chem A	Wipe	Extracted Coupon	SPE
	Bleach/Chem B	Bleach/Chem B	Bleach / Chem B	Wipe	Extracted Coupon	SPE
Mod 3	Dahlgren Decon/Chem A	Dahlgren Decon/Chem A	Dahlgren Decon/Chem A	Wipe	Extracted Coupon	SPE

Table D5. VX Paint and Sealant Comparison Sets

Test	Comparison Set 1	Comparison Set 2	Comparison Set 3	Sample Type		
Baseline	Bleach	Bleach	Bleach	Wipe	Extracted Coupon	SPE
	Dahlgren Decon	Dahlgren Decon	Dahlgren Decon	Wipe	Extracted Coupon	SPE
	D7	D7	D7	Wipe	Extracted Coupon	SPE
Mod 1	Bleach	Bleach	Bleach	Wipe	Extracted Coupon	SPE
	Dahlgren Decon	Dahlgren Decon	Dahlgren Decon	Wipe	Extracted Coupon	SPE
Mod 2	Bleach/Chem A	Bleach/Chem A	Bleach/Chem A	Wipe	Extracted Coupon	SPE
	Bleach/Chem B	Bleach/Chem B	Bleach/Chem B	Wipe	Extracted Coupon	SPE
Mod 3	Dahlgren Decon/Chem A	Dahlgren Decon/Chem A	Dahlgren Decon/Chem A	Wipe	Extracted Coupon	SPE

The Bonferroni-Holm multiple comparisons procedure was performed to adjust the *p*-values of the pairwise comparisons so that a familywise error rate of 0.05 was maintained over all fourteen comparisons of interest within an agent/material/sample type condition. This procedure limits the probability of a difference being falsely identified as statistically significant when no observable difference exists, and the difference is due to sampling variability. The familywise error rate means that the chance of a sampling-based falsely significant result is no more than 1 in 20 for the entire set of fourteen comparisons. The Bonferroni-Holm procedure was selected due to its power in detecting true differences when performing a restricted number of pairwise comparisons.

Comparison of Test Sample Results with Positive Controls

A fixed-effects ANOVA model was fitted to the total mass recovery data for decontaminants and positive controls results within each agent/material/sample type/testing method condition. In the cases for Mod 2 and Mod 3 where positive controls were tested both with and without the application of an additional chemical, only the positive controls from the conditions with chemical application were included in the model. The models contained an effect for the combination of decontaminant/positive control status and a residual error term. No random effect of trial was fitted due to only one trial being run for all replicates of each agent and material condition.

The assumptions of normality and equality of variances were better met with untransformed data than with natural logarithm-transformed data, so data were left untransformed for the analysis.

The models were fitted using SAS (version 9.4, 64-bit). The form of the model is presented in Equation D2.

$$Mass\ (\mu g) = \beta_0 + \beta_i + \varepsilon_{ij}$$

Equation D2

where:

- β_0 = intercept or overall mean total mass collected.
- β_i = the fixed effect for the i^{th} decontaminant / positive control condition.
- ε_{ij} = random error for the j^h replicate from the i^{th} decontaminant/positive control condition. The random error is assumed to be $N(0, \sigma_\epsilon^2)$.

Using the model fitted to all total mass recovery data for a given agent, material, and sample type, arithmetic means were calculated for the total mass recovered for each testing method and decontaminant combination. Pairwise comparisons were conducted to test for significant differences between each decontaminant and the positive control condition within an agent/material/sample type/testing method condition.

The Bonferroni-Holm multiple comparisons procedure was performed to adjust the *p*-values of the pairwise comparisons so that a familywise error rate of 0.05 was maintained over all comparisons of interest within an agent/material/sample type/testing method condition. The Bonferroni-Holm procedure was selected due to its power in detecting true differences when performing a restricted number of pairwise comparisons.

Comparison of Positive Control Results

A fixed effects ANOVA model was fitted to the total mass recovery data for positive controls results under each testing method within each agent/material/sample type condition. In the cases for Mod 2 and Mod 3 where positive controls were tested both with and without the application of an additional chemical, the positive controls from both the conditions with chemical application and those without the application were included in the model. The Fate and Transport data without Headspace for each agent/material/sample type were also included as a test method condition in this analysis. The models contained an effect for the combination of testing method under which the positive control was collected and the chemical application, and a residual error term. No random effect of trial was fitted due to only one trial being run for all replicates of each agent and material condition. Data were left untransformed for the analysis to remain consistent with analyses of test samples and test samples vs positive controls.

The models were fitted using SAS (version 9.4, 64-bit). The form of the model is presented in Equation D3.

$$Mass\ (\mu g) = \beta_0 + \beta_{ij} + \varepsilon_{ijk}$$

Equation D3

where:

- β_0 = intercept or overall mean total mass collected.
- β_{ij} = the fixed effect for the positive controls from the i^{th} testing method and j^{th} chemical application condition.
- ε_{ijk} = random error for the k^{h} positive control from the i^{th} testing method/j^{th} chemical application condition. The random error is assumed to be $N(0, \sigma_\epsilon^2)$.

Using the model fitted to all positive control data for a given agent, material, and sample type, arithmetic means were calculated for the total mass recovered for each testing method and decontaminant combination for each agent, material, and sample type. Pairwise comparisons were conducted to test for significant differences between all nine positive controls conditions with an agent/material/sample type, resulting in 36 pairwise comparisons per model.

The Bonferroni-Holm multiple comparisons procedure was performed to adjust the *p*-values of the pairwise comparisons so that a familywise error rate of 0.05 was maintained over all comparisons of interest within an agent/material/sample type. The Bonferroni-Holm procedure was selected for consistency with the previous analyses.

Outliers

Potential outliers were determined by calculating the deleted (externally) studentized residuals. If the absolute value of the standardized residual was greater than 3, then the observation was considered a potential outlier. If potential outliers were found, the results were checked to determine the validity of the outlying data and probable causes for the outliers. If no probable cause was found, the outlier was included in the subsequent analysis.

CWA ANOVA Results

Table D6 through Table D8 display the potential outliers identified by examining the externally studentized residuals in each study condition. Two of the outliers from the SPE disks in the VX Sealant condition were excluded from all analyses after investigator confirmation that observed data may not have reflected the intended experimental outcomes (denoted with * in the tables below).

Table D6. Potential Outliers Identified from Test Samples

Analysis	Agent	Material	Sample Type	Test Method	Decontaminant	Total Mass Recovery (µg)	Externally Studentized Residual
Test Samples	HD	Paint	SPE	Baseline	Bleach	1266.50	3.46
	HD	Sealant	Extracted Coupon	Baseline	D7	194.30	-3.58
	VX	Paint	Wipe	Baseline	Bleach	94.72	3.46
	VX	Paint	Extracted Coupon	Mod 2	Bleach + Chem A	9.60	-3.58
	VX	Sealant	Wipe	Mod 2	Bleach + Chem A	467.58	-3.01
	VX	Sealant	Wipe	Mod 2	Bleach + Chem A	241.15	-3.17
	VX	Sealant	SPE	Mod 1	Bleach	59.61	4.30

Table D7. Potential Outliers Identified from Test Samples vs. Positive Controls

Analysis	Agent	Material	Sample Type	Test Method	Decontaminant or Positive Control (PC)	Total Mass Recovery (µg)	Externally Studentized Residual
Test Samples vs. Positive Control Comparison	HD	Paint	Wipe	Baseline	PC	2.84	-9.82
	HD	Paint	Wipe	Mod 1	PC	12.0	12.4
	HD	Paint	Extracted Coupon	Mod 2 + Chem A	PC	173	-4.76
	HD	Paint	Extracted Coupon	Mod 2 + Chem B	PC	274	-5.06
	HD	Paint	SPE	Baseline	Bleach	1270	3.08
	HD	Paint	SPE	Mod 2 + Chem A	PC	1400	3.65
	HD	Sealant	Wipe	Baseline	D7	2.33	-20.6
	HD	Sealant	Wipe	Mod 1	D7	4.26	19.9
	HD	Sealant	Wipe	Mod 2 + Chem A	PC	9.49	-3.57
	HD	Sealant	Wipe	Mod 2 + Chem B	PC	16.1	5.24
	HD	Sealant	Wipe	Mod 3 + Chem A	PC	7.13	-4.01
	HD	Sealant	Extracted Coupon	Mod 2 + Chem B	PC	244.1	4.96
	VX	Paint	Wipe	Mod 2 + Chem B	Bleach + Chem B	210.1	4.46
	VX	Paint	Wipe	Mod 3 + Chem A	Positive Control	303.8	3.52
	VX	Paint	Extracted Coupon	Baseline	Bleach	321.3	3.73
	VX	Paint	Extracted Coupon	Mod 2 + Chem A	Bleach + Chem A	9.60	-4.26
	VX	Paint	Extracted Coupon	Mod 2 – Chem B	PC	195	6.00
	VX	Paint	Extracted Coupon	Mod 3 + Chem A	Dahlgren + Chem A	129	5.22
	VX	Paint	SPE	Mod 2 + Chem B	PC	59.4	-4.23
	VX	Sealant	Wipe	Baseline	PC	35.3	3.37
	VX	Sealant	Wipe	Mod 1	PC	30.0	-4.42
	VX	Sealant	Wipe	Mod 1	PC	40.6	3.39
	VX	Sealant	Wipe	Mod 2 + Chem B	PC	400	3.39
	VX	Sealant	Wipe	Mod 3 + Chem A	PC	560	6.14
	VX	Sealant	Extracted Coupon	Mod 1	Bleach	421	-3.42
	VX	Sealant	Extracted Coupon	Mod 2 + Chem B	PC	300	-3.11
	VX	Sealant	SPE	Mod 1	Bleach	59.6*	10.02
	VX	Sealant	SPE	Mod 2 + Chem A	PC	55.5	12.06

Analysis	Agent	Material	Sample Type	Test Method	Decontaminant or Positive Control (PC)	Total Mass Recovery (µg)	Externally Studentized Residual
	VX	Sealant	SPE	Mod 2 + Chem B	PC	2.93	75.67
	VX	Sealant	SPE	Mod 3 + Chem A	PC	4.89	-5.98

*Outlier excluded from analyses.

Table D8. Potential Outliers Identified from Positive Controls Analysis

Analysis	Agent	Material	Sample Type	Test Method	Decontaminant or Positive Control (PC)	Total Mass Recovery (µg)	Externally Studentized Residual
Positive Controls	HD	Paint	Extracted Coupon	Fate & Transport	Fate & Transport	893	4.73
	HD	Paint	SPE	Fate & Transport	Fate & Transport	1100	-3.81
	HD	Sealant	Wipe	Mod 3 + Chem A	PC + Chem A	7.13	-4.59
	HD	Sealant	SPE	Fate & Transport	Fate & Transport	1050	-9.35
	VX	Paint	Wipe	Fate & Transport	Fate & Transport	670	7.16
	VX	Paint	Extracted Coupon	Fate & Transport	Fate & Transport	1297	7.37
	VX	Paint	Extracted Coupon	Fate & Transport	Fate & Transport	826	-3.14
	VX	Sealant	Wipe	Mod 2 + Chem B	PC + Chem B	400	4.13
	VX	Sealant	Extracted Coupon	Mod 2 – Chem A	PC	436	-3.03
	VX	Sealant	SPE	Fate & Transport	Fate & Transport	459	4.30
	VX	Sealant	SPE	Mod 2 – Chem A	PC	324*	3.05

*Outlier excluded from the analyses.

Figure D1 to Figure D2 displays the total mass recoveries for the replicates in each agent/material/sample type condition. Statistical summaries including arithmetic means and 95% confidence intervals are presented in Table D9 to Table D12 and are sorted in order of estimated mean total mass recovery within each agent, material, and sample type. Confidence bounds were not adjusted for multiple comparisons between test conditions and thus should not be used to evaluate significant differences between test conditions.

Figure D1. Total recovery mass of test samples for all testing method and decontaminant combinations over all sample types for HD on Paint

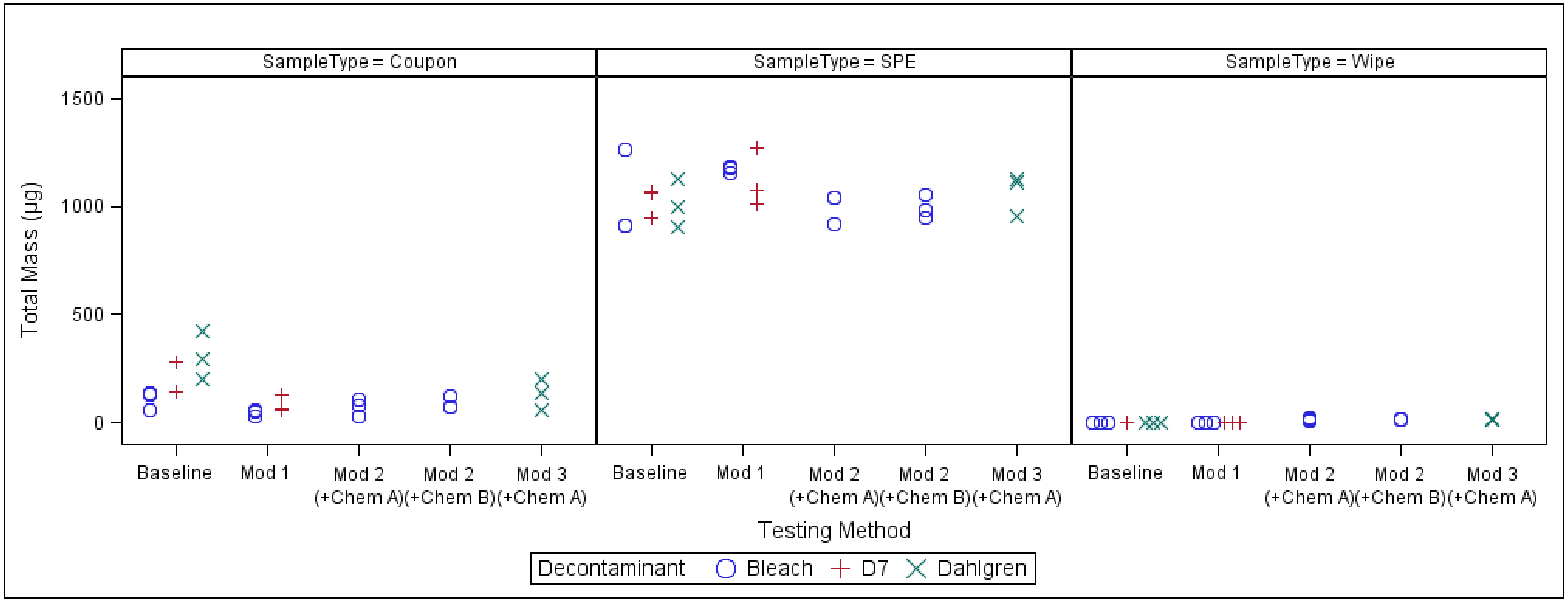

Figure D2. Total recovery mass of test samples for all testing method and decontaminant combinations over all sample types for HD on Sealant

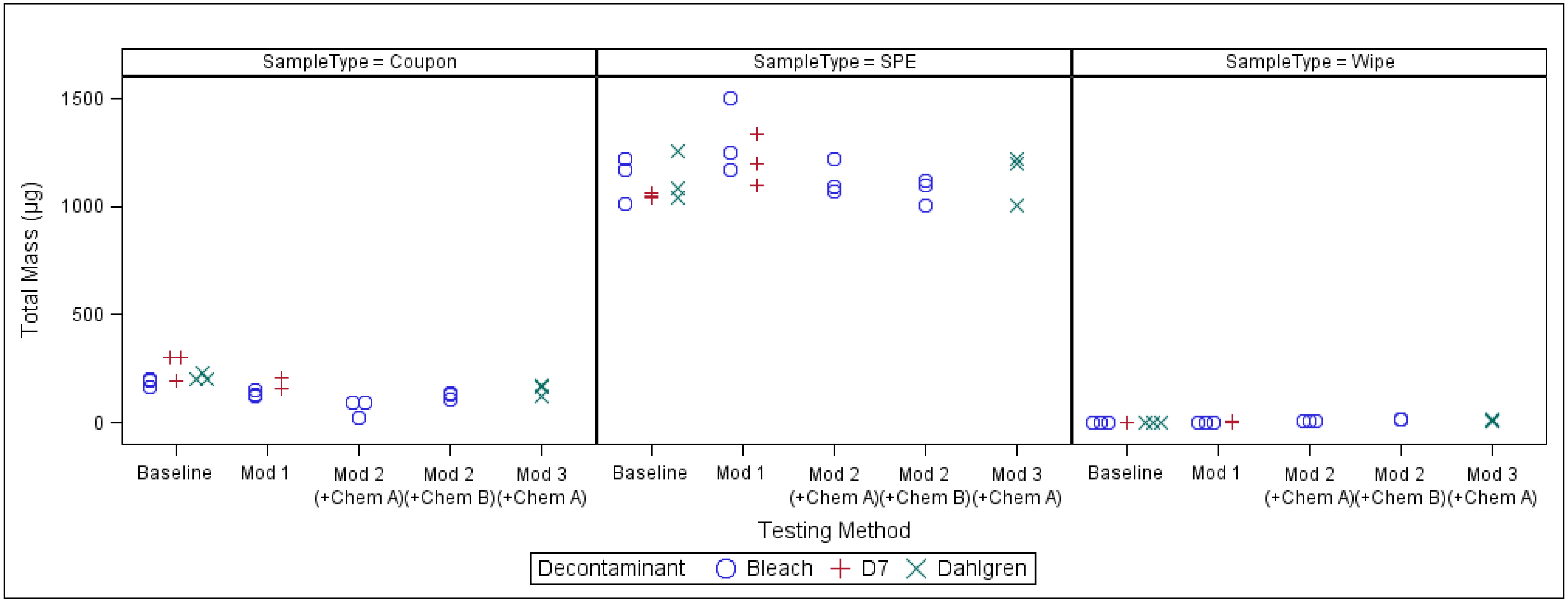

Figure D3. Total recovery mass of test samples for all testing method and decontaminant combinations over all sample types for VX on Paint

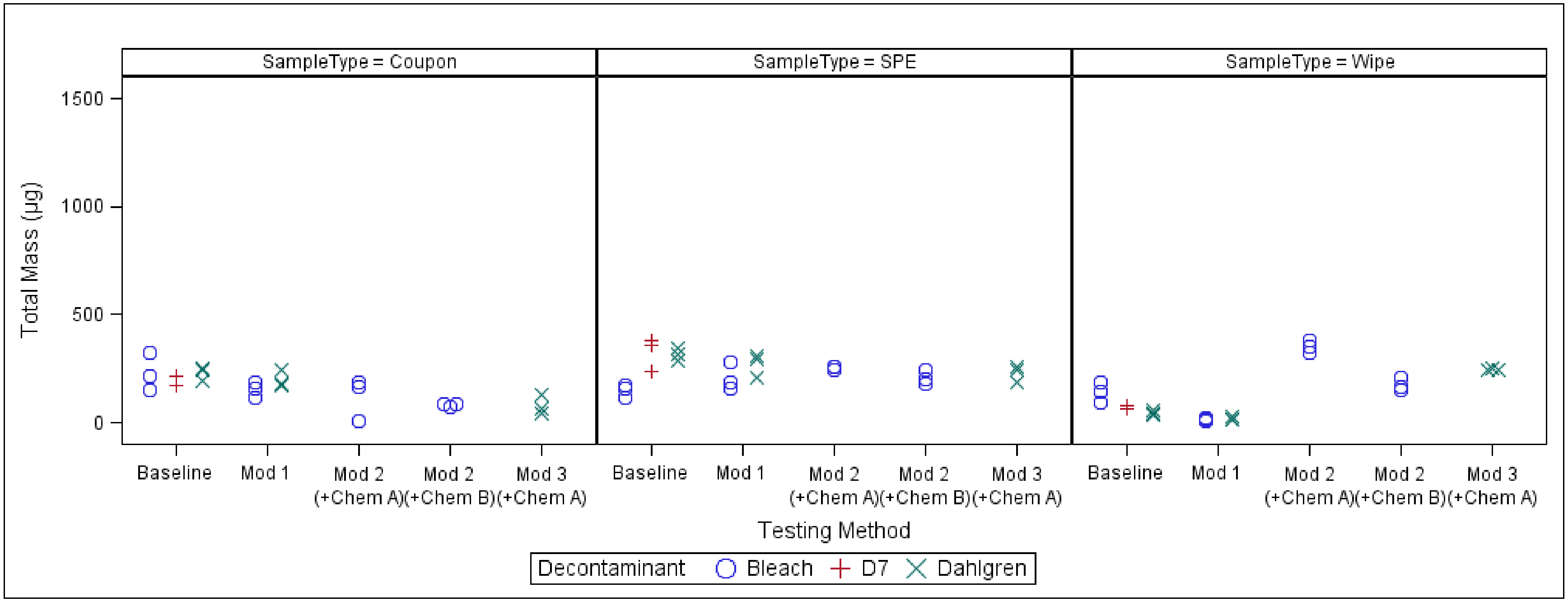

Figure D4. Total recovery mass of test samples for all testing method and decontaminant combinations over all sample types for VX on Sealant

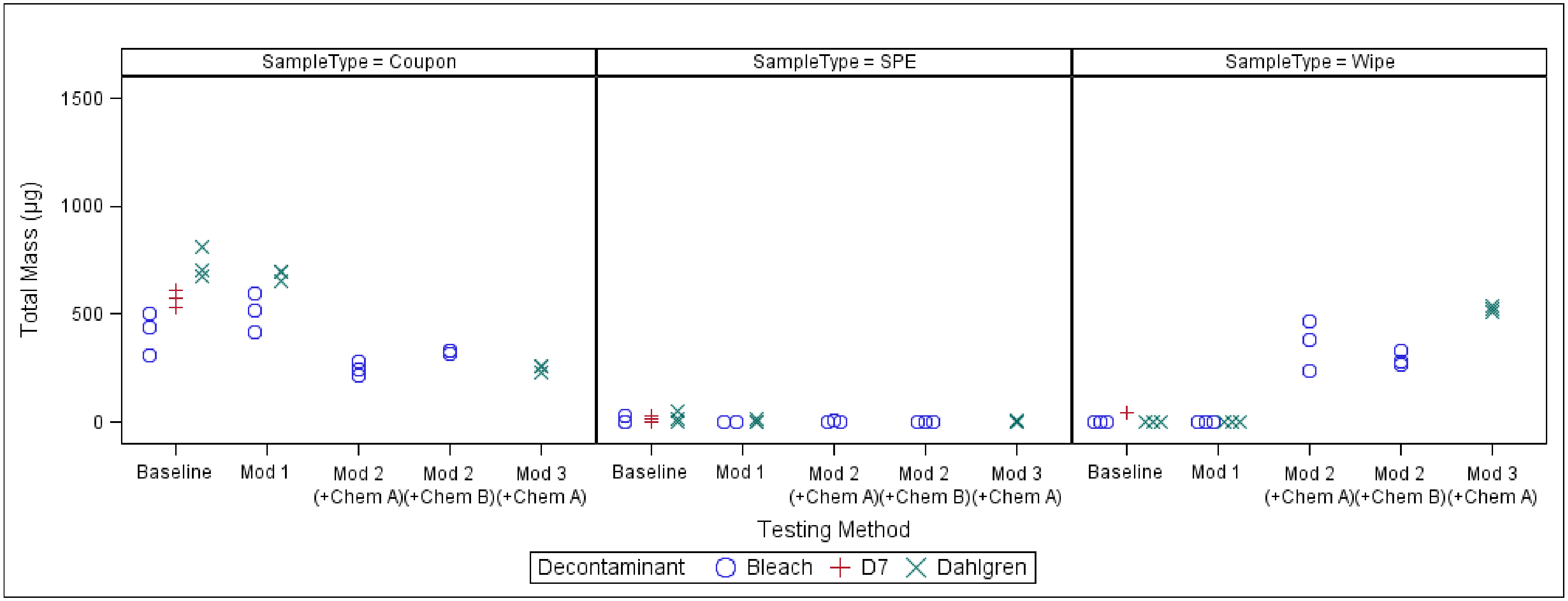

Table D9. Sorted Arithmetic Means and Unadjusted 95% Confidence Intervals (HD-Paint)

Agent	Material	Sample Type	Test Method	Decontaminant	Mean Total Mass Recovery (µg)	Lower 95% Confidence Bound	Upper 95% Confidence Bound
HD	Paint	Wipe	Baseline	Bleach	<MQL	0.27	1.93
HD	Paint	Wipe	Baseline	Dahlgren Decon	<MQL	0.27	1.93
HD	Paint	Wipe	Mod 1	Bleach	<MQL	0.27	1.93
HD	Paint	Wipe	Mod 1	D7	<MQL	0.27	1.93
HD	Paint	Wipe	Baseline	D7	2.18	1.35	3.01
HD	Paint	Wipe	Mod 3	Chem A/Dahlgren Decon	14.21	13.4	15.0
HD	Paint	Wipe	Mod 2	Chem A/Bleach	15.25	14.4	16.1
HD	Paint	Wipe	Mod 2	Chem B/Bleach	17.13	16.3	18.0
HD	Paint	Extracted Coupon	Mod 1	Bleach	46.57	<MQL	121
HD	Paint	Extracted Coupon	Mod 2	Chem A/Bleach	73.67	<MQL	149
HD	Paint	Extracted Coupon	Mod 1	D7	82.44	7.27	158
HD	Paint	Extracted Coupon	Mod 2	Chem B/Bleach	88.02	12.9	163
HD	Paint	Extracted Coupon	Baseline	Bleach	108.53	33.3	184
HD	Paint	Extracted Coupon	Mod 3	Chem A/Dahlgren Decon	132.12	57.0	207
HD	Paint	Extracted Coupon	Baseline	D7	235.23	160	310
HD	Paint	Extracted Coupon	Baseline	Dahlgren Decon	307.70	233	383
HD	Paint	SPE	Mod 2	Chem B/Bleach	997.58	864	1131
HD	Paint	SPE	Mod 2	Chem A/Bleach	1003.94	870	1137
HD	Paint	SPE	Baseline	Dahlgren Decon	1011.63	878	1145
HD	Paint	SPE	Baseline	D7	1026.53	893	1160
HD	Paint	SPE	Baseline	Bleach	1029.57	896	1163
HD	Paint	SPE	Mod 3	Chem A/Dahlgren Decon	1064.59	931	1198
HD	Paint	SPE	Mod 1	D7	1122.73	989	1256
HD	Paint	SPE	Mod 1	Bleach	1174.26	1041	1308

Table D10. Sorted Arithmetic Means and Unadjusted 95% Confidence Intervals (HD-Sealant)

Agent	Material	Sample Type	Test Method	Decontaminant	Mean Total Mass Recovery (µg)	Lower 95% Confidence Bound	Upper 95% Confidence Bound
HD	Sealant	Wipe	Mod 1	Bleach	<MQL	0.50	1.70
HD	Sealant	Wipe	Baseline	Bleach	<MQL	0.50	1.70
HD	Sealant	Wipe	Baseline	Dahlgren Decon	<MQL	0.50	1.70
HD	Sealant	Wipe	Baseline	D7	2.96	2.36	3.56
HD	Sealant	Wipe	Mod 1	D7	3.78	3.18	4.38
HD	Sealant	Wipe	Mod 2	Chem A/Bleach	9.15	8.55	9.75
HD	Sealant	Wipe	Mod 3	Chem A/Dahlgren Decon	10.5	9.87	11.1
HD	Sealant	Wipe	Mod 2	Chem B/Bleach	12.6	12.0	13.2
HD	Sealant	Extracted Coupon	Mod 2	Chem A/Bleach	68.2	28.6	108
HD	Sealant	Extracted Coupon	Mod 2	Chem B/Bleach	127	86.2	165
HD	Sealant	Extracted Coupon	Mod 1	Bleach	134	94.5	174
HD	Sealant	Extracted Coupon	Mod 3	Chem A/Dahlgren Decon	155	115	194
HD	Sealant	Extracted Coupon	Mod 1	D7	176	137	216
HD	Sealant	Extracted Coupon	Baseline	Bleach	188	148	227
HD	Sealant	Extracted Coupon	Baseline	Dahlgren Decon	209	170	249
HD	Sealant	Extracted Coupon	Baseline	D7	266	226	305
HD	Sealant	SPE	Baseline	D7	1054	922	1185
HD	Sealant	SPE	Mod 2	Chem B/Bleach	1075	943	1207
HD	Sealant	SPE	Baseline	Dahlgren Decon	1129	997	1260
HD	Sealant	SPE	Mod 2	Chem A/Bleach	1130	999	1264
HD	Sealant	SPE	Baseline	Bleach	1136	1004	1267
HD	Sealant	SPE	Mod 3	Chem A/Dahlgren Decon	1144	1012	1276
HD	Sealant	SPE	Mod 1	D7	1212	1080	1343
HD	Sealant	SPE	Mod 1	Bleach	1307	1176	1439

Table D11. Sorted Arithmetic Means and Unadjusted 95% Confidence Intervals (VX-Paint)

Agent	Material	Sample Type	Test Method	Decontaminant	Mean Total Mass Recovery (µg)	Lower 95% Confidence Bound	Upper 95% Confidence Bound
VX	Paint	Wipe	Mod 1	Bleach	14.7	<MQL	42.7
VX	Paint	Wipe	Mod 1	Dahlgren Decon	18.1	<MQL	46.1
VX	Paint	Wipe	Baseline	Dahlgren Decon	46.4	18.4	74.4
VX	Paint	Wipe	Baseline	D7	70.4	42.4	98.4
VX	Paint	Wipe	Baseline	Bleach	140	112	168
VX	Paint	Wipe	Mod 2	Chem B/Bleach	175	147	203
VX	Paint	Wipe	Mod 3	Chem A/Dahlgren	250	222	278
VX	Paint	Wipe	Mod 2	Chem A/Bleach	353	325	381
VX	Paint	Extracted Coupon	Mod 3	Chem A/Dahlgren Decon	80.0	14.3	146
VX	Paint	Extracted Coupon	Mod 2	Chem B/Bleach	83.7	18.1	149
VX	Paint	Extracted Coupon	Mod 2	Chem A/Bleach	121	55.0	186
VX	Paint	Extracted Coupon	Mod 1	Bleach	157	91.4	223
VX	Paint	Extracted Coupon	Baseline	D7	187	121	252
VX	Paint	Extracted Coupon	Mod 1	Dahlgren Decon	201	136	267
VX	Paint	Extracted Coupon	Baseline	Dahlgren Decon	230	164	295
VX	Paint	Extracted Coupon	Baseline	Bleach	230	164	295
VX	Paint	SPE	Baseline	Bleach	148	91.2	205
VX	Paint	SPE	Mod 2	Chem B/Bleach	209	152	266
VX	Paint	SPE	Mod 1	Bleach	210	153	267
VX	Paint	SPE	Mod 3	Chem A/Dahlgren Decon	233	176	290
VX	Paint	SPE	Mod 2	Chem A/Bleach	253	197	310
VX	Paint	SPE	Mod 1	Dahlgren Decon	273	217	330
VX	Paint	SPE	Baseline	Dahlgren Decon	317	261	374
VX	Paint	SPE	Baseline	D7	326	269	383

Table D12. Sorted Arithmetic Means and Unadjusted 95% Confidence Intervals (VX-Sealant)

Agent	Material	Sample Type	Test Method	Decontaminant	Mean Total Mass Recovery (µg)	Lower 95% Confidence Bound	Upper 95% Confidence Bound
VX	Sealant	Wipe	Mod 1	Bleach	0.0049	<MQL	52.1
VX	Sealant	Wipe	Mod 1	Dahlgren Decon	0.37	<MQL	52.4
VX	Sealant	Wipe	Baseline	Dahlgren Decon	0.97	<MQL	53.0
VX	Sealant	Wipe	Baseline	Bleach	1.14	<MQL	53.2
VX	Sealant	Wipe	Baseline	D7	42.1	<MQL	94.2
VX	Sealant	Wipe	Mod 2	Chem B/Bleach	294	242	346
VX	Sealant	Wipe	Mod 2	Chem A/Bleach	364	312	416
VX	Sealant	Wipe	Mod 3	Chem A/Dahlgren Decon	524	472	576
VX	Sealant	Extracted Coupon	Mod 2	Chem A/Bleach	247	176	317
VX	Sealant	Extracted Coupon	Mod 3	Chem A/Dahlgren Decon	251	180	321
VX	Sealant	Extracted Coupon	Mod 2	Chem B/Bleach	327	257	398
VX	Sealant	Extracted Coupon	Baseline	Bleach	417	347	488
VX	Sealant	Extracted Coupon	Mod 1	Bleach	513	443	584
VX	Sealant	Extracted Coupon	Baseline	D7	572	502	643
VX	Sealant	Extracted Coupon	Mod 1	Dahlgren Decon	683	613	754
VX	Sealant	Extracted Coupon	Baseline	Dahlgren Decon	730	660	801
VX	Sealant	SPE	Mod 1	Dahlgren Decon	<MQL	<MQL	19.6
VX	Sealant	SPE	Mod 2	Chem B/Bleach	0.041	<MQL	16.0
VX	Sealant	SPE	Mod 2	Chem A/Bleach	2.15	<MQL	18.1
VX	Sealant	SPE	Mod 3	Chem A/Dahlgren Decon	3.28	<MQL	19.3
VX	Sealant	SPE	Mod 1	Dahlgren Decon	5.65	<MQL	21.6
VX	Sealant	SPE	Baseline	Bleach	9.92	<MQL	25.9
VX	Sealant	SPE	Baseline	D7	15.3	<MQL	31.3
VX	Sealant	SPE	Baseline	Dahlgren Decon	23.3	7.30	39.3

Table D15 to Table D92 display the results of the Bonferroni-Holm-adjusted pairwise comparisons for the specified comparisons between the sample test conditions. Of the 168 pairwise comparisons between agent/material/sample type condition combinations, 51 were statistically significant.

The capital letters in the “Similarity Designation” column of Table D15 to Table D92 indicate the statistical similarity of the mean total mass of a given testing method and decontaminant combination to that of all other combinations tested for the given agent/material/sample type condition. All rows with the same similarity designation value are not statistically significantly different from each other, while rows that did not share any similarity designation values are significantly different. For example, in Table D15 for the HD/Paint/Wipe condition, the baseline method with the bleach decontaminant has similarity designation A, indicating that it is similar to other combinations with the A designation, including Mod 1 with bleach (designation A), but it is different from combinations without an A in the designation, such as Mod 2 with bleach (designation C).

The results of the test sample comparisons are summarized below.

Bleach comparisons:

- Bleach Mod 1 paint wipe samples resulted in lower HD values (lower HD recovery) compared to Mod 2 with Chem A and Mod 2 with Chem B. Note that bleach Mod 1 was not different from Baseline for HD paint wipes.
- Bleach Mod 1 sealant wipe samples resulted in lower HD values compared to Mod 2 with Chem A and Mod 2 with Chem B. Note that bleach Mod 1 was not different from Baseline for HD sealant wipes.
- Bleach Mod 1 paint wipe resulted in lower VX values than Baseline, Mod 2 with Chem A and Mod 2 with Chem B.
- Bleach Mod 1 sealant wipe samples resulted in lower VX values compared to Mod 2 with Chem A and Mod 2 with Chem B. Note that bleach Mod 1 was not different from Baseline for VX sealant wipes.

Dahlgren Decon comparisons and D7 comparisons:

- Mod 1 with D7 typically did not result in improved HD decontamination compared to Baseline for any material/sample type.
- Mod 3 with Dahlgren typically did not result in improved VX decontamination compared to Baseline for any material/sample type.

Baseline comparisons:

- Across all material and sample types, bleach resulted in the greatest number of significantly lower HD and VX recoveries (6 out of 36 comparisons).

Modification 1 comparisons:

- Most Mod 1 comparisons of bleach to D7 did not have significantly different HD recoveries across all material and sample types.
- Most Mod 1 comparisons of bleach to Dahlgren did not have significantly different VX recoveries across all material and sample types.

Modification 2 comparisons:

- Most comparisons were not significantly different. Lower HD and VX recoveries occurred only for wipe samples.
- Where differences did exist, there was no clear pattern as to whether Chem A or Chem B provided improved decontamination results.

Chemical A comparisons:

- Most comparisons were not significantly different. Where differences did exist, there was no clear pattern as to whether bleach or Dahlgren Decon provided improved decontamination.

Table D93 to Table D104 display the results of the Bonferroni-Holm-adjusted pairwise comparisons between the test samples and positive controls. A summary of these results showing the significant differences for the 8 comparisons between test samples to positive controls for each agent/material/sample combination is shown in Table D13. All the HD paint and sealant wipe differences occurred for Baseline and Mod 1 tests while all the HD paint and sealant extracted coupon differences occurred for Mod 1, Mod 2, and Mod 3. In all cases, the difference was due to the test samples being less than the positive controls. Relative to the positive controls, Mod 1 with D7 resulted in significantly lower HD levels on the surface of paint and sealant films and within the extracted coupons of paint and sealant films. Relative to the positive controls, Mod 1 with bleach resulted in significantly lower HD levels on the surface of paint films and within the extracted coupon of paint and sealant films. All the VX paint wipe differences occurred for Modification 1 and all of the VX sealant wipe and extracted coupon differences occurred for Baseline and Modification 1.

Table D13. Summary of Test Sample to Positive Control Comparisons

Chemical	Material	Wipe	Extracted Coupon	SPE
HD	Paint	5 differences	5 differences	No differences
	Sealant	2 differences	4 differences	No differences
VX	Paint	2 differences	1 difference	1 difference
	Sealant	5 differences	4 differences	No differences

Table D105 to Table D116 display the results of the Bonferroni-Holm-adjusted pairwise comparisons between the positive control conditions. A summary of these results showing significant differences for the 36 comparisons between positive controls for each agent/material /sample combination is shown in Table D14. Wipe differences were driven primarily by comparison of positive controls that included Chemical A or B (where rinse results were added to wipe results) to the positive controls that did not include Chemical A or B, indicating an effect of Chemical A or B in combination with water rinse on the positive controls. The HD sealant and VX paint extracted coupon differences were associated primarily with comparisons to the Fate and Transport Data; the reason for these differences is not apparent. Of particular note are the large number of differences for the VX sealant extracted coupons. These differences may be driven by variable permeation of VX into the sealant extracted coupon, with deeper permeation leading to lower extraction recoveries.

Table D14. Summary of Positive Control Comparisons

Chemical	Material	Wipe	Extracted Coupon	SPE
HD	Paint	7 differences	No differences	1 Difference
	Sealant	17 differences	6 differences	1 Difference
VX	Paint	10 differences	8 differences	No differences
	Sealant	20 differences	27 differences	No differences

Table D15. Multiple comparison adjusted p-values between test method for bleach (HD on Paint Coupon Surfaces)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Bleach			Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2 (Chem A)	Mod 2 (Chem B)	
HD	Paint	Wipe	Bleach	<MQL	Baseline	A	1.0000	<0.0001	<0.0001	Base < Mod 2A Base < Mod2B Mod 1 < Mod 2A Mod 1 < Mod 2B Mod 2A< Mod 2B
HD	Paint	Wipe	Bleach	<MQL	Mod 1	A		<0.0001	<0.0001	
HD	Paint	Wipe	Bleach + Chem A	15.2	Mod 2	B			<0.0001	
HD	Paint	Wipe	Bleach + Chem B	17.1	Mod 2	C				

Table D16. Multiple comparison adjusted p-values between test method for bleach (HD in Paint Coupons)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Bleach			Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2 (Chem A)	Mod 2 (Chem B)	
HD	Paint	Extracted Coupon	Bleach	109	Baseline	A	1.0000	1.0000	1.0000	No significant differences.
HD	Paint	Extracted Coupon	Bleach	46.6	Mod 1	A		1.0000	1.0000	
HD	Paint	Extracted Coupon	Bleach + Chem A	73.7	Mod 2	A			1.000	
HD	Paint	Extracted Coupon	Bleach + Chem B	88.0	Mod 2	A				

Table D17. Multiple comparison adjusted p-values between test method for bleach (HD in SPE Disks)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Bleach			Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2 (Chem A)	Mod 2 (Chem B)	
HD	Paint	SPE	Bleach	1030	Baseline	A	1.0000	1.0000	1.0000	No significant differences.
HD	Paint	SPE	Bleach	1174	Mod 1	A		0.9566	0.9019	
HD	Paint	SPE	Bleach + Chem A	1004	Mod 2	A			1.0000	
HD	Paint	SPE	Bleach + Chem B	998	Mod 2	A				

Table D18. Multiple comparison adjusted p-values between test methods for bleach (HD on Sealant Coupon Surfaces)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Decontaminants	Testing Method	Similarity Designation	Bleach			Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2 (Chem A)	Mod 2 (Chem B)	
HD	Sealant	Wipe	<MQL	Bleach	Baseline	A	1.0000	<0.0001	<0.0001	Base < Mod 2A Base < Mod2B Mod 1 < Mod 2A Mod 1 < Mod 2B Mod 2A< Mod 2B
HD	Sealant	Wipe	<MQL	Bleach	Mod 1	A		<0.0001	<0.0001	
HD	Sealant	Wipe	9.15	Bleach + Chem A	Mod 2	B			<0.0001	
HD	Sealant	Wipe	12.6	Bleach + Chem B	Mod 2	C				

Table D19. Multiple comparison adjusted p-values between test methods for bleach (HD in Sealant Coupons)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Decontaminants	Testing Method	Similarity Designation	Bleach			Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2 (Chem A)	Mod 2 (Chem B)	
HD	Sealant	Extracted Coupon	188	Bleach	Baseline	A	0.3549	0.0048	0.2883	Mod 2A < Baseline
HD	Sealant	Extracted Coupon	134	Bleach	Mod 1	AB		0.2382	0.8529	
HD	Sealant	Extracted Coupon	68.2	Bleach + Chem A	Mod 2	B			0.3549	
HD	Sealant	Extracted Coupon	126	Bleach + Chem B	Mod 2	AB				

Table D20. Multiple comparison adjusted p-values between test methods for bleach (HD in SPE Disks)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Decontaminants	Testing Method	Similarity Designation	Bleach			Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2 (Chem A)	Mod 2 (Chem B)	
HD	Sealant	SPE	1136	Bleach	Baseline	A	0.8229	1.0000	1.0000	No significant differences.
HD	Sealant	SPE	1307	Bleach	Mod 1	A		0.7945	0.2470	
HD	Sealant	SPE	1130	Bleach + Chem A	Mod 2	A			1.0000	
HD	Sealant	SPE	1075	Bleach + Chem B	Mod 2	A				

Table D21. Multiple comparison adjusted p-values between test methods for bleach (VX on Paint Coupon Surfaces)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Bleach			Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2 (Chem A)	Mod 2 (Chem B)	
VX	Paint	Wipe	Bleach	141	Baseline	A	<0.0001	<0.0001	0.3453	Base < Mod 2A Mod 1 < Base Mod 1 < Mod 2A Mod 1 < Mod 2B Mod 2B< Mod 2A
VX	Paint	Wipe	Bleach	14.7	Mod 1	B		<0.0001	<0.0001	
VX	Paint	Wipe	Bleach + Chem A	353	Mod 2	C			<0.0001	
VX	Paint	Wipe	Bleach + Chem B	175	Mod 2	A				

Table D22. Multiple comparison adjusted p-values between test methods for bleach (VX in Paint Coupons)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Bleach			Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2 (Chem A)	Mod 2 (Chem B)	
VX	Paint	Extracted Coupon	Bleach	230	Baseline	A	1.0000	0.2897	0.0592	No significant differences.
VX	Paint	Extracted Coupon	Bleach	157	Mod 1	A		1.0000	1.0000	
VX	Paint	Extracted Coupon	Bleach + Chem A	121	Mod 2	A			1.0000	
VX	Paint	Extracted Coupon	Bleach + Chem B	83.8	Mod 2	A				

Table D23. Multiple comparison adjusted p-values between test methods for bleach (VX in SPE Disks)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Bleach			Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2 (Chem A)	Mod 2 (Chem B)	
VX	Paint	SPE	Bleach	148	Baseline	A	1.0000	0.1704	1.0000	No significant differences.
VX	Paint	SPE	Bleach	210	Mod 1	A		1.0000	1.0000	
VX	Paint	SPE	Bleach + Chem A	253	Mod 2	A			1.0000	
VX	Paint	SPE	Bleach + Chem B	209	Mod 2	A				

Table D24. Multiple comparison adjusted p-values between test methods for bleach (VX on Sealant Coupon Surfaces)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Bleach			Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2 (Chem A)	Mod 2 (Chem B)	
VX	Sealant	Wipe	Bleach	1.14	Baseline	A	1.0000	<0.0001	<0.0001	Base < Mod 2A Base < Mod 2B Mod 1 < Mod 2A Mod 1 < Mod 2B
VX	Sealant	Wipe	Bleach	0.0049	Mod 1	A		<0.0001	<0.0001	
VX	Sealant	Wipe	Bleach + Chem A	364	Mod 2	B			0.4798	
VX	Sealant	Wipe	Bleach + Chem B	294	Mod 2	B				

Table D25. Multiple comparison adjusted p-values between test methods for bleach (VX in Sealant Coupons)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Bleach			Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2 (Chem A)	Mod 2 (Chem B)	
VX	Sealant	Extracted Coupon	Bleach	417	Baseline	AB	0.3494	0.0226	0.3667	Mod 2A < Base Mod 2A < Mod 1 Mod 2B < Mod 1
VX	Sealant	Extracted Coupon	Bleach	513	Mod 1	A		0.0004	0.0124	
VX	Sealant	Extracted Coupon	Bleach + Chem A	247	Mod 2	C			0.4255	
VX	Sealant	Extracted Coupon	Bleach + Chem B	327	Mod 2	BC				

Table D26. Multiple comparison adjusted p-values between test methods for bleach (VX in SPE Disks)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Bleach			Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2 (Chem A)	Mod 2 (Chem B)	
VX	Sealant	SPE	Bleach	9.92	Baseline	A	1.0000	1.0000	1.0000	No significant differences
VX	Sealant	SPE	Bleach	<MQL	Mod 1	A		1.0000	1.0000	
VX	Sealant	SPE	Bleach + Chem A	2.15	Mod 2	A			1.0000	
VX	Sealant	SPE	Bleach + Chem B	0.041	Mod 2	A				

Table D27. Multiple comparison adjusted p-values between test methods for Dahlgren Decon (HD on Paint Wipes)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Dahlgren Decon + Chem A	Summary of Significant Bonferroni-Holm Differences
							Mod 3	
HD	Paint	Wipe	Dahlgren Decon	<MQL	Baseline	A	<0.0001	Baseline < Mod 3
HD	Paint	Wipe	Dahlgren Decon + Chem A	14.2	Mod 3	B		

Table D28. Multiple comparison adjusted p-values between test methods for Dahlgren Decon (HD in Paint Coupons)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Dahlgren Decon + Chem A	Summary of Significant Bonferroni-Holm Differences
							Mod 3	
HD	Paint	Extracted Coupon	Dahlgren Decon	308	Baseline	A	0.0384	Mod 3 < Baseline
HD	Paint	Extracted Coupon	Dahlgren Decon + Chem A	132	Mod 3	B		

Table D29. Multiple comparison adjusted p-values between test methods for Dahlgren Decon (HD in SPE Disks)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Dahlgren Decon + Chem A	Summary of Significant Bonferroni-Holm Differences
							Mod 3	
HD	Paint	SPE	Dahlgren Decon	1010	Baseline	A	1.0000	No significant differences.
HD	Paint	SPE	Dahlgren Decon + Chem A	1060	Mod 3	A		

Table D30. Multiple comparison adjusted p-values between test methods for Dahlgren Decon (HD on Sealant Coupon Surfaces)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Dahlgren Decon + Chem A	Summary of Significant Bonferroni-Holm Differences
							Mod 3	
HD	Sealant	Wipe	Dahlgren Decon	<MQL	Baseline	A	<0.0001	Baseline < Mod 3
HD	Sealant	Wipe	Dahlgren Decon + Chem A	10.5	Mod 3	B		

Table D31. Multiple comparison adjusted p-values between test methods for Dahlgren Decon (HD in Sealant Coupons)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Dahlgren Decon + Chem A	Summary of Significant Bonferroni-Holm Differences
							Mod 3	
HD	Sealant	Extracted Coupon	Dahlgren Decon	209	Baseline	A	0.3549	No significant differences.
HD	Sealant	Extracted Coupon	Dahlgren Decon + Chem A	155	Mod 3	A		

Table D32. Multiple comparison adjusted p-values between test methods for Dahlgren Decon (HD in SPE Disks)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Dahlgren Decon + Chem A	Summary of Significant Bonferroni-Holm Differences
							Mod 3	
HD	Sealant	SPE	Dahlgren Decon	1130	Baseline	A	1.0000	No significant differences.
HD	Sealant	SPE	Dahlgren Decon + Chem A	1140	Mod 3	A		

Table D33. Multiple comparison adjusted p-values between testing methods for Dahlgren Decon (VX on Paint Coupon Surfaces)

Agent	Material	Sample Type	Decontaminant	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Dahlgren Decon		Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 3 (Chem A)	
VX	Paint	Wipe	Dahlgren Decon	46.4	Baseline	A	0.4472	<0.0001	Baseline < Mod 3 Mod 1 < Mod 3
VX	Paint	Wipe	Dahlgren Decon	18.1	Mod 1	A		<0.0001	
VX	Paint	Wipe	Dahlgren Decon + Chem A	250	Mod 3	B			

Table D34. Multiple comparison adjusted p-values between testing methods for Dahlgren Decon (VX in Paint Coupons)

Agent	Material	Sample Type	Decontaminant	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Dahlgren Decon		Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 3 (Chem A)	
VX	Paint	Extracted Coupon	Dahlgren Decon	230	Baseline	A	1.0000	0.0530	No significant differences.
VX	Paint	Extracted Coupon	Dahlgren Decon	201	Mod 1	A		0.1767	
VX	Paint	Extracted Coupon	Dahlgren Decon + Chem A	80.0	Mod 3	A			

Table D35. Multiple comparison adjusted p-values between testing methods for Dahlgren Decon (VX in SPE Disks)

Agent	Material	Sample Type	Decontaminant	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Dahlgren Decon		Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 3 (Chem A)	
VX	Paint	SPE	Dahlgren Decon	317	Baseline	A	1.0000	0.4837	No significant differences.
VX	Paint	SPE	Dahlgren Decon	273	Mod 1	A		1.0000	
VX	Paint	SPE	Dahlgren Decon + Chem A	233	Mod 3	A			

Table D36. Multiple comparison adjusted p-values between testing methods for Dahlgren Decon (VX on Sealant Coupon Surfaces)

Agent	Material	Sample Type	Decontaminant	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Dahlgren Decon		Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 3 (Chem A)	
VX	Sealant	Wipe	Dahlgren Decon	0.97	Baseline	A	1.0000	<0.0001	Baseline < Mod 3 Mod 1 < Mod 3
VX	Sealant	Wipe	Dahlgren Decon	0.37	Mod 1	A		<0.0001	
VX	Sealant	Wipe	Dahlgren + Chem A	524	Mod 3	B			

Table D37. Multiple comparison adjusted p-values between testing methods for Dahlgren Decon (VX in Sealant Coupons)

Agent	Material	Sample Type	Decontaminant	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Dahlgren Decon		Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 3 (Chem A)	
VX	Sealant	Extracted Coupon	Dahlgren Decon	730	Baseline	A	0.6720	<0.0001	Mod 3 < Base Mod 3 < Mod 1
VX	Sealant	Extracted Coupon	Dahlgren Decon	683	Mod 1	A		<0.0001	
VX	Sealant	Extracted Coupon	Dahlgren + Chem A	251	Mod 3	B			

Table D38. Multiple comparison adjusted p-values between testing methods for Dahlgren Decon (VX in SPE Disks)

Agent	Material	Sample Type	Decontaminant	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Dahlgren Decon		Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 3 (Chem A)	
VX	Sealant	SPE	Dahlgren Decon	23.3	Baseline	A	1.0000	1.0000	No significant differences.
VX	Sealant	SPE	Dahlgren Decon	5.65	Mod 1	A		1.0000	
VX	Sealant	SPE	Dahlgren Decon + Chem A	3.28	Mod 3	A			

Table D39. Multiple comparison adjusted p-values between testing methods for D7 (HD on Paint Wipes)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	D7	Summary of Significant Bonferroni-Holm Differences
							Mod 1	
HD	Paint	Wipe	D7	2.18	Baseline	A	0.4851	No significant differences.
HD	Paint	Wipe	D7	<MQL	Mod 1	A		

Table D40. Multiple comparison adjusted p-values between testing methods for D7 (HD in Paint Coupons)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	D7	Summary of Significant Bonferroni-Holm Differences
							Mod 1	
HD	Paint	Extracted Coupon	D7	235	Baseline	A	0.0922	No significant differences.
HD	Paint	Extracted Coupon	D7	82.4	Mod 1	A		

Table D41. Multiple comparison adjusted p-values between testing methods for D7 (HD in SPE Disks)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	D7	Summary of Significant Bonferroni-Holm Differences
							Mod 1	
HD	Paint	SPE	D7	1030	Baseline	A	1.0000	No significant differences.
HD	Paint	SPE	D7	1120	Mod 1	A		

Table D42. Multiple comparison adjusted p-values between testing methods for D7 (HD on Sealant Wipes)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	D7	Summary of Significant Bonferroni-Holm Differences
							Mod 1	
HD	Sealant	Wipe	D7	2.96	Baseline	A	0.1696	No significant differences.
HD	Sealant	Wipe	D7	3.78	Mod 1	A		

Table D43. Multiple comparison adjusted p-values between testing methods for D7 (HD in Sealant Coupons)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	D7	Summary of Significant Bonferroni-Holm Differences
							Mod 1	
HD	Sealant	Extracted Coupon	D7	266	Baseline	A	0.0486	Mod 1 < Base
HD	Sealant	Extracted Coupon	D7	176	Mod 1	B		

Table D44. Multiple comparison adjusted p-values between testing methods for D7 (HD in SPE Disks)

Agent	Material	Sample Type	Decontaminants	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	D7	Summary of Significant Bonferroni-Holm Differences
							Mod 1	
HD	Sealant	SPE	D7	1050	Baseline	A	1.0000	No significant differences.
HD	Sealant	SPE	D7	1210	Mod 1	A		

Table D45. Multiple comparison adjusted p-values between decontaminants for Baseline method (HD on Paint Coupon Surfaces)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline		Summary of Significant Bonferroni-Holm Differences
							D7	Dahlgren	
HD	Paint	Wipe	Baseline	<MQL	Bleach	A	0.4851	1.0000	No significant differences.
HD	Paint	Wipe	Baseline	2.18	D7	A		0.4851	
HD	Paint	Wipe	Baseline	<MQL	Dahlgren	A			

Table D46. Multiple comparison adjusted p-values between decontaminants for Baseline method (HD in Paint Coupons)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline		Summary of Significant Bonferroni-Holm Differences
							D7	Dahlgren	
HD	Paint	Extracted Coupon	Baseline	109	Bleach	A	0.2468	0.0153	Bleach < Dahlgren Decon
HD	Paint	Extracted Coupon	Baseline	235	D7	AB		1.0000	
HD	Paint	Extracted Coupon	Baseline	308	Dahlgren	B			

Table D47. Multiple comparison adjusted p-values between decontaminants for Baseline method (HD in SPE Disks)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline		Summary of Significant Bonferroni-Holm Differences
							D7	Dahlgren	
HD	Paint	SPE	Baseline	1030	Bleach	A	1.0000	1.0000	No significant differences.
HD	Paint	SPE	Baseline	1030	D7	A		1.0000	
HD	Paint	SPE	Baseline	1010	Dahlgren Decon	A			

Table D48. Multiple comparison adjusted p-values between decontaminants for Baseline method (HD on Sealant Coupon Surfaces)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline		Summary of Significant Bonferroni-Holm Differences
							D7	Dahlgren	
HD	Sealant	Wipe	Baseline	<MQL	Bleach	A	0.0016	1.0000	Bleach < D7 Dahlgren Decon < D7
HD	Sealant	Wipe	Baseline	2.96	D7	B		0.0016	
HD	Sealant	Wipe	Baseline	<MQL	Dahlgren	A			

Table D49. Multiple comparison adjusted p-values between decontaminants for Baseline method (HD in Sealant Coupons)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline		Summary of Significant Bonferroni-Holm Differences
							D7	Dahlgren	
HD	Sealant	Extracted Coupon	Baseline	188	Bleach	A	0.1009	0.8529	No significant differences.
HD	Sealant	Extracted Coupon	Baseline	266	D7	A		0.3549	
HD	Sealant	Extracted Coupon	Baseline	209	Dahlgren Decon	A			

Table D50. Multiple comparison adjusted p-values between decontaminants for Baseline method (HD in SPE Disks)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline		Summary of Significant Bonferroni-Holm Differences
							D7	Dahlgren	
HD	Sealant	SPE	Baseline	1140	Bleach	A	1.0000	1.0000	No significant differences.
HD	Sealant	SPE	Baseline	1050	D7	A		1.0000	
HD	Sealant	SPE	Baseline	1130	Dahlgren Decon	A			

Table D51. Multiple comparison adjusted p-values between decontaminants for Baseline method (VX on Paint Coupon Surfaces)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline		Summary of Significant Bonferroni-Holm Differences
							D7	Dahlgren	
VX	Paint	Wipe	Baseline	141	Bleach	A	0.0086	0.0007	D7 < Bleach Dahlgren Decon < Bleach
VX	Paint	Wipe	Baseline	70.3	D7	B		0.4472	
VX	Paint	Wipe	Baseline	46.4	Dahlgren Decon	B			

Table D52. Multiple comparison adjusted p-values between decontaminants for Baseline method (VX in Paint Coupons)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline		Summary of Significant Bonferroni-Holm Differences
							D7	Dahlgren Decon	
VX	Paint	Extracted Coupon	Baseline	229	Bleach	A	1.0000	1.0000	No significant differences.
VX	Paint	Extracted Coupon	Baseline	187	D7	A		1.0000	
VX	Paint	Extracted Coupon	Baseline	230	Dahlgren	A			

Table D53. Multiple comparison adjusted p-values between decontaminants for Baseline method (VX in SPE Disks)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline		Summary of Significant Bonferroni-Holm Differences
							D7	Dahlgren Decon	
VX	Paint	SPE	Baseline	148	Bleach	A	0.0036	0.0053	Bleach < D7 Bleach < Dahlgren Decon
VX	Paint	SPE	Baseline	326	D7	B		1.0000	
VX	Paint	SPE	Baseline	317	Dahlgren Decon	B			

Table D54. Multiple comparison adjusted p-values between decontaminants for Baseline method (VX on Sealant Wipes)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline		Summary of Significant Bonferroni-Holm Differences
							D7	Dahlgren Decon	
VX	Sealant	Wipe	Baseline	1.14	Bleach	A	1.0000	1.0000	No significant differences.
VX	Sealant	Wipe	Baseline	42.1	D7	A		1.0000	
VX	Sealant	Wipe	Baseline	0.97	Dahlgren	A			

Table D55. Multiple comparison adjusted p-values between decontaminants for Baseline method (VX in Sealant Coupons)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline		Summary of Significant Bonferroni-Holm Differences
							D7	Dahlgren Decon	
VX	Sealant	Extracted Coupon	Baseline	417	Bleach	A	0.0324	<0.0001	Bleach < D7 Bleach < Dahlgren Decon D7 < Dahlgren Decon
VX	Sealant	Extracted Coupon	Baseline	572	D7	B		0.0324	
VX	Sealant	Extracted Coupon	Baseline	730	Dahlgren Decon	C			

Table D56. Multiple comparison adjusted p-values between decontaminants for Baseline method (VX in SPE Disks)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline		Summary of Significant Bonferroni-Holm Differences
							D7	Dahlgren Decon	
VX	Sealant	SPE	Baseline	9.92	Bleach	A	1.0000	1.0000	No significant differences.
VX	Sealant	SPE	Baseline	15.3	D7	A		1.0000	
VX	Sealant	SPE	Baseline	23.3	Dahlgren Decon	A			

Table D57. Multiple comparison adjusted p-values between decontaminants for Mod 1 method (HD on Paint Wipes)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 1	Summary of Significant Bonferroni-Holm Differences
							D7	
HD	Paint	Wipe	Mod 1	<MQL	Bleach	A	1.0000	No significant differences.
HD	Paint	Wipe	Mod 1	<MQL	D7	A		

Table D58. Multiple comparison adjusted p-values between decontaminants for Mod 1 method (HD in Paint Coupons)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 1	Summary of Significant Bonferroni-Holm Differences
							D7	
HD	Paint	Extracted Coupon	Mod 1	46.6	Bleach	A	1.0000	No significant differences.
HD	Paint	Extracted Coupon	Mod 1	82.4	D7	A		

Table D59. Multiple comparison adjusted p-values between decontaminants for Mod 1 method (HD in SPE Disks)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 1	Summary of Significant Bonferroni-Holm Differences
							D7	
HD	Paint	SPE	Mod 1	1170	Bleach	A	1.0000	No significant differences.
HD	Paint	SPE	Mod 1	1120	D7	A		

Table D60. Multiple comparison adjusted p-values between decontaminants for Mod 1 method (HD on Sealant Surfaces)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 1	Summary of Significant Bonferroni-Holm Differences
							D7	
HD	Sealant	Wipe	Mod 1	<MQL	Bleach	A	<0.0001	Bleach < D7
HD	Sealant	Wipe	Mod 1	3.78	D7	B		

Table D61. Multiple comparison adjusted p-values between decontaminants for Mod 1 method (HD in Sealant Coupons)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 1	Summary of Significant Bonferroni-Holm Differences
							D7	
HD	Sealant	Extracted Coupon	Mod 1	134	Bleach	A	0.3835	No significant differences.
HD	Sealant	Extracted Coupon	Mod 1	176	D7	A		

Table D62. Multiple comparison adjusted p-values between decontaminants for Mod 1 method (HD in SPE Disks)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 1	Summary of Significant Bonferroni-Holm Differences
							D7	
HD	Sealant	SPE	Mod 1	1310	Bleach	A	1.0000	No significant differences.
HD	Sealant	SPE	Mod 1	1210	D7	A		

Table D63. Multiple comparison adjusted p-values between decontaminants for Mod 1 method (VX on Paint Coupon Surfaces)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 1	Summary of Significant Bonferroni-Holm Differences
							D7	
VX	Paint	Wipe	Mod 1	14.7	Bleach	A	0.8555	No significant differences.
VX	Paint	Wipe	Mod 1	18.1	Dahlgren	A		

Table D64. Multiple comparison adjusted p-values between decontaminants for Mod 1 method (VX in Paint Coupons)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 1	Summary of Significant Bonferroni-Holm Differences
							D7	
VX	Paint	Extracted Coupon	Mod 1	157	Bleach	A	1.0000	No significant differences.
VX	Paint	Extracted Coupon	Mod 1	201	Dahlgren	A		

Table D65. Multiple comparison adjusted p-values between decontaminants for Mod 1 method (VX in SPE Disks)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 1	Summary of Significant Bonferroni-Holm Differences
							D7	
VX	Paint	SPE	Mod 1	210	Bleach	A	1.0000	No significant differences.
VX	Paint	SPE	Mod 1	273	Dahlgren	A		

Table D66. Multiple comparison adjusted p-values between decontaminants for Mod 1 method (VX on Sealant Wipes)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 1	Summary of Significant Bonferroni-Holm Differences
							D7	
VX	Sealant	Wipe	Mod 1	0.0049	Bleach	A	1.0000	No significant differences.
VX	Sealant	Wipe	Mod 1	0.37	Dahlgren Decon	A		

Table D67. Multiple comparison adjusted p-values between decontaminants for Mod 1 method (VX in Sealant Coupons)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 1	Summary of Significant Bonferroni-Holm Differences
							D7	
VX	Sealant	Extracted Coupon	Mod 1	513	Bleach	A	0.0226	Bleach < Dahlgren
VX	Sealant	Extracted Coupon	Mod 1	683	Dahlgren Decon	B		

Table D68. Multiple comparison adjusted p-values between decontaminants for Mod 1 method (VX in SPE Disks)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 1	Summary of Significant Bonferroni-Holm Differences
							D7	
VX	Sealant	SPE	Mod 1	<MQL	Bleach	A	1.0000	No significant differences.
VX	Sealant	SPE	Mod 1	5.65	Dahlgren Decon	A		

Table D69. Multiple comparison adjusted p-values between decontaminants for Mod 2 method (HD on Paint Coupon Surfaces)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 2	Summary of Significant Bonferroni-Holm Differences
							Bleach + Chem B	
HD	Paint	Wipe	Mod 2	15.3	Bleach + Chem A	A	0.0298	Mod 2A < Mod 2B
HD	Paint	Wipe	Mod 2	17.1	Bleach + Chem B	B		

Table D70. Multiple comparison adjusted p-values between decontaminants for Mod 2 method (HD in Paint Coupons)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 2	Summary of Significant Bonferroni-Holm Differences
							Bleach + Chem B	
HD	Paint	Extracted Coupon	Mod 2	73.7	Bleach + Chem A	A	1.0000	No significant differences.
HD	Paint	Extracted Coupon	Mod 2	88.0	Bleach + Chem B	A		

Table D71. Multiple comparison adjusted p-values between decontaminants for Mod 2 method (HD in SPE Disks)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 2	Summary of Significant Bonferroni-Holm Differences
							Bleach + Chem B	
HD	Paint	SPE	Mod 2	1000	Bleach + Chem A	A	1.0000	No significant differences.
HD	Paint	SPE	Mod 2	998	Bleach + Chem B	A		

Table D72. Multiple comparison adjusted p-values between decontaminants for Mod 2 method (HD on Sealant Surfaces)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 2 Bleach + Chem B	Summary of Significant Bonferroni-Holm Differences
HD	Sealant	Wipe	Mod 2	9.15	Bleach + Chem A	A	<0.0001	Mod 2A < Mod 2B
HD	Sealant	Wipe	Mod 2	12.6	Bleach + Chem B	B		

Table D73. Multiple comparison adjusted p-values between decontaminants for Mod 2 method (HD in Sealant Coupons)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 2 Bleach + Chem B	Summary of Significant Bonferroni-Holm Differences
HD	Sealant	Extracted Coupon	Mod 2	68.2	Bleach + Chem A	A	0.3549	No significant differences.
HD	Sealant	Extracted Coupon	Mod 2	126	Bleach + Chem B	A		

Table D74. Multiple comparison adjusted p-values between decontaminants for Mod 2 method (HD in SPE Disks)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 2 Bleach + Chem B		Summary of Significant Bonferroni-Holm Differences
HD	Sealant	SPE	Mod 2	1130	Bleach + Chem A	A	1.0000		No significant differences.
HD	Sealant	SPE	Mod 2	1070	Bleach + Chem B	A			

Table D75. Multiple comparison adjusted p-values between decontaminants for Mod 2 method (VX on Paint Coupon Surfaces)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 2	Summary of Significant Bonferroni-Holm Differences
							Bleach + Chem B	
VX	Paint	Wipe	Mod 2	353	Bleach + Chem A	A	<0.0001	Mod 2B < Mod 2A
VX	Paint	Wipe	Mod 2	175	Bleach + Chem B	B		

Table D76. Multiple comparison adjusted p-values between decontaminants for Mod 2 method (VX in Paint Coupons)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 2	Summary of Significant Bonferroni-Holm Differences
							Bleach + Chem B	
VX	Paint	Extracted Coupon	Mod 2	121	Bleach + Chem A	A	1.0000	No significant differences.
VX	Paint	Extracted Coupon	Mod 2	83.8	Bleach + Chem B	A		

Table D77. Multiple comparison adjusted p-values between decontaminants for Mod 2 method (VX in SPE Disks)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 2	Summary of Significant Bonferroni-Holm Differences
							Bleach + Chem B	
VX	Paint	SPE	Mod 2	253	Bleach + Chem A	A	1.0000	No significant differences.
VX	Paint	SPE	Mod 2	209	Bleach + Chem B	A		

Table D78. Multiple comparison adjusted p-values between decontaminants for Mod 2 method (VX on Sealant Coupon Surfaces)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 2	Summary of Significant Bonferroni-Holm Differences
							Bleach + Chem B	
VX	Sealant	Wipe	Mod 2	364	Bleach + Chem A	A	0.4798	No significant differences.
VX	Sealant	Wipe	Mod 2	294	Bleach + Chem B	A		

Table D79. Multiple comparison adjusted p-values between decontaminants for Mod 2 method (VX in Sealant Coupons)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 2	Summary of Significant Bonferroni-Holm Differences
							Bleach + Chem B	
VX	Sealant	Extracted Coupon	Mod 2	247	Bleach + Chem A	A	0.4255	No significant differences.
VX	Sealant	Extracted Coupon	Mod 2	327	Bleach + Chem B	A		

Table D80. Multiple comparison adjusted p-values between decontaminants for Mod 2 method (VX in SPE Disks)

Agent	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Mod 2	Summary of Significant Bonferroni-Holm Differences
							Bleach + Chem B	
VX	Sealant	SPE	Mod 2	2.15	Bleach + Chem A	A	1.0000	No significant differences.
VX	Sealant	SPE	Mod 2	0.041	Bleach + Chem B	A		

Table D81. Multiple comparison adjusted p-values between decontaminants for Chem A (HD on Paint Coupon Surfaces)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Similarity Designation	Mod 3	Summary of Significant Bonferroni-Holm Differences
							Dahlgren Decon + Chem A	
HD	Paint	Wipe	15.3	Mod 2	Bleach + Chem A	A	0.4851	No significant differences.
HD	Paint	Wipe	14.2	Mod 3	Dahlgren + Chem A	A		

Table D82. Multiple comparison adjusted p-values between decontaminants for Chem A (HD in Paint Coupons)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Similarity Designation	Mod 3	Summary of Significant Bonferroni-Holm Differences
							Dahlgren Decon + Chem A	
HD	Paint	Extracted Coupon	73.8	Mod 2	Bleach + Chem A	A	1.0000	No significant differences.
HD	Paint	Extracted Coupon	132	Mod 3	Dahlgren Decon + Chem A	A		

Table D83. Multiple comparison adjusted p-values between decontaminants for Chem A (HD in SPE Disks)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Similarity Designation	Mod 3	Summary of Significant Bonferroni-Holm Differences
							Dahlgren Decon + Chem A	
HD	Paint	SPE	1000	Mod 2	Bleach + Chem A	A	1.0000	No significant differences.
HD	Paint	SPE	1060	Mod 3	Dahlgren Decon + Chem A	A		

Table D84. Multiple comparison adjusted p-values between decontaminants for Chem A (HD on Sealant Coupon Surfaces)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Similarity Designation	Mod 3 Dahlgren Decon + Chem A	Summary of Significant Bonferroni-Holm Differences
HD	Sealant	Wipe	9.15	Mod 2	Bleach + Chem A	A	0.0182	Bleach < Dahlgren Decon
HD	Sealant	Wipe	10.5	Mod 3	Dahlgren Decon + Chem A	B		

Table D85. Multiple comparison adjusted p-values between decontaminants for Chem A (HD in Sealant Coupons)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Similarity Designation	Mod 3 Dahlgren Decon + Chem A	Summary of Significant Bonferroni-Holm Differences
HD	Sealant	Extracted Coupon	68.2	Mod 2	Bleach + Chem A	A	0.0565	No significant differences.
HD	Sealant	Extracted Coupon	155	Mod 3	Dahlgren Decon + Chem A	A		

Table D86. Multiple comparison adjusted p-values between decontaminants for Chem A (HD in SPE Disks)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Similarity Designation	Mod 3 Dahlgren Decon + Chem A	Summary of Significant Bonferroni-Holm Differences
HD	Sealant	SPE	1130	Mod 2	Bleach + Chem A	A	1.0000	No significant differences.
HD	Sealant	SPE	1140	Mod 3	Dahlgren Decon + Chem A	A		

Table D87. Multiple comparison adjusted p-values between decontaminants for Chem A (VX on Paint Coupon Surfaces)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Similarity Designation	Mod 3 Dahlgren Decon + Chem A	Summary of Significant Bonferroni-Holm Differences
VX	Paint	Wipe	353	Mod 2	Bleach + Chem A	A	0.0003	Dahlgren Decon < Bleach
VX	Paint	Wipe	250	Mod 3	Dahlgren Decon + Chem A	B		

Table D88. Multiple comparison adjusted p-values between decontaminants for Chem A (VX in Paint Coupons)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Similarity Designation	Mod 3 Dahlgren Decon + Chem A	Summary of Significant Bonferroni-Holm Differences
VX	Paint	Extracted Coupon	121	Mod 2	Bleach + Chem A	A	1.0000	No significant differences.
VX	Paint	Extracted Coupon	80.0	Mod 3	Dahlgren Decon + Chem A	A		

Table D89. Multiple comparison adjusted p-values between decontaminants for Chem A (VX in SPE Disks)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Similarity Designation	Mod 3 Dahlgren Decon + Chem A	Summary of Significant Bonferroni-Holm Differences
VX	Paint	SPE	253	Mod 2	Bleach + Chem A	A	1.0000	No significant differences.
VX	Paint	SPE	233	Mod 3	Dahlgren Decon + Chem A	A		

Table D90. Multiple comparison adjusted p-values between decontaminants for Chem A (VX on Sealant Coupon Surfaces)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Similarity Designation	Mod 3	Summary of Significant Bonferroni-Holm Differences
							Dahlgren + Chem A	
VX	Sealant	Wipe	364	Mod 2	Bleach + Chem A	A	0.0027	Bleach < Dahlgren Decon
VX	Sealant	Wipe	524	Mod 3	Dahlgren Decon + Chem A	B		

Table D91. Multiple comparison adjusted p-values between decontaminants for Chem A (VX in Sealant Coupons)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Similarity Designation	Mod 3	Summary of Significant Bonferroni-Holm Differences
							Dahlgren Decon + Chem A	
VX	Sealant	Extracted Coupon	247	Mod 2	Bleach + Chem A	A	0.9311	No significant differences.
VX	Sealant	Extracted Coupon	251	Mod 3	Dahlgren Decon + Chem A	A		

Table D92. Multiple comparison adjusted p-values between decontaminants for Chem A (VX in SPE Disks)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Similarity Designation	Mod 3	Summary of Significant Bonferroni-Holm Differences
							Dahlgren Decon + Chem A	
VX	Sealant	SPE	2.15	Mod 2	Bleach + Chem A	A	1.0000	No significant differences.
VX	Sealant	SPE	3.28	Mod 3	Dahlgren Decon + Chem A	A		

Positive Control Results: Within-Test Comparisons

Table D93. Multiple comparison adjusted p-values for between decontaminants and positive controls with testing methods (HD on Paint Coupon Surfaces)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
HD	Paint	Wipe	<MQL	Baseline	Bleach	0.0091	Bleach < PC
HD	Paint	Wipe	2.18	Baseline	D7	0.0111	D7 < PC
HD	Paint	Wipe	<MQL	Baseline	Dahlgren Decon	0.0091	Dahlgren Decon < PC
HD	Paint	Wipe	6.09	Baseline	Positive Control		
HD	Paint	Wipe	<MQL	Mod 1	Bleach	0.0001	Bleach < PC
HD	Paint	Wipe	<MQL	Mod 1	D7	0.0001	D7 < PC
HD	Paint	Wipe	10.0	Mod 1	Positive Control		
HD	Paint	Wipe	15.2	Mod 2 + Chem A	Bleach + Chem A	0.3878	No significant differences.
HD	Paint	Wipe	20.1	Mod 2 + Chem A	Positive Control		
HD	Paint	Wipe	17.1	Mod 2 + Chem B	Bleach + Chem B	0.2535	No significant differences.
HD	Paint	Wipe	20.4	Mod 2 + Chem B	Positive Control		
HD	Paint	Wipe	14.2	Mod 3 + Chem A	Dahlgren Decon + Chem A	0.0593	No significant differences.
HD	Paint	Wipe	21.6	Mod 3 + Chem A	Positive Control		

Table D94. Multiple comparison adjusted p-values for between decontaminants and positive controls with testing methods (HD in Paint Coupons)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
HD	Paint	Extracted Coupon	109	Baseline	Bleach	0.0775	No significant differences.
HD	Paint	Extracted Coupon	235	Baseline	D7	0.5798	No significant differences.
HD	Paint	Extracted Coupon	308	Baseline	Dahlgren Decon	0.8316	No significant differences.
HD	Paint	Extracted Coupon	325	Baseline	Positive Control		
HD	Paint	Extracted Coupon	46.6	Mod 1	Bleach	<0.0001	Bleach < PC
HD	Paint	Extracted Coupon	82.4	Mod 1	D7	<0.0001	Mod 1 < PC
HD	Paint	Extracted Coupon	467	Mod 1	Positive Control		
HD	Paint	Extracted Coupon	73.7	Mod 2 + Chem A	Bleach + Chem A	0.0310	Mod 2A < PC
HD	Paint	Extracted Coupon	308	Mod 2 + Chem A	Positive Control		
HD	Paint	Extracted Coupon	88.0	Mod 2 + Chem B	Bleach + Chem B	0.0068	Mod 2B < PC
HD	Paint	Extracted Coupon	383	Mod 2 + Chem B	Positive Control		
HD	Paint	Extracted Coupon	132	Mod 3 + Chem A	Dahlgren + Chem A	0.0271	Mod 3A < PC
HD	Paint	Extracted Coupon	366	Mod 3 + Chem A	Positive Control		

Table D95. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (HD in SPE Disks)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
HD	Paint	SPE	1030	Baseline	Bleach	0.6495	No significant differences.
HD	Paint	SPE	1030	Baseline	D7	0.6495	No significant differences.
HD	Paint	SPE	1010	Baseline	Dahlgren Decon	0.6495	No significant differences.
HD	Paint	SPE	881	Baseline	Positive Control		
HD	Paint	SPE	1170	Mod 1	Bleach	1.0000	No significant differences.
HD	Paint	SPE	1120	Mod 1	D7	1.0000	No significant differences.
HD	Paint	SPE	1140	Mod 1	Positive Control		
HD	Paint	SPE	1000	Mod 2 + Chem A	Bleach + Chem A	0.1816	No significant differences.
HD	Paint	SPE	1190	Mod 2 + Chem A	Positive Control		
HD	Paint	SPE	997	Mod 2 + Chem B	Bleach + Chem B	0.2772	No significant differences.
HD	Paint	SPE	1090	Mod 2 + Chem B	Positive Control		
HD	Paint	SPE	1060	Mod 3 + Chem A	Dahlgren + Chem A	0.5459	No significant differences.
HD	Paint	SPE	1140	Mod 3 + Chem A	Positive Control		

Table D96. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (HD on Sealant Coupon Surfaces)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
HD	Sealant	Wipe	<MQL	Baseline	Bleach	1.0000	No significant differences.
HD	Sealant	Wipe	2.96	Baseline	D7	0.0001	PC < D7
HD	Sealant	Wipe	<MQL	Baseline	Dahlgren Decon	1.0000	No significant differences.
HD	Sealant	Wipe	<MQL	Baseline	Positive Control		
HD	Sealant	Wipe	<MQL	Mod 1	Bleach	1.0000	No significant differences.
HD	Sealant	Wipe	3.78	Mod 1	D7	<0.0001	PC < D7
HD	Sealant	Wipe	<MQL	Mod 1	Positive Control		
HD	Sealant	Wipe	9.15	Mod 2 + Chem A	Bleach + Chem A	0.0750	No significant differences.
HD	Sealant	Wipe	10.5	Mod 2 + Chem A	Positive Control		
HD	Sealant	Wipe	12.6	Mod 2 + Chem B	Bleach + Chem B	0.0808	No significant differences.
HD	Sealant	Wipe	14.5	Mod 2 + Chem B	Positive Control		
HD	Sealant	Wipe	10.5	Mod 3 + Chem A	Dahlgren + Chem A	0.3945	No significant differences.
HD	Sealant	Wipe	9.33	Mod 3 + Chem A	Positive Control		

Table D97. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (HD in Sealant Coupons)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
HD	Sealant	Extracted Coupon	1878	Baseline	Bleach	1.0000	No significant differences.
HD	Sealant	Extracted Coupon	266	Baseline	D7	0.2799	No significant differences.
HD	Sealant	Extracted Coupon	209	Baseline	Dahlgren Decon	1.0000	No significant differences.
HD	Sealant	Extracted Coupon	188	Baseline	Positive Control		
HD	Sealant	Extracted Coupon	134	Mod 1	Bleach	0.0080	Bleach < PC
HD	Sealant	Extracted Coupon	176	Mod 1	D7	0.0285	D7 < PC
HD	Sealant	Extracted Coupon	250	Mod 1	Positive Control		
HD	Sealant	Extracted Coupon	68.2	Mod 2 + Chem A	Bleach + Chem A	0.0136	Bleach + A < PC
HD	Sealant	Extracted Coupon	207	Mod 2 + Chem A	Positive Control		
HD	Sealant	Extracted Coupon	126	Mod 2 + Chem B	Bleach + Chem B	0.0309	Bleach + B < PC
HD	Sealant	Extracted Coupon	201	Mod 2 + Chem B	Positive Control		
HD	Sealant	Extracted Coupon	155	Mod 3 + Chem A	Dahlgren + Chem A	0.2121	No significant differences.
HD	Sealant	Extracted Coupon	203	Mod 3 + Chem A	Positive Control		

Table D98. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (HD in SPE Disks)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
HD	Sealant	SPE	1140	Baseline	Bleach	1.0000	No significant differences.
HD	Sealant	SPE	1050	Baseline	D7	1.0000	No significant differences.
HD	Sealant	SPE	1130	Baseline	Dahlgren Decon	1.0000	No significant differences.
HD	Sealant	SPE	1070	Baseline	Positive Control		
HD	Sealant	SPE	1310	Mod 1	Bleach	0.1714	No significant differences.
HD	Sealant	SPE	1210	Mod 1	D7	0.3073	No significant differences.
HD	Sealant	SPE	1100	Mod 1	Positive Control		
HD	Sealant	SPE	1130	Mod 2 + Chem A	Bleach + Chem A	0.9509	No significant differences.
HD	Sealant	SPE	1130	Mod 2 + Chem A	Positive Control		
HD	Sealant	SPE	1070	Mod 2 + Chem B	Bleach + Chem B	0.5071	No significant differences.
HD	Sealant	SPE	1040	Mod 2 + Chem B	Positive Control		
HD	Sealant	SPE	1140	Mod 3 + Chem A	Dahlgren + Chem A	0.5483	No significant differences.
HD	Sealant	SPE	1090	Mod 3 + Chem A	Positive Control		

Table D99. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (VX on Paint Coupon Surfaces)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
VX	Paint	Wipe	141	Baseline	Bleach	0.2001	No significant differences.
VX	Paint	Wipe	70.4	Baseline	D7	0.3700	No significant differences.
VX	Paint	Wipe	46.4	Baseline	Dahlgren Decon	0.2001	No significant differences.
VX	Paint	Wipe	92.1	Baseline	Positive Control		
VX	Paint	Wipe	14.7	Mod 1	Bleach	0.0002	Bleach < PC
VX	Paint	Wipe	18.1	Mod 1	Dahlgren Decon	0.0002	Dahlgren < PC
VX	Paint	Wipe	87.9	Mod 1	Positive Control		
VX	Paint	Wipe	353	Mod 2 + Chem A	Bleach + Chem A	0.9140	No significant differences.
VX	Paint	Wipe	357	Mod 2 + Chem A	Positive Control		
VX	Paint	Wipe	175	Mod 2 + Chem B	Bleach + Chem B	0.2322	No significant differences.
VX	Paint	Wipe	148	Mod 2 + Chem B	Positive Control		
VX	Paint	Wipe	250	Mod 3 + Chem A	Dahlgren Decon + Chem A	0.4855	No significant differences.
VX	Paint	Wipe	270	Mod 3 + Chem A	Positive Control		

Table D100. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (VX in Paint Coupons)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
VX	Paint	Extracted Coupon	230	Baseline	Bleach	0.2725	No significant differences.
VX	Paint	Extracted Coupon	187	Baseline	D7	0.0772	No significant differences.
VX	Paint	Extracted Coupon	230	Baseline	Dahlgren Decon	0.2725	No significant differences.
VX	Paint	Extracted Coupon	295	Baseline	Positive Control		
VX	Paint	Extracted Coupon	157	Mod 1	Bleach	0.0387	Bleach < PC
VX	Paint	Extracted Coupon	201	Mod 1	Dahlgren Decon	0.1299	No significant differences.
VX	Paint	Extracted Coupon	256	Mod 1	Positive Control		
VX	Paint	Extracted Coupon	121	Mod 2 + Chem A	Bleach + Chem A	0.0631	No significant differences.
VX	Paint	Extracted Coupon	274	Mod 2 + Chem A	Positive Control		
VX	Paint	Extracted Coupon	83.8	Mod 2 + Chem B	Bleach + Chem B	0.0593	No significant differences.
VX	Paint	Extracted Coupon	148	Mod 2 + Chem B	Positive Control		
VX	Paint	Extracted Coupon	80.0	Mod 3 + Chem A	Dahlgren + Chem A	0.3353	No significant differences.
VX	Paint	Extracted Coupon	109	Mod 3 + Chem A	Positive Control		

Table D101. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (VX in SPE Disks)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
VX	Paint	SPE	148	Baseline	Bleach	0.3412	No significant differences.
VX	Paint	SPE	326	Baseline	D7	0.3412	No significant differences.
VX	Paint	SPE	317	Baseline	Dahlgren Decon	0.3412	No significant differences.
VX	Paint	SPE	233	Baseline	Positive Control		
VX	Paint	SPE	210	Mod 1	Bleach	0.3351	No significant differences.
VX	Paint	SPE	273	Mod 1	Dahlgren Decon	0.9390	No significant differences.
VX	Paint	SPE	277	Mod 1	Positive Control		
VX	Paint	SPE	253	Mod 2 + Chem A	Bleach + Chem A	0.0456	Bleach + A < PC
VX	Paint	SPE	350	Mod 2 + Chem A	Positive Control		
VX	Paint	SPE	209	Mod 2 + Chem B	Bleach + Chem B	0.5381	No significant differences.
VX	Paint	SPE	169	Mod 2 + Chem B	Positive Control		
VX	Paint	SPE	233	Mod 3 + Chem A	Dahlgren + Chem A	0.5133	No significant differences.
VX	Paint	SPE	202	Mod 3 + Chem A	Positive Control		

Table D102. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (VX on Sealant Coupon Surfaces)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
VX	Sealant	Wipe	1.14	Baseline	Bleach	<0.0001	Bleach < PC
VX	Sealant	Wipe	42.1	Baseline	D7	0.0005	PC < D7
VX	Sealant	Wipe	0.97	Baseline	Dahlgren Decon	<0.0001	Dahlgren < PC
VX	Sealant	Wipe	30.8	Baseline	Positive Control		
VX	Sealant	Wipe	0.0049	Mod 1	Bleach	<0.0001	Bleach < PC
VX	Sealant	Wipe	0.37	Mod 1	Dahlgren Decon	<0.0001	Dahlgren < PC
VX	Sealant	Wipe	35.8	Mod 1	Positive Control		
VX	Sealant	Wipe	364	Mod 2 + Chem A	Bleach + Chem A	0.5128	No significant differences.
VX	Sealant	Wipe	416	Mod 2 + Chem A	Positive Control		
VX	Sealant	Wipe	294	Mod 2 + Chem B	Bleach + Chem B	0.5841	No significant differences.
VX	Sealant	Wipe	321	Mod 2 + Chem B	Positive Control		
VX	Sealant	Wipe	524	Mod 3 + Chem A	Dahlgren Decon + Chem A	0.5382	No significant differences.
VX	Sealant	Wipe	505	Mod 3 + Chem A	Positive Control		

Table D103. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (VX in Sealant Coupons)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
VX	Sealant	Extracted Coupon	417	Baseline	Bleach	0.0003	Bleach < PC
VX	Sealant	Extracted Coupon	572	Baseline	D7	0.0046	D7 < PC
VX	Sealant	Extracted Coupon	730	Baseline	Dahlgren Decon	0.1466	No significant differences.
VX	Sealant	Extracted Coupon	821	Baseline	Positive Control		
VX	Sealant	Extracted Coupon	513	Mod 1	Bleach	0.0003	Bleach < PC
VX	Sealant	Extracted Coupon	683	Mod 1	Dahlgren Decon	0.0036	Dahlgren < PC
VX	Sealant	Extracted Coupon	893	Mod 1	Positive Control		
VX	Sealant	Extracted Coupon	246	Mod 2 + Chem A	Bleach + Chem A	0.0782	No significant differences.
VX	Sealant	Extracted Coupon	197	Mod 2 + Chem A	Positive Control		
VX	Sealant	Extracted Coupon	327	Mod 2 + Chem B	Bleach + Chem B	0.6037	No significant differences.
VX	Sealant	Extracted Coupon	340	Mod 2 + Chem B	Positive Control		
VX	Sealant	Extracted Coupon	251	Mod 3 + Chem A	Dahlgren Decon + Chem A	0.0522	No significant differences.
VX	Sealant	Extracted Coupon	180	Mod 3 + Chem A	Positive Control		

Table D104. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (VX in SPE Disks)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
VX	Sealant	SPE	9.92	Baseline	Bleach	1.0000	No significant differences.
VX	Sealant	SPE	15.3	Baseline	D7	1.0000	No significant differences.
VX	Sealant	SPE	23.3	Baseline	Dahlgren Decon	1.0000	No significant differences.
VX	Sealant	SPE	8.74	Baseline	Positive Control		
VX	Sealant	SPE	<MQL	Mod 1	Bleach	0.7242	No significant differences.
VX	Sealant	SPE	5.65	Mod 1	Dahlgren Decon	0.7207	No significant differences.
VX	Sealant	SPE	1.66	Mod 1	Positive Control		
VX	Sealant	SPE	2.15	Mod 2 + Chem A	Bleach + Chem A	0.3545	No significant differences.
VX	Sealant	SPE	20.6	Mod 2 + Chem A	Positive Control		
VX	Sealant	SPE	0.041	Mod 2 + Chem B	Bleach + Chem B	0.3770	No significant differences.
VX	Sealant	SPE	1.00	Mod 2 + Chem B	Positive Control		
VX	Sealant	SPE	3.28	Mod 3 + Chem A	Dahlgren Decon + Chem A	0.1225	No significant differences.
VX	Sealant	SPE	101	Mod 3 + Chem A	Positive Control		

Positive Controls: Across-Test Method Comparisons

Table D105. Multiple comparison adjusted p-values between testing methods for positive controls (HD on Paint Coupon Surfaces)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1	Mod 2 – Chem A	Mod 2 + Chem A	Mod 2 – Chem B	Mod 2 + Chem B	Mod 3 – Chem A	Mod 3 + Chem A	Summary of Significant Bonferroni-Holm Differences
HD	Paint	Wipe	6.09	Baseline	0.3539	1.0000	1.0000	0.0038	0.7408	0.0031	1.0000	0.0013	Baseline < Mod 2 + Chem A Baseline < Mod 2 + Chem B Baseline < Mod 3 + Chem A Mod 1 < Mod 3 + Chem A Mod 3 – Chem A < Mod 2 + Chem A Mod 3 – Chem A < Mod 2 + Chem B Mod 3 – Chem A < Mod 3 + Chem A
HD	Paint	Wipe	13.5	Fate and Transport		1.0000	1.0000	0.6035	1.0000	0.5046	1.0000	0.2415	
HD	Paint	Wipe	10.0	Mod 1			1.0000	0.0640	1.0000	0.0519	1.0000	0.0223	
HD	Paint	Wipe	11.2	Mod 2 – Chem A				0.1403	1.0000	0.1137	1.0000	0.0519	
HD	Paint	Wipe	20.1	Mod 2 + Chem A					0.2817	1.0000	0.0239	1.0000	
HD	Paint	Wipe	12.3	Mod 2 – Chem B						0.2415	1.0000	0.1090	
HD	Paint	Wipe	20.4	Mod 2 + Chem B							0.0198	1.0000	
HD	Paint	Wipe	8.62	Mod 3 – Chem A								0.0080	
HD	Paint	Wipe	21.6	Mod 3 + Chem A									

Table D106. Multiple comparison adjusted p-values between testing methods for positive controls (HD in Paint Coupons)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1	Mod 2 – Chem A	Mod 2 + Chem A	Mod 2 – Chem B	Mod 2 + Chem B	Mod 3 – Chem A	Mod 3 + Chem A	Summary of Significant Bonferroni-Holm Differences
HD	Paint	Extracted Coupon	325	Baseline	1.0000	1.0000	0.8033	1.0000	1.0000	1.0000	1.0000	1.0000	No significant differences.
HD	Paint	Extracted Coupon	520	Fate and Transport		1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	
HD	Paint	Extracted Coupon	467	Mod 1			1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	
HD	Paint	Extracted Coupon	615	Mod 2 – Chem A				0.6121	1.0000	1.0000	1.0000	1.0000	
HD	Paint	Extracted Coupon	308	Mod 2 + Chem A					1.0000	1.0000	1.0000	1.0000	
HD	Paint	Extracted Coupon	492	Mod 2 – Chem B						1.0000	1.0000	1.0000	
HD	Paint	Extracted Coupon	383	Mod 2 + Chem B							1.0000	1.0000	
HD	Paint	Extracted Coupon	424	Mod 3 – Chem A								1.0000	
HD	Paint	Extracted Coupon	365	Mod 3 + Chem A									

Table D107. Multiple comparison adjusted p-values between testing methods for positive controls (HD in SPE Disks)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1	Mod 2 – Chem A	Mod 2 + Chem A	Mod 2 – Chem B	Mod 2 + Chem B	Mod 3 – Chem A	Mod 3 + Chem A	Summary of Significant Bonferroni-Holm Differences
HD	Paint	SPE	881	Baseline	0.0126	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	Baseline < Fate and Transport
HD	Paint	SPE	1520	Fate and Transport		0.5419	0.1823	1.0000	0.4599	0.3012	0.3574	0.5268	
HD	Paint	SPE	1140	Mod 1			1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	
HD	Paint	SPE	1060	Mod 2 – Chem A				1.0000	1.0000	1.0000	1.0000	1.0000	
HD	Paint	SPE	1190	Mod 2 + Chem A					1.0000	1.0000	1.0000	1.0000	
HD	Paint	SPE	1120	Mod 2 – Chem B						1.0000	1.0000	1.0000	
HD	Paint	SPE	1090	Mod 2 + Chem B							1.0000	1.0000	
HD	Paint	SPE	1110	Mod 3 – Chem A								1.0000	
HD	Paint	SPE	1140	Mod 3 + Chem A									

Table D108. Multiple comparison adjusted p-values between testing methods for positive controls (HD on Sealant Coupon Surfaces)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1	Mod 2 – Chem A	Mod 2 + Chem A	Mod 2 – Chem B	Mod 2 + Chem B	Mod 3 – Chem A	Mod 3 + Chem A	Summary of Significant Bonferroni-Holm Differences
HD	Sealant	Wipe	<MQL	Baseline	1.0000	1.0000	1.0000	<0.0001	1.0000	<0.0001	1.0000	<0.0001	Base < (Mod 2 + Chem A, Mod 2 + Chem B, Mod 3 + Chem A) Fate < (Mod 2 + Chem A, Mod 2 + Chem B, Mod 3 + Chem A) Mod 1 < (Mod 2 + Chem A, Mod 2 + Chem B, Mod 3 + Chem A) Mod 2 – Chem A < (Mod 2 + Chem A, Mod 2 + Chem B, Mod 3 + Chem A) Mod 2 + Chem A < Mod 2 + Chem B Mod 2 – Chem B < (Mod 2 + Chem A, Mod 2 + Chem B Mod 3 + Chem A) Mod 3 – Chem A < (Mod 2 + Chem A Mod 2 + Chem B, Mod 3 + Chem A) Mod 3 + Chem A < Mod 2 + Chem B
HD	Sealant	Wipe	1.15	Fate and Transport		1.0000	1.0000	<0.0001	1.0000	<0.0001	1.0000	<0.0001	
HD	Sealant	Wipe	<MQL	Mod 1			1.0000	<0.0001	1.0000	<0.0001	1.0000	<0.0001	
HD	Sealant	Wipe	<MQL	Mod 2 – Chem A				<0.0001	1.0000	<0.0001	1.0000	<0.0001	
HD	Sealant	Wipe	10.5	Mod 2 + Chem A					<0.0001	0.0003	<0.0001	1.0000	
HD	Sealant	Wipe	<MQL	Mod 2 – Chem B						<0.0001	1.0000	<0.0001	
HD	Sealant	Wipe	14.5	Mod 2 + Chem B							<0.0001	<0.0001	
HD	Sealant	Wipe	<MQL	Mod 3 – Chem A								<0.0001	
HD	Sealant	Wipe	9.33	Mod 3 + Chem A									

Table D109. Multiple comparison adjusted p-values between testing methods for positive controls (HD in Sealant Coupons)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1	Mod 2 – Chem A	Mod 2 + Chem A	Mod 2 – Chem B	Mod 2 + Chem B	Mod 3 – Chem A	Mod 3 + Chem A	Summary of Significant Bonferroni-Holm Differences
HD	Sealant	Extracted Coupon	188	Baseline	0.0099	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	Base < Fate Mod 2 – Chem A < Fate Mod 2 + Chem A < Fate Mod 2 + Chem B < Fate Mod 3 + Chem A < Fate
HD	Sealant	Extracted Coupon	384	Fate and Transport		0.1973	0.0292	0.0236	0.2184	0.0182	0.6527	0.0198	
HD	Sealant	Extracted Coupon	250	Mod 1			1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	
HD	Sealant	Extracted Coupon	212	Mod 2 – Chem A				1.0000	1.0000	1.0000	1.0000	1.0000	
HD	Sealant	Extracted Coupon	207	Mod 2 + Chem A					1.0000	1.0000	1.0000	1.0000	
HD	Sealant	Extracted Coupon	253	Mod 2 – Chem B						1.0000	1.0000	1.0000	
HD	Sealant	Extracted Coupon	201	Mod 2 + Chem B							1.0000	1.0000	
HD	Sealant	Extracted Coupon	276	Mod 3 – Chem A								1.0000	
HD	Sealant	Extracted Coupon	203	Mod 3 + Chem A									

Table D110. Multiple comparison adjusted p-values between testing methods for positive controls (HD in SPE Disks)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1	Mod 2 – Chem A	Mod 2 + Chem A	Mod 2 – Chem B	Mod 2 + Chem B	Mod 3 – Chem A	Mod 3 + Chem A	Summary of Significant Bonferroni-Holm Differences
HD	Sealant	SPE	1070	Baseline	0.0536	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	Mod 2 + Chem B < Fate and Transport
HD	Sealant	SPE	1470	Fate and Transport		0.0938	0.0705	0.1821	0.3150	0.0343	0.1417	0.0782	
HD	Sealant	SPE	100	Mod 1			1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	
HD	Sealant	SPE	1080	Mod 2 – Chem A				1.0000	1.0000	1.0000	1.0000	1.0000	
HD	Sealant	SPE	1130	Mod 2 + Chem A					1.0000	1.0000	1.0000	1.0000	
HD	Sealant	SPE	1160	Mod 2 – Chem B						1.0000	1.0000	1.0000	
HD	Sealant	SPE	1040	Mod 2 + Chem B							1.0000	1.0000	
HD	Sealant	SPE	1120	Mod 3 – Chem A								1.0000	
HD	Sealant	SPE	1090	Mod 3 + Chem A									

Table D111. Multiple comparison adjusted p-values between testing methods for positive controls (VX on Paint Coupon Surfaces)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1	Mod 2 – Chem A	Mod 2 + Chem A	Mod 2 – Chem B	Mod 2 + Chem B	Mod 3 – Chem A	Mod 3 + Chem A	Summary of Significant Bonferroni-Holm Differences
VX	Paint	Wipe	92.0	Baseline	0.0010	1.0000	1.0000	0.0154	1.0000	1.0000	1.0000	0.2860	Baseline < (Fate, Mod 2 + Chem A) Mod 1 < (Fate and Transport, Mod 2 + Chem A) Mod 2 – Chem A < Fate and Transport Mod 2 – Chem B < (Fate and Transport, Mod 2 + Chem A) Mod 2 + Chem B < Fate and Transport Mod 3 – Chem A < (Fate and Transport, Mod 2 + Chem A)
VX	Paint	Wipe	440	Fate and Transport		0.0009	0.0172	1.0000	0.0017	0.0062	0.0013	0.2860	
VX	Paint	Wipe	87.9	Mod 1			1.0000	0.0137	1.0000	1.0000	1.0000	0.2706	
VX	Paint	Wipe	180	Mod 2 – Chem A				0.2706	1.0000	1.0000	1.0000	1.0000	
VX	Paint	Wipe	357	Mod 2 + Chem A					0.0260	0.0967	0.0193	1.0000	
VX	Paint	Wipe	110	Mod 2 – Chem B						1.0000	1.0000	0.4541	
VX	Paint	Wipe	148	Mod 2 + Chem B							1.0000	1.0000	
VX	Paint	Wipe	100	Mod 3 – Chem A								0.3476	
VX	Paint	Wipe	266	Mod 3 + Chem A									

Table D112. Multiple comparison adjusted p-values between testing methods for positive controls (VX in Paint Coupons)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1	Mod 2 – Chem A	Mod 2 + Chem A	Mod 2 – Chem B	Mod 2 + Chem B	Mod 3 – Chem A	Mod 3 + Chem A	Summary of Significant Bonferroni-Holm Differences
VX	Paint	Extracted Coupon	295	Baseline	<0.0001	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	0.5610	All conditions < Fate and Transport
VX	Paint	Extracted Coupon	1020	Fate and Transport		<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	
VX	Paint	Extracted Coupon	256	Mod 1			1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	
VX	Paint	Extracted Coupon	368	Mod 2 – Chem A				1.0000	0.8226	0.2370	0.2370	0.0776	
VX	Paint	Extracted Coupon	274	Mod 2 + Chem A					1.0000	1.0000	1.0000	0.9332	
VX	Paint	Extracted Coupon	196	Mod 2 – Chem B						1.0000	1.0000	1.0000	
VX	Paint	Extracted Coupon	148	Mod 2 + Chem B							1.0000	1.0000	
VX	Paint	Extracted Coupon	147	Mod 3 – Chem A								1.0000	
VX	Paint	Extracted Coupon	108	Mod 3 + Chem A									

Table D113. Multiple comparison adjusted p-values between testing methods for positive controls (VX on Paint with SPE Disks)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1	Mod 2 – Chem A	Mod 2 + Chem A	Mod 2 – Chem B	Mod 2 + Chem B	Mod 3 – Chem A	Mod 3 + Chem A	Summary of Significant Bonferroni-Holm Differences
VX	Paint	SPE	233	Baseline	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	No significant differences.
VX	Paint	SPE	288	Fate and Transport		1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	
VX	Paint	SPE	277	Mod 1			1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	
VX	Paint	SPE	394	Mod 2 – Chem A				1.0000	1.0000	0.4777	1.0000	1.0000	
VX	Paint	SPE	350	Mod 2 + Chem A					1.0000	1.0000	1.0000	1.0000	
VX	Paint	SPE	231	Mod 2 – Chem B						1.0000	1.0000	1.0000	
VX	Paint	SPE	169	Mod 2 + Chem B							1.0000	1.0000	
VX	Paint	SPE	217	Mod 3 – Chem A								1.0000	
VX	Paint	SPE	202	Mod 3 + Chem A									

Table D114. Multiple comparison adjusted p-values between testing methods for positive controls (VX on Sealant Coupon Surfaces)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1	Mod 2 – Chem A	Mod 2 + Chem A	Mod 2 – Chem B	Mod 2 + Chem B	Mod 3 – Chem A	Mod 3 + Chem A	Summary of Significant Bonferroni-Holm Differences
VX	Sealant	Wipe	30.8	Baseline	1.0000	1.0000	1.0000	<0.0001	1.0000	<0.0001	1.0000	<0.0001	Baseline < (Mod 2 + Chem A, Mod 2 + Chem B, Mod 3 + Chem A) Fate and Transport < (Mod 2 + Chem A, Mod 2 + Chem B, Mod 3 + Chem A), Mod 1 < (Mod 2 + Chem A, Mod 2 + Chem B, Mod 3 + Chem A) Mod 2 – Chem A < (Mod 2 + Chem A, Mod 2 + Chem B, Mod 3 + Chem B) Mod 2 – Chem B < (Mod 2 + Chem A, Mod 2 + Chem B, Mod 3 + Chem A) Mod 2 + Chem B < (Mod 2 + Chem A, Mod 3 + Chem A) Mod 3 – Chem A < (Mod 2 + Chem A, Mod 2 + Chem B, Mod 3 + Chem A)
VX	Sealant	Wipe	69.2	Fate and Transport		1.0000	1.0000	<0.0001	1.0000	<0.0001	1.0000	<0.0001	
VX	Sealant	Wipe	35.5	Mod 1			1.0000	<0.0001	1.0000	<0.0001	1.0000	<0.0001	
VX	Sealant	Wipe	28.8	Mod 2 – Chem A				<0.0001	1.0000	<0.0001	1.0000	<0.0001	
VX	Sealant	Wipe	416	Mod 2 + Chem A					<0.0001	0.0356	<0.0001	0.0554	
VX	Sealant	Wipe	31.9	Mod 2 – Chem B						<0.0001	1.0000	<0.0001	
VX	Sealant	Wipe	321	Mod 2 + Chem B							<0.0001	<0.0001	
VX	Sealant	Wipe	38.1	Mod 3 – Chem A								<0.0001	
VX	Sealant	Wipe	505	Mod 3 + Chem A									

Table D115. Multiple comparison adjusted p-values between testing methods for positive controls (VX in Sealant Coupons)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1	Mod 2 – Chem A	Mod 2 + Chem A	Mod 2 – Chem B	Mod 2 + Chem B	Mod 3 – Chem A	Mod 3 + Chem A	Summary of Significant Bonferroni-Holm Differences
VX	Sealant	Extracted Coupon	821	Baseline	0.0214	0.6814	0.0003	<0.0001	0.2595	<0.0001	0.0160	<0.0001	Fate and Transport < (Baseline, Mod 1) Mod 2 – Chem A < (Baseline, Mod 1, Mod 2 – Chem B) Mod 2 + Chem A < (Baseline, Fate and Transport, Mod 1, Mod 2 – Chem A, Mod 2 – Chem B, Mod 3 – Chem A) Mod 2 + Chem B < (Baseline, Fate and Transport, Mod 1, Mod 2 – Chem A, Mod 2 – Chem B, Mod 3 – Chem A) Mod 2 – Chem B < Mod 1 Mod 3 – Chem A < (Baseline, Mod 1) Mod 3 + Chem A < (Baseline, Fate and Transport, Mod 1, Mod 2 – Chem A, Mod 2 – Chem B, Mod 2 + Chem B, Mod 3 – Chem A)
VX	Sealant	Extracted Coupon	653	Fate and Transport		0.0009	0.2711	<0.0001	0.6814	<0.0001	1.0000	<0.0001	
VX	Sealant	Extracted Coupon	893	Mod 1			<0.0001	<0.0001	0.0154	<0.0001	0.0007	<0.0001	
VX	Sealant	Extracted Coupon	551	Mod 2 – Chem A				<0.0001	0.0238	0.0032	0.3230	<0.0001	
VX	Sealant	Extracted Coupon	197	Mod 2 + Chem A					<0.0001	0.0544	<0.0001	1.0000	
VX	Sealant	Extracted Coupon	715	Mod 2 – Chem B						<0.0001	0.6814	<0.0001	
VX	Sealant	Extracted Coupon	340	Mod 2 + Chem B							0.0001	0.0259	
VX	Sealant	Extracted Coupon	646	Mod 3 – Chem A								<0.0001	
VX	Sealant	Extracted Coupon	180	Mod 3 + Chem A									

Table D116. Multiple comparison adjusted p-values between testing methods for positive controls (VX in SPE Disks)

Agent	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1	Mod 2 – Chem A	Mod 2 + Chem A	Mod 2 – Chem B	Mod 2 + Chem B	Mod 3 – Chem A	Mod 3 + Chem A	Summary of Significant Bonferroni-Holm Differences
VX	Sealant	SPE	8.7	Baseline	0.4883	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	No significant differences.
VX	Sealant	SPE	199	Fate and Transport		0.4307	0.7034	0.6708	0.4307	0.4307	0.4307	1.0000	
VX	Sealant	SPE	1.66	Mod 1			1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	
VX	Sealant	SPE	2.70	Mod 2 – Chem A				1.0000	1.0000	1.0000	1.0000	1.0000	
VX	Sealant	SPE	20.6	Mod 2 + Chem A					1.0000	1.0000	1.0000	1.0000	
VX	Sealant	SPE	0.58	Mod 2 – Chem B						1.0000	1.0000	1.0000	
VX	Sealant	SPE	1.00	Mod 2 + Chem B							1.0000	1.0000	
VX	Sealant	SPE	0.80	Mod 3 – Chem A								1.0000	
VX	Sealant	SPE	101	Mod 3 + Chem A									

REFERENCES

[D1] See, e.g., https://itl.nist.gov/div898/handbook/eda/section3/eda35a.htm. Last accessed March 15, 2022.

APPENDIX E

Pesticide Statistical Analysis

The objective of this analysis was to compare total fipronil and malathion mass recovered from each of two different materials (paint and sealant) and on each of three different sample types (wipes, extracted Coupon, and SPE disks) using four different combinations of one of two decontaminants applied under one of three testing methods (see Table E1). Three replicates were tested for each condition. Table E2 summarizes the study design.

Additional evaluation was performed using positive control tests. The positive control tests excluded application of the target decontaminant. The decontamination test conditions were compared to their analogous positive control tests to determine if statistically significant decontamination occurred. One set of positive controls was tested for the Baseline, and one set was tested for both Mod 1 and Mod 2. Positive controls were also compared between testing methods to evaluate whether the differences between methods might be attributable to external factors other than the decontamination process.

Table E1. Description of Testing Methods and Decontaminant Combinations

Testing Method	Decontaminant Dwell Time	Water Rinse?	Decontaminant Application
Baseline	0 minutes	No	10x Diluted Bleach or D7
Mod 1	60 minutes + Rinse/ Reapplication + 60 minutes	Yes	D7
Mod 2	120 minutes	No	D7

Observations below the method quantification limit (MQL) were set equal to the MQL, which ranged from 0.001 to 0.01 µg for fipronil and ranged from 1.0 µg to 11 µg for malathion dependent on sample type and required sample dilutions. Table E3 displays the percentage of observations below the MQL in each test condition, as well as the overall percentage of < MQL observations within each pesticide/material/sample type analysis. Many substitutions at the MQL value likely bias the estimates high and using a single substitution value artificially reduces the variance associated with the estimates. The reduction in variance may make the estimates more likely to be significantly different from other estimates when a real difference is not present.

For Mod 1 where the decontaminant was applied, rinsed, and applied again, the mass recovery from the rinse data was added to the mass recovery from the wipe data to obtain the total mass recovered for each replicate. The counts of data below the MQL in the wipe conditions reflect whether either the rinse or the wipe extraction mass was < MQL, so that samples with both rinse and wipe values below the MQL and samples with only one of the rinse or wipe values below the MQL were counted similarly.

All SPE disk replicates across both pesticides and materials and for both test samples and positive controls were measured to be below the MQL. Therefore, no comparisons could be conducted between testing method and decontaminant conditions for SPE disks. Similarly, only one observation for wipe replicates in each of the paint or sealant conditions with malathion was above the MQL; therefore, no comparisons were conducted between testing method and decontaminant conditions for wipes with malathion.

Table E2. Study Design for Testing Method and Decontaminant Comparison

Pesticide	Material	Testing Method	Decontaminant	Number of Replicates		
				Wipe Samples	Extracted Coupon Samples	SPE Disk Samples
Fipronil	Paint	Baseline	10x Diluted Bleach	3	3	3
Fipronil	Paint	Baseline	D7	3	3	3
Fipronil	Paint	Mod 1	D7	3	3	3
Fipronil	Paint	Mod 2	D7	3	3	3
Fipronil	Sealant	Baseline	10x Diluted Bleach	3	3	3
Fipronil	Sealant	Baseline	D7	3	3	3
Fipronil	Sealant	Mod 1	D7	3	3	3
Fipronil	Sealant	Mod 2	D7	3	3	3
Malathion	Paint	Baseline	10x Diluted Bleach	3	3	3
Malathion	Paint	Baseline	D7	3	3	3
Malathion	Paint	Mod 1	D7	3	3	3
Malathion	Paint	Mod 2	D7	3	3	3
Malathion	Sealant	Baseline	10x Diluted Bleach	3	3	3
Malathion	Sealant	Baseline	D7	3	3	3
Malathion	Sealant	Mod 1	D7	3	3	3
Malathion	Sealant	Mod 2	D7	3	3	3

Table E3. Percent of Observations < MQL in Each Test Condition

Pesticide	Material	Testing Method	Decontaminant	Percent of Replicates		
				Wipe Samples	Extracted Extracted Coupon Samples	SPE Disk Samples
Fipronil	Paint	Baseline	10x Diluted Bleach	0%	0%	100%
Fipronil	Paint	Baseline	D7	0%	0%	100%
Fipronil	Paint	Mod 1	D7	100%	33.3%	100%
Fipronil	Paint	Mod 2	D7	0%	0%	100%
Fipronil	Paint	**Total**		25%	8.33%	100%
Fipronil	Sealant	Baseline	10x Diluted Bleach	0%	0%	100%
Fipronil	Sealant	Baseline	D7	0%	0%	100%
Fipronil	Sealant	Mod 1	D7	0%	0%	100%
Fipronil	Sealant	Mod 2	D7	0%	0%	100%
Fipronil	Sealant	**Total**		0%	0%	100%
Malathion	Paint	Baseline	10x Diluted Bleach	66.7%	0%	100%
Malathion	Paint	Baseline	D7	100%	0%	100%
Malathion	Paint	Mod 1	D7	100%	0%	100%
Malathion	Paint	Mod 2	D7	100%	0%	100%
Malathion	Paint	**Total**		91.7%	0%	100%
Malathion	Sealant	Baseline	10x Diluted Bleach	100%	100%	100%
Malathion	Sealant	Baseline	D7	100%	0%	100%
Malathion	Sealant	Mod 1	D7	100%	0%	100%
Malathion	Sealant	Mod 2	D7	66.7%	0%	100%
Malathion	Sealant	**Total**		91.7%	25%	100%

Pesticide ANOVA

Comparison of Test Sample Results

A fixed effects ANOVA model was fitted to the pesticide total mass recovery data over all testing method and decontaminant combinations separately for each pesticide, material, and sample type condition. The models contained an effect for the combination of testing method and decontaminant and a residual error term. No random effect of trial was fitted due to only one trial being run for all replicates of each pesticide and material condition.

The assumptions of normally distributed errors with approximately equal variances were better met with untransformed data than with natural logarithm-transformed data, so data were left untransformed for the analysis. The models were fitted using SAS (version 9.4, 64-bit). The form of the model is presented in Equation E1.

$$Mass\ (\mu g) = \beta_0 + \beta_{ij} + \varepsilon_{ijk}$$

Equation E1

where:

- β_0 = intercept or overall mean total mass collected.
- β_{ij} = the fixed effect for the i^{th} testing method and j^{th} decontaminant.
- ε_{ijk} = random error for the k^{th} replicate from the i^{th} testing method, and j^{th} decontaminant. The random error is assumed to be $N(0, \sigma_\epsilon^2)$.

Using the model fitted to all test sample total mass recovery data for a given pesticide, material, and sample type, arithmetic means and 95% confidence intervals were calculated for the total mass recovered for each testing method and decontaminant combination. Pairwise comparisons were conducted to test for significant differences between pairs of testing method/decontaminant. There were six possible pairwise comparisons between the four testing method/decontaminant combinations for each pesticide/material/sample type condition, but not all such comparisons were of interest. Instead, the pairwise comparisons performed were restricted to include:

3. The three comparisons between testing methods with a shared decontaminant (e.g., the Baseline method with a D7 decontaminant vs Mod 1 with D7).
4. The one comparison between different decontaminants using the same testing method (Baseline method with 10x diluted bleach vs Baseline method with D7).

This comparison scheme amounted to 4 comparisons for each of the 12 pesticide/material/sample type conditions, or 48 total comparisons. See also Table E4 for a summary of the comparisons performed for fipronil and malathion on paint and sealant. Note that due to the large number of measurements below the MQL for the SPE and the malathion wipe on paint conditions, it was possible to conduct only 24 of these comparisons.

Table E4. Fipronil and Malathion Paint and Sealant Comparison Sets

Test	Comparison Set 1	Comparison Set 2	Sample Type			
Baseline	10x Diluted Bleach	10x Diluted Bleach		Wipe	Extracted Coupon	SPE
	D7	D7		Wipe	Extracted Coupon	SPE
Mod 1	D7	D7	Rinse	Wipe	Extracted Coupon	SPE
Mod 2	D7	D7		Wipe	Extracted Coupon	SPE

The Bonferroni-Holm multiple comparisons procedure was performed to adjust the *p*-values of the pairwise comparisons so that a familywise error rate of 0.05 was maintained over all 4 comparisons of interest within a pesticide/material/sample type condition. This procedure limits the probability of a difference being falsely identified as statistically significant when no true difference exists, and the difference is due to sampling variability. The familywise error rate means that the chance of a sampling-based falsely significant result is no more than 1 in 20 for the entire set of 4 comparisons. The Bonferroni-Holm procedure was selected due to its power in detecting true differences when performing a restricted number of pairwise comparisons.

Comparison of Test Sample Results with Positive Controls

A fixed effects ANOVA model was fitted to the total mass recovery data for decontaminants and positive controls results within each pesticide/material/sample type/testing method condition. The models contained an effect for the combination of decontaminant/positive control status and a residual error term. No random effect of trial was fitted due to only one trial being run for all replicates of each pesticide and material condition.

The assumptions of normality and equality of variances were better met with untransformed data than with natural logarithm-transformed data, so data were left untransformed for the analysis. The models were fitted using SAS (version 9.4, 64-bit). The form of the model is presented in Equation E2.

$$Mass\ (\mu g) = \beta_0 + \beta_i + \varepsilon_{ij}$$

Equation E2

where:

- β_0 = intercept or overall mean total mass collected.
- β_i = the fixed effect for the i^{th} decontaminant / positive control condition.
- ε_{ij} = random error for the j^h replicate from the i^{th} decontaminant / positive control condition. The random error is assumed to be $N(0, \sigma_\epsilon^2)$.

Using the model fitted to all total mass recovery data for a given pesticide, material, and sample type, arithmetic means were calculated for the total mass recovered for each testing method and decontaminant combination. Pairwise comparisons were conducted to test for significant differences between each decontaminant and the positive control condition within a pesticide/material/sample type/testing method condition.

The Bonferroni-Holm multiple comparisons procedure was performed to adjust the *p*-values of the pairwise comparisons so that a familywise error rate of 0.05 was maintained over all comparisons of interest within a pesticide/material/sample type/testing method condition. The Bonferroni-Holm procedure was selected due to its power in detecting true differences when performing a restricted number of pairwise comparisons.

Comparison of Positive Control Results

A fixed effects ANOVA model was fitted to the total mass recovery data for positive controls results under each testing method within each pesticide/material/sample type condition. The 72-Hour Fate and Transport data for each pesticide/material/sample type were also included as a test method condition in this analysis. The models contained an effect for the combination of testing method under which the positive control was collected and the chemical application, and a residual error term. No random effect of trial was fitted due to only one trial being run for all replicates of each pesticide and material condition. Data were left untransformed for the analysis to remain consistent with analyses of test samples and test samples vs positive controls. The models were fitted using SAS (version 9.4, 64-bit). The form of the model is presented in Equation E3.

$$Mass\ (\mu g) = \beta_0 + \beta_{ij} + \varepsilon_{ijk}$$

Equation E3

where:

- β_0 = intercept or overall mean total mass collected.
- β_{ij} = the fixed effect for the positive controls from the i^{th} testing method and j^{th} chemical application condition.
- ε_{ijk} = random error for the k^{h} positive control from the i^{th} testing method / j^{th} chemical application condition. The random error is assumed to be $N(0, \sigma_{\epsilon}^2)$.

Using the model fitted to all positive control data for a given pesticide, material, and sample type, arithmetic means were calculated for the total mass recovered for each testing method and decontaminant combination for each pesticide, material, and sample type. Pairwise comparisons were conducted to test for significant differences between all three positive controls conditions with a pesticide/material/sample type, resulting in three pairwise comparisons per model.

The Bonferroni-Holm multiple comparisons procedure was performed to adjust the *p*-values of the pairwise comparisons so that a familywise error rate of 0.05 was maintained over all comparisons of interest within a pesticide/material/sample type. The Bonferroni-Holm procedure was selected for consistency with the previous analyses.

Outliers

Potential outliers were determined by calculating the deleted (externally) studentized residuals. If the absolute value of the standardized residual was greater than 3, then the observation was considered a potential outlier. If potential outliers were found, the results were checked to determine the validity of the outlying data and probable causes for the outliers. If no probable cause was found, the outlier was included in the subsequent analysis.

Pesticide ANOVA Results

Table E5 through Table E7 displays the potential outliers identified by examining the externally studentized residuals in each study condition. Probable cause was not identified for any of the listed outliers. Thus, all replicates were included in the final analysis.

Table E5. Potential Outliers Identified from Test Samples

Analysis	Pesticide	Material	Sample Type	Test Method	Decontaminant	Replicate Number	Total Mass Recovery (µg)	Externally Studentized Residual
Test Samples	Fipronil	Paint	Extracted Coupon	Baseline	10x Diluted Bleach	1	3.04	3.21
	Fipronil	Paint	Wipe	Baseline	10x Diluted Bleach	1	5.43	19.21
	Fipronil	Sealant	Extracted Coupon	Mod 2	D7	1	0.63	3.5
	Fipronil	Sealant	Extracted Coupon	Mod 2	D7	3	0.41	-3.65
	Fipronil	Sealant	Wipe	Mod 2	D7	3	0.84	-3.59
	Malathion	Paint	Extracted Coupon	Mod 2	D7	3	6.01	-3.9
	Malathion	Sealant	Extracted Coupon	Mod 2	D7	1	7.63	-4.42

Table E6. Potential Outliers Identified from Test Samples vs Positive Controls

Analysis	Pesticide	Material	Sample Type	Test Method	Decontaminant or Positive Control (PC)	Replicate Number	Total Mass Recovery (µg)	Externally Studentized Residual
Test Samples vs Positive Control Comparison	Fipronil	Paint	Extracted Coupon	Baseline	Positive controls	2	2.29	-3.27
	Fipronil	Paint	Extracted Coupon	Mod 1/ Mod 2	Positive controls	2	4.48	6.98
	Fipronil	Paint	Wipe	Baseline	Bleach	1	5.43	3.90
	Fipronil	Paint	Wipe	Mod 1/ Mod 2	Positive controls	2	6.13	-13.7
	Fipronil	Sealant	Extracted Coupon	Baseline	Positive controls	2	1.67	5.18
	Fipronil	Sealant	Extracted Coupon	Mod 1/ Mod 2	Positive controls	2	1.96	10.3
	Malathion	Sealant	Extracted Coupon	Baseline	D7	3	4.64	-3.92
	Malathion	Sealant	Extracted Coupon	Mod 1/Mod 2	Positive controls	1	15.4	6.71
	Malathion	Sealant	Wipe	Baseline	D7	3	11.0	4.41
	Malathion	Sealant	Wipe	Mod 1/ Mod 2	Positive controls	1	1.64	-4.27
	Malathion	Sealant	Wipe	Mod 1/ Mod 2	Positive controls	3	8.49	3.36

Table E7. Potential Outliers Identified from Positive Controls Analysis

Analysis	Pesticide	Material	Sample Type	Test Method	Replicate Number	Total Mass Recovery (µg)	Externally Studentized Residual
Positive Controls	Fipronil	Paint	Extracted Coupon	Mod 1/Mod 2	2	4.48	3.24
	Fipronil	Sealant	Extracted Coupon	Mod 1 / Mod 2	2	1.96	3.88
	Malathion	Paint	Wipe	Fate and Transport	1	1.25	-10.6
	Malathion	Sealant	Extracted Coupon	Mod 1/Mod 2	1	15.4	7.34

Figure E1 to Figure E4 display the total mass recoveries for the replicates in each pesticide/material/sample type condition. Statistical summaries including arithmetic means and 95% confidence intervals are presented in Table E8 to Table E11 and are sorted in order of estimated mean total mass recovery within each pesticide, material, and sample type. Confidence bounds were not adjusted for multiple comparisons between test conditions, and thus should not be used to evaluate significant differences between test conditions.

Figure E1. Total recovery mass of test samples for all testing method and decontaminant combinations over all sample types for Fipronil on Paint Coupon

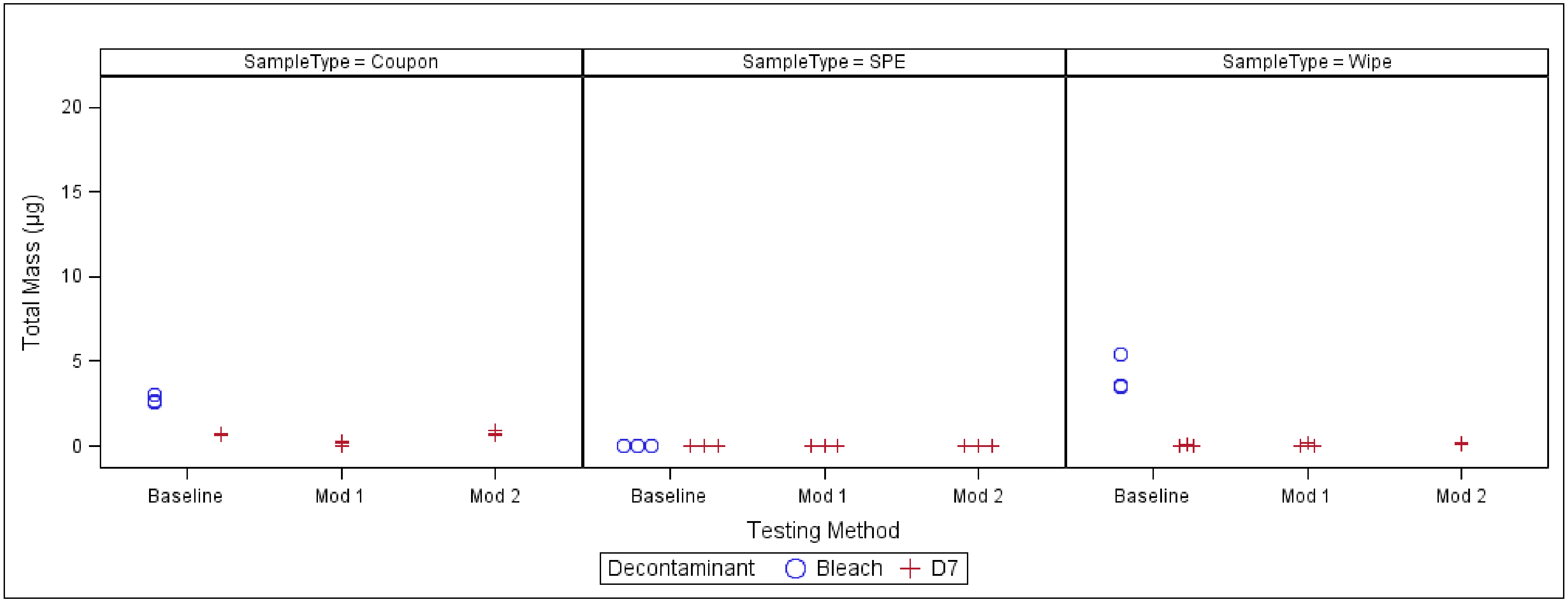

Figure E2. Total recovery mass of test samples for all testing method and decontaminant combinations over all sample types for Fipronil on Sealant Coupon

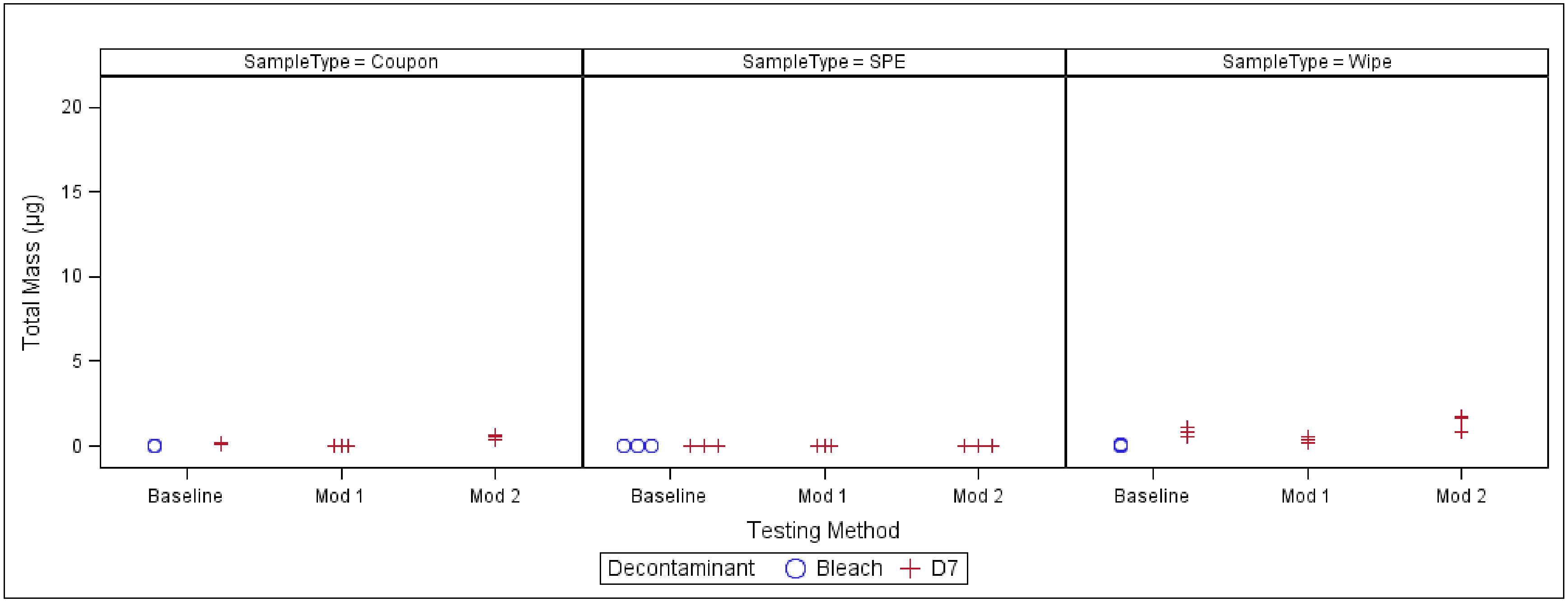

Figure E3. Total recovery mass of test samples for all testing method and decontaminant combinations over all sample types for Malathion on Paint Coupon

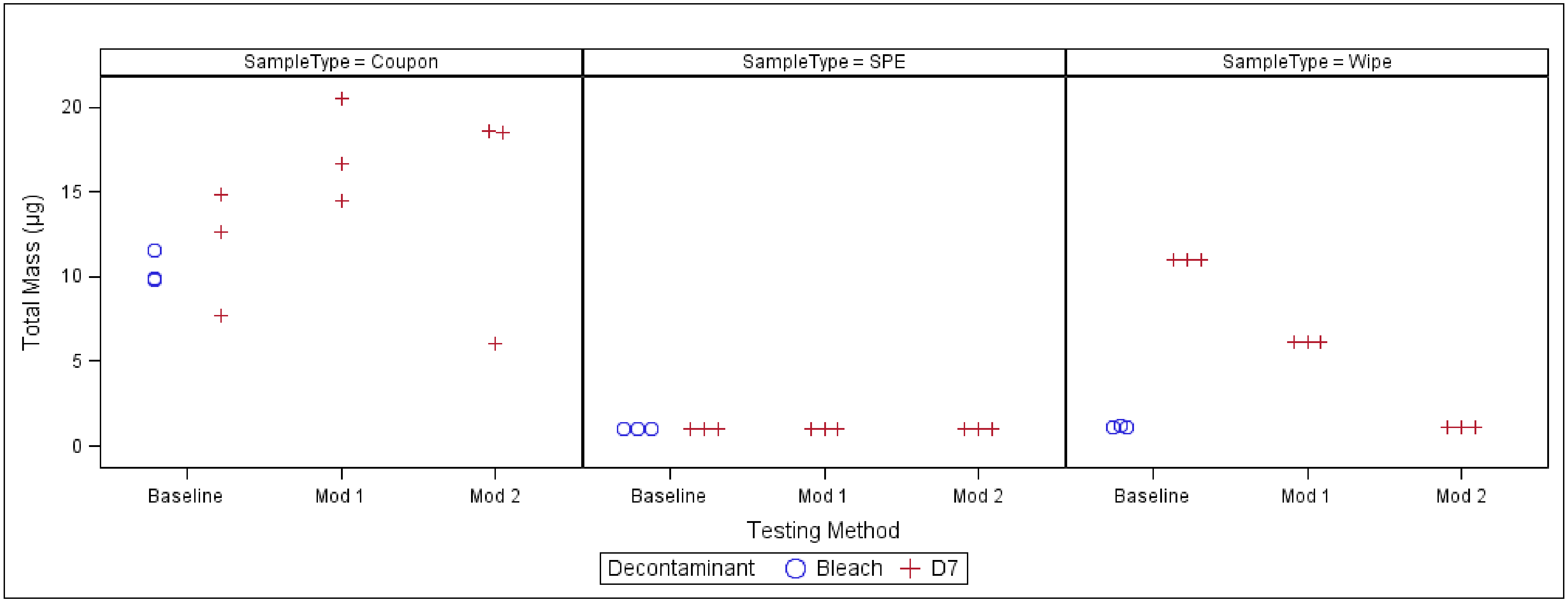

Figure E4. Total recovery mass of test samples for all testing method and decontaminant combinations over all sample types for Malathion on Sealant Coupon

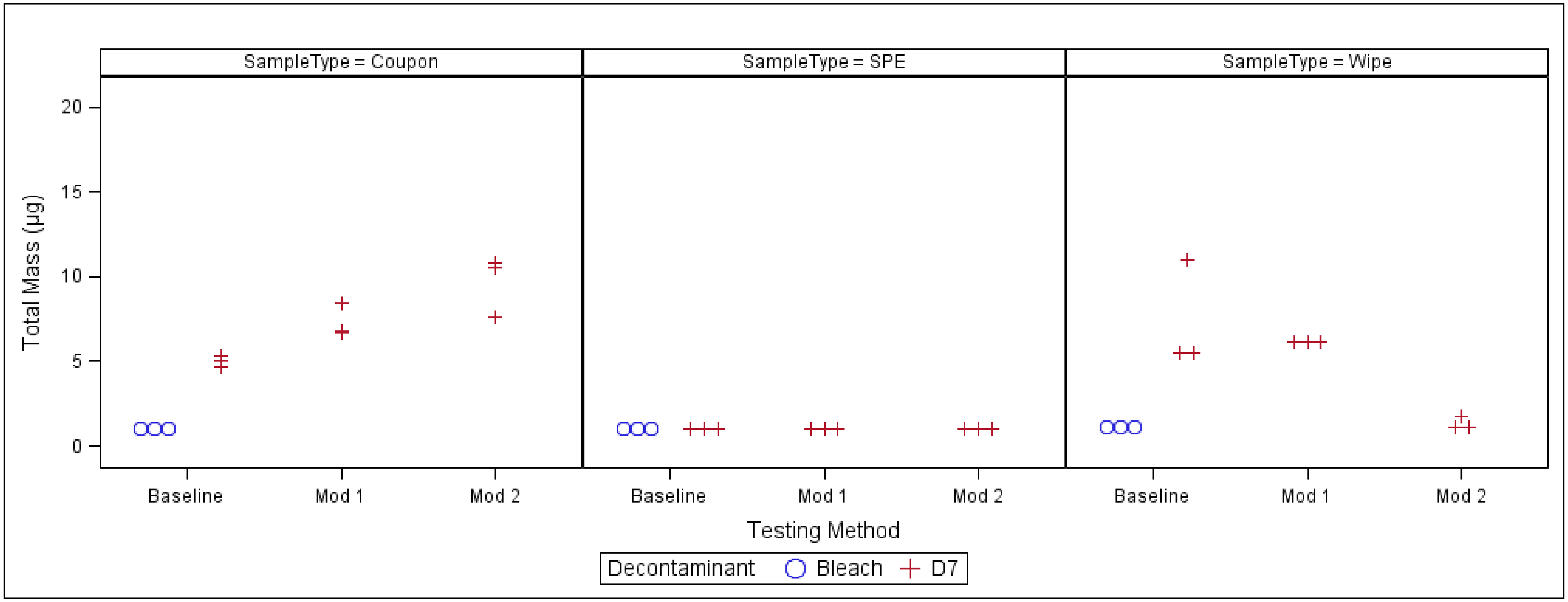

Table E8. Sorted Arithmetic Means and Unadjusted 95% Confidence Intervals (Fipronil-Paint Coupons)

Pesticide	Material	Sample Type	Test Method	Decontaminant	Mean Total Mass Recovery (µg)	Lower 95% Confidence Bound	Upper 95% Confidence Bound
Fipronil	Paint	Wipe	Baseline	D7	0.057	<MQL	0.80
Fipronil	Paint	Wipe	Mod 1	D7	0.089	<MQL	0.83
Fipronil	Paint	Wipe	Mod 2	D7	0.12	<MQL	0.85
Fipronil	Paint	Wipe	Baseline	10x Diluted Bleach	4.16	3.42	4.90
Fipronil	Paint	Extracted Coupon	Mod 1	D7	0.15	<MQL	0.37
Fipronil	Paint	Extracted Coupon	Baseline	D7	0.69	0.48	0.90
Fipronil	Paint	Extracted Coupon	Mod 2	D7	0.79	0.58	1.01
Fipronil	Paint	Extracted Coupon	Baseline	10x Diluted Bleach	2.76	2.54	2.97
Fipronil	Paint	SPE	Baseline	10x Diluted Bleach	<MQL	N/A	N/A
Fipronil	Paint	SPE	Baseline	D7	<MQL	N/A	N/A
Fipronil	Paint	SPE	Mod 1	D7	<MQL	N/A	N/A
Fipronil	Paint	SPE	Mod 2	D7	<MQL	N/A	N/A

Table E9. Sorted Arithmetic Means and Unadjusted 95% Confidence Intervals (Fipronil-Sealant Coupons)

Pesticide	Material	Sample Type	Test Method	Decontaminant	Mean Total Mass Recovery (µg)	Lower 95% Confidence Bound	Upper 95% Confidence Bound
Fipronil	Sealant	Wipe	Baseline	10x Diluted Bleach	0.068	<MQL	0.48
Fipronil	Sealant	Wipe	Mod 1	D7	0.37	<MQL	0.78
Fipronil	Sealant	Wipe	Baseline	D7	0.83	0.41	1.24
Fipronil	Sealant	Wipe	Mod 2	D7	1.42	1.01	1.83
Fipronil	Sealant	Extracted Coupon	Mod 1	D7	0.015	<MQL	0.093
Fipronil	Sealant	Extracted Coupon	Baseline	10x Diluted Bleach	0.037	<MQL	0.12
Fipronil	Sealant	Extracted Coupon	Baseline	D7	0.15	0.073	0.23
Fipronil	Sealant	Extracted Coupon	Mod 2	D7	0.52	0.44	0.60
Fipronil	Sealant	SPE	Baseline	10x Diluted Bleach	<MQL	N/A	N/A
Fipronil	Sealant	SPE	Baseline	D7	<MQL	N/A	N/A
Fipronil	Sealant	SPE	Mod 1	D7	<MQL	N/A	N/A
Fipronil	Sealant	SPE	Mod 2	D7	<MQL	N/A	N/A

Table E10. Sorted Arithmetic Means and Unadjusted 95% Confidence Intervals (Malathion-Paint Coupons)

Pesticide	Material	Sample Type	Test Method	Decontaminant	Mean Total Mass Recovery (µg)	Lower 95% Confidence Bound	Upper 95% Confidence Bound
Malathion	Paint	Wipe	Baseline	D7	<MQL	N/A	N/A
Malathion	Paint	Wipe	Mod 1	D7	<MQL	N/A	N/A
Malathion	Paint	Wipe	Mod 2	D7	<MQL	N/A	N/A
Malathion	Paint	Wipe	Baseline	10x Diluted Bleach	1.13	1.10	1.17
Malathion	Paint	Extracted Coupon	Baseline	10x Diluted Bleach	10.43	4.60	16.25
Malathion	Paint	Extracted Coupon	Baseline	D7	11.72	5.89	17.54
Malathion	Paint	Extracted Coupon	Mod 2	D7	14.36	8.54	20.18
Malathion	Paint	Extracted Coupon	Mod 1	D7	17.22	11.40	23.04
Malathion	Paint	SPE	Baseline	10x Diluted Bleach	<MQL	N/A	N/A
Malathion	Paint	SPE	Baseline	D7	<MQL	N/A	N/A
Malathion	Paint	SPE	Mod 1	D7	<MQL	N/A	N/A
Malathion	Paint	SPE	Mod 2	D7	<MQL	N/A	N/A

Table E11. Sorted Arithmetic Means and Unadjusted 95% Confidence Intervals (Malathion-Sealant Coupons)

Pesticide	Material	Sample Type	Test Method	Decontaminant	Mean Total Mass Recovery (µg)	Lower 95% Confidence Bound	Upper 95% Confidence Bound
Malathion	Sealant	Wipe	Baseline	10x Diluted Bleach	<MQL	N/A	N/A
Malathion	Sealant	Wipe	Baseline	D7	<MQL	N/A	N/A
Malathion	Sealant	Wipe	Mod 1	D7	<MQL	N/A	N/A
Malathion	Sealant	Wipe	Mod 2	D7	1.33	<MQL	3.45
Malathion	Sealant	Extracted Coupon	Baseline	10x Diluted Bleach	1.00	<MQL	2.37
Malathion	Sealant	Extracted Coupon	Baseline	D7	4.98	3.61	6.35
Malathion	Sealant	Extracted Coupon	Mod 1	D7	7.29	5.93	8.66
Malathion	Sealant	Extracted Coupon	Mod 2	D7	9.66	8.29	11.03
Malathion	Sealant	SPE	Baseline	10x Diluted Bleach	<MQL	N/A	N/A
Malathion	Sealant	SPE	Baseline	D7	<MQL	N/A	N/A
Malathion	Sealant	SPE	Mod 1	D7	<MQL	N/A	N/A
Malathion	Sealant	SPE	Mod 2	D7	<MQL	N/A	N/A

Table E14 to Table E37 display the results of the Bonferroni-Holm-adjusted pairwise comparisons for the specified comparisons between the test conditions. Of the 24 pairwise comparisons between testing method and decontaminant combinations with sufficient observations above the MQL, 13 were statistically significant.

The capital letters in the "Similarity Designation" column of Table E14 to Table E37 indicate the statistical similarity of the mean total mass of a given testing method and decontaminant combination to the statistical similarity of all other combinations tested for the given pesticide/material/sample type condition. All rows with the same similarity designation value are not statistically significantly different from each other, while rows that did not share any similarity designation values are significantly different. For example, in Table E14 for the Fipronil/Sealant/Wipe condition, the Mod 1 method with the D7 decontaminant has similarity designation A, indicating that it is similar to other combinations with the A designation, including the baseline method with D7 (designation AB), but it is different from combinations without an A in the designation, such as Mod 2 with D7 (designation B).

The results of the test sample comparisons are summarized below.

D7 comparisons:

- Mod 1 paint and sealant extracted coupon samples resulted in lower fipronil values (lower fipronil recovery) compared to Baseline and Mod 2.
- Baseline sealant extracted coupon samples resulted in lower malathion values compared to Mod 1 or Mod 2. Note that for malathion there was no difference for Baseline, Mod 1, or Mod 2 paint coupon samples.

Baseline comparisons:

- D7 resulted in lower fipronil values for both paint coupon wipes and paint extracted coupons compared to 10x diluted bleach.
- D7 resulted in lower fipronil values for sealant coupons compared to bleach.
- 10x diluted bleach resulted in lower malathion values only for sealant coupons.

Table E38 to Table E49 display the results of the Bonferroni-Holm-adjusted pairwise comparisons between the test samples and positive controls (within-test comparisons). A summary of these results showing the significant differences for the four pairwise comparisons between test samples to positive controls for each pesticide/material/sample combination is shown in Table E12. Recall that SPE results were all nondetects, as were most malathion paint wipes, so comparisons could not be made. For D7 in Baseline, Mod 1, and Mod 2 tests, all fipronil extracted coupon and wipe samples for paint and sealant coupons were less that the positive controls. For 10x diluted bleach (used only for Baseline tests) all fipronil coupon and wipe samples for paint and sealant were less that the positive controls except for paint coupons, which were not different from the positive controls. No differences from the positive controls were observed for malathion testing, aside from the D7 baseline sealant coupon where the Baseline result was actually higher than the positive control due to required sample dilutions that resulted in elevated nondetect results.

Table E12. Summary of Test Sample to Positive Control Comparisons

Chemical	Material	Wipe	Extracted Coupon	SPE
Fipronil	Paint	4 differences	3 differences	Not Applicable
	Sealant	4 differences	4 differences	Not Applicable
Malathion	Paint	Not Applicable	No differences	Not Applicable
	Sealant	No differences	1 difference	Not Applicable

Table E50 to Table E61 display the results of the Bonferroni-Holm-adjusted pairwise comparisons between the positive control conditions (across-test method comparisons). A summary of these results showing significant differences for the three pairwise comparisons between positive controls for each pesticide/material/sample combination is shown in Table E13. Note that comparisons for the SPE data were not possible as all results were nondetects. The three differences that were observed were associated with comparisons to the Fate and Transport data; the reason for these differences is not apparent.

Table E13. Summary of Positive Control Comparisons

Chemical	Material	Wipe	Extracted Coupon	SPE
Fipronil	Paint	No differences	1 difference	Not Applicable
	Sealant	No differences	No differences	Not Applicable
Malathion	Paint	2 differences	No differences	Not Applicable
	Sealant	No differences	No differences	Not Applicable

Table E14. Multiple comparison adjusted p-values between test methods for D7 (Fipronil on Paint Coupon Surfaces)

Pesticide	Material	Sample Type	Decontaminant	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Dahlgren		Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2	
Fipronil	Paint	Wipe	D7	0.057	Baseline	A	1.0000	1.0000	No significant differences
Fipronil	Paint	Wipe	D7	0.089	Mod 1	A		1.0000	
Fipronil	Paint	Wipe	D7	0.12	Mod 2	A			

Table E15. Multiple comparison adjusted p-values between test methods for D7 (Fipronil in Paint Coupons)

Pesticide	Material	Sample Type	Decontaminant	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	Dahlgren		Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2	
Fipronil	Paint	Extracted Coupon	D7	0.69	Baseline	A	0.0066	0.4470	Mod 1 < Baseline Mod 1 < Mod 2
Fipronil	Paint	Extracted Coupon	D7	0.15	Mod 1	B		0.0034	
Fipronil	Paint	Extracted Coupon	D7	0.79	Mod 2	A			

Table E16. Multiple comparison adjusted p-values between test methods for D7 (Fipronil in SPE Disks)

Pesticide	Material	Sample Type	Decontaminant	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	D7		Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2	
Fipronil	Paint	SPE	D7	<MQL	Baseline	N/A	N/A	N/A	N/A
Fipronil	Paint	SPE	D7	<MQL	Mod 1	N/A		N/A	
Fipronil	Paint	SPE	D7	<MQL	Mod 2	N/A			

Table E17. Multiple comparison adjusted p-values between test methods for D7 (Fipronil on Sealant Coupon Surfaces)

Pesticide	Material	Sample Type	Decontaminant	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	D7		Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2	
Fipronil	Sealant	Wipe	D7	0.83	Baseline	AB	0.1076	0.0924	Mod 1 < Mod 2
Fipronil	Sealant	Wipe	D7	0.37	Mod 1	A		0.0125	
Fipronil	Sealant	Wipe	D7	1.42	Mod 2	B			

Table E18. Multiple comparison adjusted p-values between test methods for D7 (Fipronil in Sealant Coupons)

Pesticide	Material	Sample Type	Decontaminant	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	D7		Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2	
Fipronil	Sealant	Extracted Coupon	D7	0.15	Baseline	A	0.0428	0.0002	Baseline < Mod 2 Mod 1 < Baseline Mod 1 < Mod 2
Fipronil	Sealant	Extracted Coupon	D7	0.015	Mod 1	B		<0.0001	
Fipronil	Sealant	Coupon	D7	0.52	Mod 2	C			

Table E19. Multiple comparison adjusted p-values between test methods for D7 (Fipronil in SPE Disks)

Pesticide	Material	Sample Type	Decontaminant	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	D7		Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2	
Fipronil	Sealant	SPE	D7	<MQL	Baseline	N/A	N/A	N/A	N/A
Fipronil	Sealant	SPE	D7	<MQL	Mod 1	N/A		N/A	
Fipronil	Sealant	SPE	D7	<MQL	Mod 2	N/A			

Table E20. Multiple comparison adjusted p-values between test methods for D7 (Malathion on Paint Coupon Surfaces)

Pesticide	Material	Sample Type	Decontaminant	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	D7		Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2	
Malathion	Paint	Wipe	D7	<MQL	Baseline	N/A	N/A	N/A	N/A
Malathion	Paint	Wipe	D7	<MQL	Mod 1	N/A		N/A	
Malathion	Paint	Wipe	D7	<MQL	Mod 2	N/A			

Table E21. Multiple comparison adjusted p-values between test methods for D7 (Malathion in Paint Coupons)

Pesticide	Material	Sample Type	Decontaminant	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	D7			Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2		
Malathion	Paint	Extracted Coupon	D7	11.7	Baseline	A	0.6471	1.0000		No significant differences
Malathion	Paint	Extracted Coupon	D7	17.2	Mod 1	A		1.0000		
Malathion	Paint	Extracted Coupon	D7	14.4	Mod 2	A				

Table E22. Multiple comparison adjusted p-values between test methods for D7 (Malathion in SPE Disks)

Pesticide	Material	Sample Type	Decontaminant	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	D7		Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2	
Malathion	Paint	SPE	D7	<MQL	Baseline	N/A	N/A	N/A	N/A
Malathion	Paint	SPE	D7	<MQL	Mod 1	N/A		N/A	
Malathion	Paint	SPE	D7	<MQL	Mod 2	N/A			

Table E23. Multiple comparison adjusted p-values between test methods for D7 (Malathion on Sealant Coupon Surfaces)

Pesticide	Material	Sample Type	Decontaminant	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	D7		Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2	
Malathion	Sealant	Wipe	D7	<MQL	Baseline	N/A	N/A	N/A	N/A
Malathion	Sealant	Wipe	D7	<MQL	Mod 1	N/A		N/A	
Malathion	Sealant	Wipe	D7	1.33	Mod 2	N/A			

Table E24. Multiple comparison adjusted p-values between test methods for D7 (Malathion in Sealant Coupons)

Pesticide	Material	Sample Type	Decontaminant	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	D7		Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2	
Malathion	Sealant	Extracted Coupon	D7	4.98	Baseline	A	0.0447	0.0021	Baseline < Mod 1
Malathion	Sealant	Extracted Coupon	D7	7.29	Mod 1	B		0.0447	Baseline < Mod 2
Malathion	Sealant	Extracted Coupon	D7	9.66	Mod 2	C			Mod 1 < Mod 2

Table E25. Multiple comparison adjusted p-values between test methods for D7 (Malathion in SPE Disks)

Pesticide	Material	Sample Type	Decontaminant	Mean Total Mass Recovery (µg)	Testing Method	Similarity Designation	D7		Summary of Significant Bonferroni-Holm Differences
							Mod 1	Mod 2	
Malathion	Sealant	SPE	D7	<MQL	Baseline	N/A	N/A	N/A	N/A
Malathion	Sealant	SPE	D7	<MQL	Mod 1	N/A		N/A	
Malathion	Sealant	SPE	D7	<MQL	Mod 2	N/A			

Table E26. Multiple comparison adjusted p-values between decontaminants for Baseline testing method (Fipronil on Paint Coupon Surfaces)

Pesticide	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline	Summary of Significant Bonferroni-Holm Differences
							D7	
Fipronil	Paint	Wipe	Baseline	4.16	10x Diluted Bleach	A	0.0001	D7 < 10x Diluted Bleach
Fipronil	Paint	Wipe	Baseline	0.057	D7	B		

Table E27. Multiple comparison adjusted p-values between decontaminants for Baseline testing method (Fipronil in Paint Coupons)

Pesticide	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline	Summary of Significant Bonferroni-Holm Differences
							D7	
Fipronil	Paint	Extracted Coupon	Baseline	2.76	10x Diluted Bleach	A	<0.0001	D7 < 10x Diluted Bleach
Fipronil	Paint	Extracted Coupon	Baseline	0.69	D7	B		

Table E28. Multiple comparison adjusted p-values between decontaminants for Baseline testing method (Fipronil in SPE Disks)

Pesticide	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline	Summary of Significant Bonferroni-Holm Differences
							D7	
Fipronil	Paint	SPE	Baseline	<MQL	10x Diluted Bleach	N/A	N/A	N/A
Fipronil	Paint	SPE	Baseline	<MQL	D7	N/A		

Table E29. Multiple comparison adjusted p-values between decontaminants for Baseline testing method (Fipronil on Sealant Coupon Surfaces)

Pesticide	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline	Summary of Significant Bonferroni-Holm Differences
							D7	
Fipronil	Sealant	Wipe	Baseline	0.068	10x Diluted Bleach	A	0.0510	No significant differences
Fipronil	Sealant	Wipe	Baseline	0.83	D7	A		

Table E30. Multiple comparison adjusted p-values between decontaminants for Baseline testing method (Fipronil in Sealant Coupons)

Pesticide	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline	Summary of Significant Bonferroni-Holm Differences
							D7	
Fipronil	Sealant	Extracted Coupon	Baseline	0.037	10x Diluted Bleach	A	0.0440	10x Diluted Bleach < D7
Fipronil	Sealant	Extracted Coupon	Baseline	0.15	D7	B		

Table E31. Multiple comparison adjusted p-values between decontaminants for Baseline testing method (Fipronil in SPE Disks)

Pesticide	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline	Summary of Significant Bonferroni-Holm Differences
							D7	
Fipronil	Sealant	SPE	Baseline	<MQL	10x Diluted Bleach	N/A	N/A	N/A
Fipronil	Sealant	SPE	Baseline	<MQL	D7	N/A		

Table E32. Multiple comparison adjusted p-values between decontaminants for Baseline testing method (Malathion on Paint Coupon Surfaces)

Pesticide	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline	Summary of Significant Bonferroni-Holm Differences
							D7	
Malathion	Paint	Wipe	Baseline	1.13	10x Diluted Bleach	N/A	N/A	N/A
Malathion	Paint	Wipe	Baseline	<MQL	D7	N/A		

Table E33. Multiple comparison adjusted p-values between decontaminants for Baseline testing method (Malathion in Paint Coupons)

Pesticide	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline	Summary of Significant Bonferroni-Holm Differences
							D7	
Malathion	Paint	Extracted Coupon	Baseline	10.4	10x Diluted Bleach	A	1.0000	No significant differences
Malathion	Paint	Extracted Coupon	Baseline	11.7	D7	A		

Table E34. Multiple comparison adjusted p-values between decontaminants for Baseline testing method (Malathion in SPE Disks)

Pesticide	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline D7	Summary of Significant Bonferroni-Holm Differences
Malathion	Paint	SPE	Baseline	<MQL	10x Diluted Bleach	N/A	N/A	N/A
Malathion	Paint	SPE	Baseline	<MQL	D7	N/A		

Table E35. Multiple comparison adjusted p-values between decontaminants for Baseline testing method (Malathion on Sealant Coupon Surfaces)

Pesticide	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline D7	Summary of Significant Bonferroni-Holm Differences
Malathion	Sealant	Wipe	Baseline	<MQL	10x Diluted Bleach	N/A	N/A	N/A
Malathion	Sealant	Wipe	Baseline	<MQL	D7	N/A		

Table E36. Multiple comparison adjusted p-values between decontaminants for Baseline testing method (Malathion in Sealant Coupons)

Pesticide	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline D7	Summary of Significant Bonferroni-Holm Differences
Malathion	Sealant	Extracted Coupon	Baseline	1.00	10x Diluted Bleach	A	0.0044	10x Diluted Bleach < D7
Malathion	Sealant	Extracted Coupon	Baseline	4.98	D7	B		

Table E37. Multiple comparison adjusted p-values between decontaminants for Baseline testing method (Malathion in SPE Disks)

Pesticide	Material	Sample Type	Testing Method	Mean Total Mass Recovery (µg)	Decontaminant	Similarity Designation	Baseline	Summary of Significant Bonferroni-Holm Differences
							D7	
Malathion	Sealant	SPE	Baseline	<MQL	10x Diluted Bleach	N/A	N/A	N/A
Malathion	Sealant	SPE	Baseline	<MQL	D7	N/A		

Within-Test Comparisons

Table E38. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (Fipronil on Paint Coupon Surfaces)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
Fipronil	Paint	Wipe	4.16	Baseline	Bleach	0.0007	Bleach < PC
Fipronil	Paint	Wipe	0.057	Baseline	D7	<0.0001	D7 < PC
Fipronil	Paint	Wipe	7.96	Baseline	Positive Control		
Fipronil	Paint	Wipe	0.089	Mod 1	D7	<0.00001	D7 (Mod 1) < PC
Fipronil	Paint	Wipe	0.12	Mod 2	D7	<0.00001	D7 (Mod 2) < PC
Fipronil	Paint	Wipe	7.38	Mod 1 / Mod 2	Positive Control		

Table E39. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (Fipronil in Paint Coupons)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
Fipronil	Paint	Extracted Coupon	2.76	Baseline	Bleach	0.7698	Not significantly different
Fipronil	Paint	Extracted Coupon	0.69	Baseline	D7	0.0004	D7 < PC
Fipronil	Paint	Extracted Coupon	2.84	Baseline	Positive Control		
Fipronil	Paint	Extracted Coupon	0.15	Mod 1	D7	0.0002	D7 (Mod 1) < PC
Fipronil	Paint	Extracted Coupon	0.79	Mod 2	D7	0.0004	D7 (Mod 2) < PC
Fipronil	Paint	Extracted Coupon	3.57	Mod 1/ Mod 2	Positive Control		

Table E40. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (Fipronil in SPE Disks)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
Fipronil	Paint	SPE	<MQL	Baseline	Bleach	N/A	N/A
Fipronil	Paint	SPE	<MQL	Baseline	D7	N/A	N/A
Fipronil	Paint	SPE	<MQL	Baseline	Positive Control		
Fipronil	Paint	SPE	<MQL	Mod 1	D7	N/A	N/A
Fipronil	Paint	SPE	<MQL	Mod 2	D7	N/A	N/A
Fipronil	Paint	SPE	<MQL	Mod 1/ Mod 2	Positive Control		

Table E41. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (Fipronil on Sealant Coupon Surfaces)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
Fipronil	Sealant	Wipe	0.068	Baseline	Bleach	<0.0001	Bleach < PC
Fipronil	Sealant	Wipe	0.83	Baseline	D7	<0.0001	D7 < PC
Fipronil	Sealant	Wipe	9.63	Baseline	Positive Control		
Fipronil	Sealant	Wipe	0.37	Mod 1	D7	<0.0001	D7 (Mod 1) < PC
Fipronil	Sealant	Wipe	1.42	Mod 2	D7	<0.0001	D7 (Mod 2) < PC
Fipronil	Sealant	Wipe	10.05	Mod 1 / Mod 2	Positive Control		

Table E42. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (Fipronil in Sealant Coupons)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
Fipronil	Sealant	Extracted Coupon	0.039	Baseline	Bleach	<0.0001	Bleach < PC
Fipronil	Sealant	Extracted Coupon	0.15	Baseline	D7	<0.0001	D7 < PC
Fipronil	Sealant	Extracted Coupon	1.49	Baseline	Positive Control		
Fipronil	Sealant	Extracted Coupon	0.015	Mod 1	D7	0.0037	D7 (Mod 1) < PC
Fipronil	Sealant	Extracted Coupon	0.52	Mod 2	D7	0.0162	D7 (Mod 2) < PC
Fipronil	Sealant	Extracted Coupon	1.36	Mod 1 / Mod 2	Positive Control		

Table E43. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (Fipronil in SPE Disks)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
Fipronil	Sealant	SPE	<MQL	Baseline	Bleach	N/A	N/A
Fipronil	Sealant	SPE	<MQL	Baseline	D7	N/A	N/A
Fipronil	Sealant	SPE	<MQL	Baseline	Positive Control		
Fipronil	Sealant	SPE	<MQL	Mod 1	D7	N/A	N/A
Fipronil	Sealant	SPE	<MQL	Mod 2	D7	N/A	N/A
Fipronil	Sealant	SPE	<MQL	Mod 1/ Mod 2	Positive Control		

Table E44. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (Malathion on Paint Coupon Surfaces)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
Malathion	Paint	Wipe	1.13	Baseline	Bleach	N/A	N/A
Malathion	Paint	Wipe	<MQL	Baseline	D7	N/A	N/A
Malathion	Paint	Wipe	<MQL	Baseline	Positive Control		
Malathion	Paint	Wipe	<MQL	Mod 1	D7	N/A	N/A
Malathion	Paint	Wipe	<MQL	Mod 2	D7	N/A	N/A
Malathion	Paint	Wipe	<MQL	Mod 1 / Mod 2	Positive Control		

Table E45. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (Malathion in Paint Coupons)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
Malathion	Paint	Extracted Coupon	10.43	Baseline	Bleach	1.0000	Not significantly different
Malathion	Paint	Extracted Coupon	11.72	Baseline	D7	1.0000	Not significantly different
Malathion	Paint	Extracted Coupon	12.40	Baseline	Positive Control		
Malathion	Paint	Extracted Coupon	17.22	Mod 1	D7	0.8772	Not significantly different
Malathion	Paint	Extracted Coupon	14.36	Mod 2	D7	0.8772	Not significantly different
Malathion	Paint	Extracted Coupon	12.90	Mod 1/ Mod 2	Positive Control		

Table E46. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (Malathion in SPE Disks)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
Malathion	Paint	SPE	<MQL	Baseline	Bleach	N/A	N/A
Malathion	Paint	SPE	<MQL	Baseline	D7	N/A	N/A
Malathion	Paint	SPE	<MQL	Baseline	Positive Control		
Malathion	Paint	SPE	<MQL	Mod 1	D7	N/A	N/A
Malathion	Paint	SPE	<MQL	Mod 2	D7	N/A	N/A
Malathion	Paint	SPE	<MQL	Mod 1 / Mod 2	Positive Control		

Table E47. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (Malathion on Sealant Coupon Surfaces)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
Malathion	Sealant	Wipe	1.10	Baseline	Bleach	0.2502	Not significantly different
Malathion	Sealant	Wipe	7.33	Baseline	D7	0.1009	Not significantly different
Malathion	Sealant	Wipe	3.24	Baseline	Positive Control		
Malathion	Sealant	Wipe	6.10	Mod 1	D7	0.5896	Not significantly different
Malathion	Sealant	Wipe	1.33	Mod 2	D7	0.1118	Not significantly different
Malathion	Sealant	Wipe	5.17	Mod 1 / Mod 2	Positive Control		

Table E48. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (Malathion in Sealant Coupons)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
Malathion	Sealant	Extracted Coupon	1.00	Baseline	Bleach	0.6890	Not significantly different
Malathion	Sealant	Extracted Coupon	4.98	Baseline	D7	<0.0001	PC < Baseline
Malathion	Sealant	Extracted Coupon	1.07	Baseline	Positive Control		
Malathion	Sealant	Extracted Coupon	7.29	Mod 1	D7	1.0000	Not significantly different
Malathion	Sealant	Extracted Coupon	9.66	Mod 2	D7	1.0000	Not significantly different
Malathion	Sealant	Extracted Coupon	7.60	Mod 1/ Mod 2	Positive Control		

Table E49. Multiple comparison adjusted p-values between decontaminants and positive controls with testing methods (Malathion in SPE Disks)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Decontaminant	Comparison vs. Positive Control (PC)	Test Direction
Malathion	Sealant	SPE	<MQL	Baseline	Bleach	N/A	N/A
Malathion	Sealant	SPE	<MQL	Baseline	D7	N/A	N/A
Malathion	Sealant	SPE	<MQL	Baseline	Positive Control		
Malathion	Sealant	SPE	<MQL	Mod 1	D7	N/A	N/A
Malathion	Sealant	SPE	<MQL	Mod 2	D7	N/A	N/A
Malathion	Sealant	SPE	<MQL	Mod 1/ Mod 2	Positive Control		

Across-Test Method Comparisons

Table E50. Multiple comparison adjusted p-values between testing methods for positive controls (Fipronil on Paint Coupon Surfaces)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1/ Mod 2	Summary of Significant Bonferroni-Holm Differences
Fipronil	Paint	Wipe	7.96	Baseline	0.9113	0.9113	No significant differences
Fipronil	Paint	Wipe	7.24	Fate & Transport		0.9113	
Fipronil	Paint	Wipe	7.38	Mod 1 / Mod 2			

Table E51. Multiple comparison adjusted p-values between testing methods for positive controls (Fipronil in Paint Coupons)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate & Transport	Mod 1 / Mod 2	Summary of Significant Bonferroni-Holm Differences
Fipronil	Paint	Extracted Coupon	2.84	Baseline	0.0181	0.1573	Baseline < Fate and Transport
Fipronil	Paint	Extracted Coupon	4.71	Fate and Transport		0.0892	
Fipronil	Paint	Extracted Coupon	3.57	Mod 1 / Mod 2			

Table E52. Multiple comparison adjusted p-values between testing methods for positive controls (Fipronil in SPE Disks)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1 / Mod 2	Summary of Significant Bonferroni-Holm Differences
Fipronil	Paint	SPE	<MQL	Baseline	N/A	N/A	N/A
Fipronil	Paint	SPE	<MQL	Fate and Transport		N/A	
Fipronil	Paint	SPE	<MQL	Mod 1/Mod 2			

Table E53. Multiple comparison adjusted p-values between testing methods for positive controls (Fipronil on Sealant Coupon Surfaces)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1 / Mod 2	Summary of Significant Bonferroni-Holm Differences
Fipronil	Sealant	Wipe	9.63	Baseline	0.5422	0.4212	No significant differences
Fipronil	Sealant	Wipe	9.43	Fate and Transport		0.2596	
Fipronil	Sealant	Wipe	10.05	Mod 1/Mod 2			

Table E54. Multiple comparison adjusted p-values between testing methods for positive controls (Fipronil in Sealant Coupons)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1/ Mod 2	Summary of Significant Bonferroni-Holm Differences
Fipronil	Sealant	Extracted Coupon	1.49	Baseline	0.0671	0.6549	No significant differences
Fipronil	Sealant	Extracted Coupon	2.27	Fate and Transport		0.0548	
Fipronil	Sealant	Extracted Coupon	1.36	Mod 1 / Mod 2			

Table E55. Multiple comparison adjusted p-values between testing methods for positive controls (Fipronil in SPE Disks)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1/ Mod 2	Summary of Significant Bonferroni-Holm Differences
Fipronil	Sealant	SPE	<MQL	Baseline	N/A	N/A	N/A
Fipronil	Sealant	SPE	<MQL	Fate and Transport		N/A	
Fipronil	Sealant	SPE	<MQL	Mod 1/Mod 2			

Table E56. Multiple comparison adjusted p-values between testing methods for positive controls (Malathion on Paint Coupon Surfaces)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1/ Mod 2	Summary of Significant Bonferroni-Holm Differences
Malathion	Paint	Wipe	<MQL	Baseline	0.0450	1.0000	Baseline < Fate Modifications < Fate
Malathion	Paint	Wipe	1.62	Fate and Transport		0.0450	
Malathion	Paint	Wipe	<MQL	Mod 1 / Mod 2			

Table E57. Multiple comparison adjusted p-values between testing methods for positive controls (Malathion in Paint Coupons)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1 / Mod 2	Summary of Significant Bonferroni-Holm Differences
Malathion	Paint	Extracted Coupon	12.40	Baseline	1.0000	1.0000	No significant differences
Malathion	Paint	Extracted Coupon	17.89	Fate and Transport		1.0000	
Malathion	Paint	Extracted Coupon	12.90	Mod 1/Mod 2			

Table E58. Multiple comparison adjusted p-values between testing methods for positive controls (Malathion in SPE Disks)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1/ Mod 2	Summary of Significant Bonferroni-Holm Differences
Malathion	Paint	SPE	<MQL	Baseline	N/A	N/A	N/A
Malathion	Paint	SPE	<MQL	Fate and Transport		N/A	
Malathion	Paint	SPE	<MQL	Mod 1/Mod 2			

Table E59. Multiple comparison adjusted p-values between testing methods for positive controls (Malathion on Sealant Coupon Surfaces)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1 / Mod 2	Summary of Significant Bonferroni-Holm Differences
Malathion	Sealant	Wipe	3.24	Baseline	0.8333	0.7271	No significant differences
Malathion	Sealant	Wipe	2.84	Fate and Transport		0.7271	
Malathion	Sealant	Wipe	5.17	Mod 1 / Mod 2			

Table E60. Multiple comparison adjusted p-values between testing methods for positive controls (Malathion in Sealant Coupons)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1/ Mod 2	Summary of Significant Bonferroni-Holm Differences
Malathion	Sealant	Extracted Coupon	1.07	Baseline	0.1767	0.1962	No significant differences
Malathion	Sealant	Extracted Coupon	8.83	Fate and Transport		0.7242	
Malathion	Sealant	Extracted Coupon	7.60	Mod 1 / Mod 2			

Table E61. Multiple comparison adjusted p-values between testing methods for positive controls (Malathion in SPE Disks)

Pesticide	Material	Sample Type	Mean Total Mass Recovery (µg)	Testing Method	Fate and Transport	Mod 1 / Mod 2	Summary of Significant Bonferroni-Holm Differences
Malathion	Sealant	SPE	<MQL	Baseline	N/A	N/A	N/A
Malathion	Sealant	SPE	<MQL	Fate and Transport		N/A	
Malathion	Sealant	SPE	<MQL	Mod 1/Mod 2			

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