United States
Environmental Protection
Agency

EPA/600/R-19/075 | June 2019
www.epa.gov/homeland-security-research

Efficacy and Compatibility of Decontamination Options for Sensitive Equipment-Related Materials Contaminated with Persistent Chemical Warfare Agents

Office of Research and Development
Homeland Security Research Program

This page left intentionally blank

Efficacy and Compatibility of Decontamination Options for Sensitive Equipment-Related Materials Contaminated with Persistent Chemical Warfare Agents

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

DISCLAIMER

The U.S. Environmental Protection Agency (EPA) through its Office of Research and Development funded and managed the research described herein under Contract Number EP-C-15-002, Task Order 0015 with Battelle. It has been subjected to the Agency's review and has been approved for publication. Note that approval does not signify that the contents necessarily reflect the views of the Agency. Any mention of trade names, products, or services does not imply an endorsement by the U.S. Government or EPA. The EPA does not endorse any commercial products, services, or enterprises. The contractor role did not include establishing Agency policy.

Questions concerning this document or its application should be addressed to:

Lukas Oudejans, Ph.D.
National Homeland Security Research Center
Office of Research and Development
U.S. Environmental Protection Agency (MD-E343-06)
109 T.W. Alexander Drive
Research Triangle Park, NC 27711
Phone: 919-541-2973
Fax: 919-541-0496
E-mail: Oudejans.Lukas@epa.gov

ACKNOWLEDGMENTS

Contributions of the following individuals and organization to this report are gratefully acknowledged:

U.S. Environmental Protection Agency (EPA) Project Team

Lukas Oudejans (Principal Investigator, Office of Research and Development, National Homeland Security Research Center (ORD/NHSRC)
Catherine Young (Region 1)
Charlie Fitzsimmons (Region 3)
Lawrence [Larry] Kaelin (Office of Land and Emergency Management, Office of Emergency Management, Consequence Management Advisory Division (OLEM/OEM/CMAD)
Shannon Serre (OLEM/OEM/CMAD)
Elise Jakabhazy (OLEM/OEM/CMAD)
Timothy Boe (ORD/NHSRC)
Paul Lemieux (ORD/NHSRC)

US EPA Technical Reviewers of Report

Leroy Mickelsen (OLEM/OEM/CMAD)
Stuart Willison (ORD/NHSRC)

US EPA Quality Assurance

Eletha Brady-Roberts

Battelle

Carissa Dodds
William Hayes
Amy Andrews
Anthony Ellingson
David See

The National Caucus and Center on Black Aging, Inc.

Joan Bursey (Technical Editing and Quality Assurance Review)

EXECUTIVE SUMMARY

Under U.S. Environmental Protection Agency's Homeland Security Research Program (US EPA HSRP), the National Homeland Security Research Center conducts research necessary to identify methods and technologies that can be used for the decontamination of equipment and surfaces contaminated with chemical warfare agents. Typical decontamination approaches such as those using the chlorine oxidation approach can be efficacious in decontamination of chemical warfare agents (CWAs) but can also often be destructive and leave items or surfaces to which the approaches are applied damaged or deteriorated. Approaches for decontamination of CWAs from sensitive equipment (SE) items such as computer server systems or other electronic equipment have thus been identified as a critical knowledge gap, as SE is typically associated with high procurement costs and long lead times, and the integrity and usability of the equipment must be preserved following decontamination.

This project focused on the evaluation of selected technologies for their efficacy and compatibility in decontamination of persistent CWAs from SE-related materials. Decontaminants that were anticipated to be simultaneously efficacious and material-compatible were first identified via searches of existing literature and secondary data. From the technologies identified during the literature searches, three were selected for inclusion during decontamination efficacy testing during this work: Dahlgren Decon from First Line Technology, EasyDECON DF200 from Intelagard, and the Handheld Decontamination Apparatus (HDA), which used electrochemically-generated chlorine dioxide ($eClO_2$) as the active decontaminant, by TDA Research, Inc. Decontamination of O-ethyl S-(2-[diisopropylamino]ethyl) methylphosphonothioate (VX) and sulfur mustard (bis(2-chloroethyl) sulfide, HD) from the SE-related materials acrylonitrile butadiene styrene (ABS) molded plastic, acrylic, and aluminum was evaluated. These materials were selected because they are frequently included in construction of SE.

Based on measured efficacy values, Dahlgren Decon demonstrated the highest efficacy for decontamination of VX from all three SE-related material types, achieving 99% efficacy on ABS plastic and acrylic and 98% on aluminum. DF200 demonstrated similarly high efficacy in decontaminating VX from acrylic (98%) and aluminum (97%), though DF200 decontamination of VX from ABS plastic was slightly lower at 83%. A statistical comparison (Student's t-test) showed that the recovered VX amounts for Dahlgren Decon and DF200 for the ABS Plastic and aluminum were not significantly different ($p<0.05$). The TDA $eClO_2$ decontaminant demonstrated the lowest VX decontamination efficacies, measuring 30% from aluminum, 29% from acrylic, and only 4.4% from ABS plastic.

Conversely, $eClO_2$ demonstrated generally higher efficacies for decontamination of HD compared to Dahlgren Decon and DF200. Efficacy of $eClO_2$ against HD was 37% from ABS plastic, 39% from aluminum, and 61% from acrylic (which was also the highest HD decontamination efficacy measured during this work). Dahlgren Decon measured 54% HD decontamination efficacy from aluminum, 53% from acrylic, and 38% from ABS plastic while DF200 demonstrated only 29% efficacy against HD on ABS plastic, 19% on acrylic, and 9.4% on aluminum. A statistical comparison showed that recovered amounts on ABS plastic and

acrylic were not significantly different among all three decontaminants. Figure ES-1 summarizes the average percent decontamination efficacy measured for each decontaminant for VX and HD from the surface of each of the three SE-related material types included during testing.

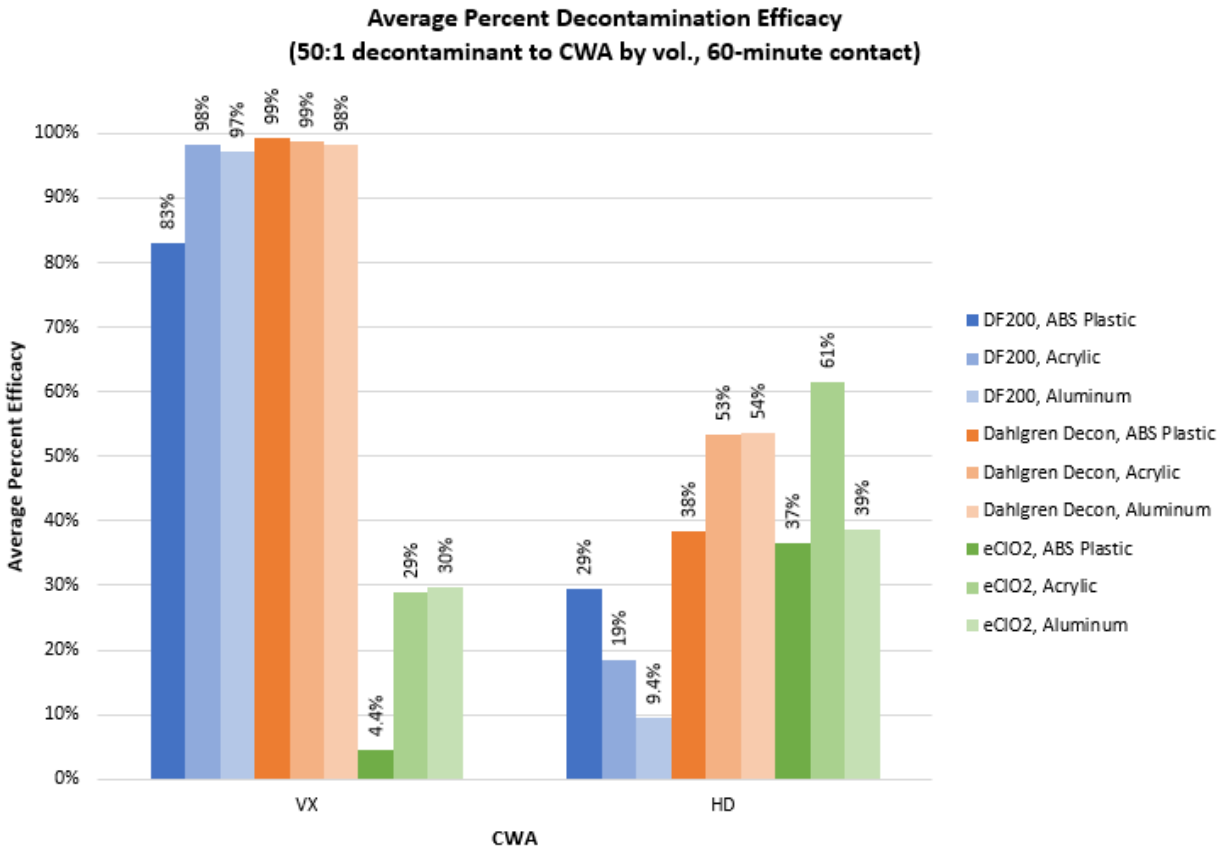


Figure ES-1. Average Percent Decontamination Efficacy by CWA/Decontaminant/Material

In summary, all three decontaminants evaluated demonstrated some degree of efficacy for decontamination of both VX and HD from all three SE-related materials. Generally, Dahlgren Decon and DF200 were much more efficacious in decontamination of VX than of HD. Conversely, $eClO_2$ demonstrated greater efficacy in decontamination of HD than in decontamination of VX.

With regard to VX and HD degradation/decontamination byproducts, the relatively toxic mustard sulfone was detected in several samples, including four of five wipe sample extracts taken from HD-contaminated aluminum coupons decontaminated with Dahlgren Decon, and all wipe extracts taken from all three SE-related materials decontaminated with the $eClO_2$ decontaminant. Mustard sulfone was also detected in extracts of HD-contaminated ABS plastic and acrylic coupons decontaminated with the $eClO_2$ decontaminant. The toxic byproduct of VX degradation, EA-2192, cannot be identified by GC/MS. Analysis for EA-2192 requires the use of liquid

chromatography (LC)/MS, which was outside the scope of this testing. Thus, degradation of VX into EA-2192 was not evaluated during this work.

Generally, Dahlgren Decon appeared to demonstrate the highest degree of compatibility with the three SE-related materials included in this evaluation. Residual decontaminant was easily wiped from the surface of all three material types, leaving no lasting observable effects on acrylic and only very slight discoloration of ABS plastic and aluminum. In contrast, DF200 and $eClO_2$ discolored ABS plastic to a greater degree and left residues on aluminum that were not easily removed. Actual physical damage to/deterioration of the aluminum coupon surface was observed after contact with $eClO_2$. This suggests that the use of $eClO_2$ may not be suitable for decontamination of sensitive equipment including electronic equipment.

Impact of the Study:

Based on the results obtained from this study, VX and to a lesser degree HD can be neutralized using the Dahlgren Decon or DF200 decontamination products while maintaining a material compatibility. Caution should be used in extrapolating from bench testing to field application of these decontamination solutions. Measurable amounts of VX and HD were found to remain on the surface following any of the decontamination solution applications. Hence, additional decontamination may be required to further degrade the residual agent to reach a clearance level. Such may be accomplished through an extended dwell time beyond 1 h or a reapplication of the decontaminant. Neither approach was part of the test matrix and was not investigated as part of this study. Decontamination research studies need to consider the quenching of the residual decontamination reaction at the end of the intended decontamination contact time. Incomplete quenching will result in a low bias in recovered agent from wipes and/or extracted materials, resulting in a high bias in efficacy values.

TABLE OF CONTENTS

DISCLAIMER II
ACKNOWLEDGMENTS III
EXECUTIVE SUMMARY IV
TABLE OF CONTENTS VII
LIST OF TABLES IX
LIST OF FIGURES X
LIST OF ACRONYMS XI
1. INTRODUCTION 1
1.1 Purpose 1
1.2 Project Objectives 1
1.3 Test Facility Description 2
2. EXPERIMENTAL METHODS 3
2.1 Experimental Design 3
2.1.1 Method Demonstration 3
2.1.2 Decontamination Efficacy 6
2.1.3 Material Compatibility 6
2.2 Test Matrices 7
2.2.1 Method Demonstration Test Matrices 7
2.2.2 Decontamination Efficacy Test Matrix 9
2.3 Experimental Methods and Materials 10
2.3.1 Coupon Materials 11
2.3.2 CWA Application 13
2.3.3 Decontamination Technologies 14
2.3.4 Coupon Surface (Wipe) Sampling 17
2.3.5 Coupon and Wipe Solvent Extraction 18
2.4 Analytical Methods 18
2.4.1 VX and HD Quantitative Analysis 18
2.4.2 VX Byproduct Qualitative Analysis 19
2.4.3 HD Byproduct Qualitative Analysis 20
2.5 Calculations 21
2.6 Analysis of Variance 22
3. RESULTS 23
3.1 Method Demonstration 23
3.1.1 Wipe Sampling and Solvent Extraction Method Development Results 23
3.1.2 Quench Method Development Test Results 27
3.2 Decontamination Efficacy 35
3.2.1 VX Residual Contamination 35
3.2.2 HD Residual Contamination 38
3.2.3 VX and HD Decontamination Efficacy 42

3.3 ANOVA Results 44
3.3.1 Comparison of Positive Control Results 44
3.3.2 Comparison of Test Sample Results 46
3.4 Material Compatibility 47
4. QUALITY ASSURANCE/QUALITY CONTROL 55
4.1 Control of Monitoring and Measuring Devices 55
4.2 Equipment Calibrations 55
4.2.1 Calibration Procedures and Schedules 55
4.2.2 GC/MS Calibration 56
4.3 Technical Systems Audit 57
4.4 Performance Evaluation Audit 57
4.5 Data Quality Audit 58
5. SUMMARY 59
6. REFERENCES 63

LIST OF TABLES

Table 1. Surface (Wipe) Sampling and Solvent Extraction Method Demonstration Tests 8
Table 2. Quench Method Demonstration Tests 9
Table 3. Decontamination Efficacy Test Matrix..... 9
Table 4. SE-Related Materials 13
Table 5. CWA Purity 13
Table 6. Decontamination Technology Application Volumes and Contact Periods 17
Table 7. GC/MS Conditions for VX and HD Analysis 19
Table 8. Wipe Sampling and Coupon Solvent Extraction Average Mass Recoveries 24
Table 9. Wipe Sampling and Coupon Solvent Extraction Percent Recoveries 24
Table 10. Quench Method Test 1, Solvent Extraction Alone, Average Mass Recoveries 28
Table 11. Quench Method Test 1, Solvent Extraction Alone, Percent Recoveries 28
Table 12. Quench Method Test 2, 3M STS, Average Mass Recoveries 30
Table 13. Quench Method Test 2, 3M STS, Percent Recoveries 31
Table 14. Quench Sample Reanalyses Percent Differences, 1st Test..... 34
Table 15. Quench Sample Reanalyses Percent Differences, 2nd Test..... 34
Table 16. VX Frequency of Detection..... 35
Table 17. VX Average Recoveries and Residual Contamination..... 36
Table 18. HD Frequency of Detection..... 38
Table 19. Mustard Sulfone Detection 39
Table 20. HD Average Recoveries and Residual Contamination..... 40
Table 21. Average Percent Decontamination Efficacies 42
Table 22. ANOVA Results for ABS Plastic with HD (Positives)..... 45
Table 23. ANOVA Results for Acrylic with HD (Positives) 45
Table 24. ANOVA Results for Aluminum with HD (Positives) 45
Table 25. ANOVA Results for ABS Plastic with VX (Positives)..... 45
Table 26. ANOVA Results for Acrylic with VX (Positives) 45
Table 27. ANOVA Results for Aluminum with VX (Positives) 45
Table 28. ANOVA Results for ABS Plastic with HD (Test Samples) 46
Table 29. ANOVA Results for Acrylic with HD (Test Samples)..... 46
Table 30. ANOVA Results for Aluminum with HD (Test Samples) 46
Table 31. ANOVA Results for ABS Plastic with VX (Test Samples) 47
Table 32. ANOVA Results for Acrylic with VX (Test Samples)..... 47
Table 33. ANOVA Results for Aluminum with VX (Test Samples) 47
Table 34. Dahlgren Decon Material Compatibility 49
Table 35. EasyDECON DF200 Material Compatibility..... 50
Table 36. TDA Research HDA $eClO_2$ Material Compatibility 51
Table 37. Quality Control Requirements and Results 55
Table 38. Equipment Calibration Schedule 56
Table 39. GC Performance Parameters and Acceptance Criteria..... 57
Table 40. Performance Evaluation Audit Results..... 58
Table 41. Average Residual Contamination Summary..... 60

LIST OF FIGURES

Figure ES-1. Average Percent Decontamination Efficacy by CWA/Decontaminant/Material v
Figure 1. Wipe Sampling and Coupon Solvent Extraction, VX Mass Recovery 25
Figure 2. Wipe Sampling and Coupon Solvent Extraction, HD Mass Recovery 26
Figure 3. Quench Method Test 1, Solvent Extraction Alone, Average Mass Recoveries 29
Figure 4. Quench Method Test 2, 3M STS, Average Mass Recoveries 32
Figure 5. Average Residual VX Contamination 37
Figure 6. Average Residual HD Contamination 41
Figure 7. Average VX Decontamination Efficacy 42
Figure 8. Average HD Decontamination Efficacy 43
Figure 9. Damage to ABS plastic caused by HD (1). 52
Figure 10. Damage to ABS plastic caused by HD (2). 53
Figure 11. Damage to ABS plastic caused by HD (3). 53
Figure 12. Damage to ABS plastic caused by HD (4). 54
Figure 13. ABS plastic debris on wipe sample. 54
Figure 14. Average Percent Decontamination Efficacy by CWA/Decontaminant/Material 61

ATTACHMENTS

Attachment A – Environmental Data

LIST OF ACRONYMS

ABS	acrylonitrile butadiene styrene
amu	atomic mass unit(s)
ANOVA	Analysis of Variance
°C	degree(s) Celsius
CAS	Chemical Abstracts Services
CI	critical infrastructure
cm	centimeter(s)
cm^2	square centimeter(s)
CoV	coefficient of variation
CCV	continuing calibration verification
CWA	chemical warfare agent
DFTPP	decafluorotriphenylphosphine
DIC	N,N'-diisopropylcarbodiimide
$eClO_2$	electrochemically-generated chlorine dioxide
EMPA	ethyl methylphosphonic acid
EPA	U.S. Environmental Protection Agency
°F	degree(s) Fahrenheit
GC/MS	gas chromatography/mass spectrometry
HD	sulfur mustard, bis(2-chloroethyl) sulfide
HDA	Handheld Decontamination Apparatus
HMRC	Hazardous Materials Research Center
HSRP	Homeland Security Research Program
IPA	isopropyl alcohol
IS	internal standard
L	liter(s)
LC	liquid chromatography
M	molar
mg	milligram(s)
mL	milliliter(s)
mm	millimeter(s)
MQL	minimum quantifiable limit

NHSRC	National Homeland Security Research Center
PE	Performance Evaluation
PMMA	poly(methyl methacrylate)
QA	quality assurance
r^2	coefficient of determination
RH	relative humidity
RSD	relative standard deviation
SE	sensitive equipment
SS	stainless steel
STS	sodium thiosulfate
TDG	thiodiglycol
TSA	Technical Systems Audit
µL	microliter(s)
VX	O-ethyl S-[2-(diisopropylamino)-ethyl] methylphosphonothioate

1. INTRODUCTION

The U.S. Environmental Protection Agency (EPA) is responsible for preparing for, responding to, and recovering from threats to public health, welfare, or the environment caused by actual or potential hazardous materials incidents. Hazardous materials include chemical, biological, and radiological substances, whether accidentally or intentionally released. The threat and potential impact of a chemical warfare agent (CWA) release is driving EPA's Homeland Security Research Program (HSRP) to systematically evaluate potential decontamination technologies for CWAs.

In the event of either an accidental or intentional release of CWAs, or as the result of use during response to a CWA incident, sensitive equipment (SE) that may be part of critical infrastructure (CI) can become contaminated by the CWA. CI is essential in support of the response and recovery following such release and the decontamination of CI would be of a high priority. Meanwhile, the procurement of SE is often associated with high costs and/or long lead times. Hence, the approach to decontamination of SE has the additional requirement that the decontamination process does not impact the function of the SE. A decontaminant may degrade the exterior or housing of the equipment or deter the functionality of the equipment. The intent of the SE decontamination would be to retain it for future use.

Traditional decontaminants such as bleach products using the chlorine oxidation approach are known to be corrosive and would impact the functionality of electronic equipment and similar items. Alternative decontaminants against CWAs exist that have been developed in recent years with the intended purpose of being more material-compatible. The efficacy of these newly-developed decontamination technologies against CWAs on SE surfaces is relatively unknown. Additionally, existing decontamination technologies with demonstrated efficacy against CWAs that have not previously been evaluated for use with SE surfaces may demonstrate material compatibility. EPA responders have identified this high-priority knowledge gap for the HSRP to address.

1.1 Purpose

This project focused on the evaluation of selected technologies for their efficacy and compatibility in decontamination of persistent CWAs from SE-related materials.

1.2 Project Objectives

The primary objective of this project was to quantitatively evaluate the efficacy of candidate decontamination technologies to decontaminate CWAs from select SE-related materials through performance of bench-scale laboratory studies using neat O-ethyl S-(2-[diisopropylamino]ethyl) methylphosphonothioate (VX, Chemical Abstracts Services (CAS) 50782-69-9) and sulfur mustard (bis(2-chloroethyl) sulfide, HD, CAS 505-60-2) and the SE-related materials acrylonitrile butadiene styrene (ABS) molded plastic, acrylic, and aluminum. These materials were selected because they are frequently included in construction of SE (refer to Section 2.3.1). VX and HD were selected

as two of the more persistent CWAs. Decontaminants that were anticipated to be simultaneously efficacious and material-compatible were first identified via searches of existing literature and secondary data. From the technologies identified during the literature searches, three were selected for inclusion during decontamination efficacy testing during this work: Dahlgren Decon from First Line Technology, EasyDECON DF200 from Intelagard, and the Handheld Decontamination Apparatus (HDA) by TDA Research, Inc.

Additionally, during the decontamination efficacy evaluation, compatibility of the decontamination technologies/methodologies with the SE-related materials were evaluated qualitatively. This evaluation included visual assessment and documentation of any visible deterioration or damage caused to the materials by application of the decontamination technology or methodology.

1.3 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 material through its Bailment Agreement W911SR-10-H-0001 with the U.S. Department of the Army. Wherever applicable and required, the reporting requirements for this Bailment Agreement were followed.

2. EXPERIMENTAL METHODS

2.1 Experimental Design

Generally, multiple coupons (small representative samples; see Section 2.3.1) of SE-related materials were contaminated with neat VX or HD. After a 60-minute CWA dwell period, one of three candidate decontaminants was applied to the surface of the coupons over the CWA-contaminated area at a decontaminant:CWA ratio of 50:1 by volume. The applied decontaminants were allowed to react with the CWA on the surface of the coupons for a predetermined period. Following the decontamination period (one hour), coupons were sampled via surface wiping and subsequent extraction in solvent (both wipe and coupon separately). Wipe and coupon extracts were then analyzed via gas chromatography/mass spectrometry (GC/MS) to quantify residual CWA contamination and assess the efficacy of the decontaminants.

Prior to decontamination efficacy testing, the experimental methods planned for use were demonstrated to ensure valid data were generated. Concurrently with decontamination efficacy testing, compatibility of the decontaminants with the SE-related materials to which they were applied was assessed qualitatively through visual inspection of decontaminated and not decontaminated coupons.

The experimental designs for each of these phases of testing, including method demonstration, decontamination efficacy testing, and decontaminant/materials compatibility evaluation, are described in the following subsections.

2.1.1 Method Demonstration

2.1.1.1. *Surface (Wipe) Sampling and Solvent Extraction of VX and HD from SE-Related Materials*

The methods for wipe-sampling of coupons and for solvent extraction of wipes and coupons developed during previous CWA/material interaction studies [1] were evaluated for use during the project using VX and HD and the SE-related materials selected for evaluation to ensure sufficient recovery of CWA would be achieved from the materials.

The wipe sampling and solvent extraction methods were evaluated concurrently through the execution of method demonstration tests that incorporated both methods. During each test, 2 microliters (µL) of neat VX or HD was applied as a liquid challenge (spiked) onto designated coupons as described in Section 2.3.2 and allowed to remain undisturbed during a 60-min dwell period. Following the dwell period, coupons were either wipe-sampled (Section 2.3.4) and then extracted in solvent (Section 2.3.5) or extracted in solvent alone without prior wipe sampling. Wipe and coupon extracts were then analyzed for VX or HD via GC/MS (see Section 2.4).

Specific procedures and materials used for wipe sampling, including the specific wipe type that was used, are described in Section 2.3.4. The wipe sampling method that was evaluated for use during the project includes the following details:

- 95% *n*-hexane (H306-SK4, Fisher Scientific, Pittsburgh, PA, hereafter "hexane") was evaluated as the wipe wetting and extraction solvent.
- Wipes were wetted with 1.5 milliliters (mL) of hexane. This volume of hexane added to the wipe was found during previous work [1] to be an amount that is approximately half-saturating for the wipe, as determined gravimetrically by weighing three wipes before and after soaking the wipes in hexane (half of the amount of solvent remaining on the wipe 30 seconds after immersion in solvent and hanging vertically to allow excess solvent to drip off).

The procedure and materials used for solvent extraction of wipes and coupons are described in Section 2.3.5. As with wipe wetting and extraction, hexane was evaluated as the coupon extraction solvent for all three SE-related material types.

The experimental methods were deemed acceptable for use in the subsequent decontamination efficacy evaluation if the mean total recoveries from all SE-related materials were within the range of 70% to 120% of the mean of the stainless steel (SS, refer to Section 2.2.1.1) evaporation controls with a coefficient of variation (CoV) between replicates of less than 30%. Total recoveries equaled the sum of the wipe and coupon extraction recoveries for samples that were wiped, or coupon extraction recoveries alone for samples that were not wiped. Refer to Section 3.1.1 for results from the wipe sampling and coupon solvent extraction tests.

2.1.1.2. *Decontamination Technology Neutralization (Quench)*

During decontamination efficacy testing, the decontamination reaction had to be stopped at the end of a specified contact period to determine how much decontamination occurred during the period. Adequate decontaminant neutralization (quench) methods were required for each of the three decontamination technologies and had to be effective for both VX and HD on the surface of all three SE-related materials. Ultimately, two quench methods were evaluated and used during decontamination efficacy testing to halt the decontamination reactions and allow for assessment of decontamination efficacy as a function of decontaminant contact time: (1) extraction in hexane alone, and (2) addition of a 3 molar (M) sodium thiosulfate ($Na_2S_2O_3$, STS) solution to the wipe/coupon extraction solvent. The method used was dependent on the CWA/decontaminant combination. Refer to Section 3.1.2 for quench method development test results and discussion.

The adequacy of the quench methods was demonstrated by post-spiking the extracts of procedural blanks with dilute solutions of VX and HD. Aluminum (see Section 2.3.1) procedural blanks were generated using each of the three decontamination technologies by applying 100 µL of decontaminant to unspiked coupons and allowing the decontaminant to remain undisturbed on the coupons for the required decontamination contact period (see Section 2.3.3.4). Following the contact period, the coupons (with the decontaminant remaining on the coupon surface) were wipe sampled using the method selected for testing (see Sections 2.1.1.1 and 2.3.4), and wipes and coupons were extracted in solvent as described in Section 2.3.5. During tests evaluating the

use of 3M STS as a quench method, 15 mL of the STS solution was added to the extraction solvent in the jar prior to addition of the wipes or coupons. Additional aluminum procedural blanks were generated as described above, but no decontaminant was applied to the coupons (coupons were wipe sampled as described in Section 2.3.4, with wipes and coupons extracted in solvent as described in Section 2.3.5; no decontaminant was applied to the coupons prior to sampling).

Following extraction, select wipe and coupon extracts were spiked with a dilute solution of VX or HD in hexane such that the final CWA concentration fell at approximately the mid-point of the "low curve" that was used for GC/MS analysis. During the first test evaluating solvent extraction alone as a quench method, extracts were post-spiked with either:

- 155 µL of a dilute VX solution at 863 µg/mL (134 µg spiked), yielding a coupon extract concentration of 5.3 µg/mL and a wipe extract concentration of 5.0 µg/mL.
- 70 µL of a dilute HD solution at 1903 µg/mL (133 µg spiked), yielding a coupon extract concentration of 5.3 µg/mL and a wipe extract concentration of 5.0 µg/mL.

During the second test evaluating 3M STS as a quench method, extracts were post-spiked with either:

- 153 µL of a dilute VX solution at 873 µg/mL (134 µg spiked), yielding a coupon extract concentration of 5.3 µg/mL and a wipe extract concentration of 5.0 µg/mL.
- 70 µL of a dilute HD solution at 1903 µg/mL (133 µg spiked), yielding a coupon extract concentration of 5.3 µg/mL and a wipe extract concentration of 5.0 µg/mL.

For those extracts also containing 3M STS as a quench agent, the dilute solution of VX or HD was added to the extraction solvent (top) layer.

Post-spiked extracts were vortexed for 10 seconds and allowed to stand for one hour. Following the one-hour stand, an aliquot of each extract was taken and immediately analyzed (no later than the same business day that the samples were generated) via GC/MS to evaluate whether decontamination of the post-spiked CWA had occurred. For those extracts also containing 3M STS as a quench agent, the aliquot was taken from the extraction solvent (top) layer.

Immediately following the initial analysis, the GC vials containing the extract samples were recapped (with new, unpierced septa) and stored at -20 ± 10 degrees Celsius (°C). Three days following generation of the samples, the samples were retrieved from storage and analyzed again via GC/MS to evaluate whether decontamination and/or degradation of the post-spiked CWA had occurred. Samples were allowed to equilibrate to room temperature prior to the second analysis.

Hexane was used for wipe and coupon extraction for the quench method evaluations. Quench methods were considered sufficient if the amounts of VX and HD recovered from post-spiked

extracts containing decontaminant were each at least 70% of the mean amount of CWA recovered from post-spiked extracts that did not contain decontaminant.

2.1.2 Decontamination Efficacy

A post-test only control group experimental design was used for the decontamination efficacy evaluation. Decontamination was the experimental variable. Test coupons were contaminated, decontaminated, sampled, and analyzed for VX or HD. Positive control coupons were contaminated but not decontaminated, and subsequently sampled and analyzed for VX or HD along with the test coupons. The effect of decontamination (efficacy) was defined as the percentage of CWA remaining on the test coupons compared to the positive control coupons (refer to Section 2.5). The higher the efficacy, the greater the effect of decontamination by the specific technology.

Procedurally, 2 µL of neat VX or HD was spiked onto the center of each test coupon (five replicates) and positive control coupon (three replicates) as described in Section 2.3.2. The spiked coupons were allowed to remain undisturbed during a set CWA dwell period of 60 minutes. Following the CWA dwell period, 100 µL of the decontamination technology under test was applied as a liquid directly to the CWA challenge on each test coupon (applied on top of the liquid CWA droplet on the coupon surface) and allowed to remain in contact with the CWA on the coupon surface for a set decontamination period of 60 minutes. Decontamination technology application procedures as well as specific application volumes and decontaminant contact periods that were used for each technology are provided in Section 2.3.3. Following the decontamination period, the test and positive control coupons were sampled for residual CWA via wipe-sampling according to Section 2.3.4 and solvent extraction according to Section 2.3.5. Wipe and coupon extracts were analyzed for VX or HD via GC/MS according to Section 2.4.

2.1.3 Material Compatibility

The effect of the decontamination technologies on the test coupons was evaluated qualitatively during decontamination efficacy testing. During decontamination and following wipe sampling, test coupons and procedural blanks were visually inspected and compared to other coupons of the same SE-related material types that were not exposed to the decontamination technologies. Comparison of the test coupons and procedural blanks to coupons to which no decontaminant was applied allowed for assessment of damage to the coupons from application of the decontamination technologies. Any obvious changes (any corrosion, deterioration, damage, or any other effect) on the appearance of the coupons, for example in the color, reflectivity, or apparent roughness of the coupon surfaces, were documented. Representative photographs were taken to document any visually-obvious changes that occurred. Additionally, positive controls were compared to other unspiked coupons (procedural and laboratory blanks). Comparison of the positive controls, following wiping, to blank samples allowed for assessment of damage to the SE-related materials from application of the CWA.

Additionally, a second procedural blank for each decontamination technology/material type combination was generated that was not wiped or extracted alongside the test coupons following the decontamination period (see the footnote included in Table 3). Rather, the decontaminant was allowed to remain undisturbed on the surface of the coupon in a covered Petri dish for a period of one week. Photographs of the additional procedural blanks were taken at one day and one week following application of the decontaminants to assess the effects of extended contact between the decontaminants and the materials. For any residue remaining on the surface of the materials following one week (following evaporation of the liquid decontamination technologies), the effort required to wipe the residue from the material surface and the extent to which the residue could be removed were investigated.

2.2 Test Matrices

2.2.1 Method Demonstration Test Matrices

2.2.1.1. *Wipe Sampling and Coupon Solvent Extraction Test Matrix*

Two method demonstration tests were conducted to evaluate the surface wipe sampling and coupon solvent extraction methods. Each test included all three SE-related materials selected for evaluation during the project and a single CWA. In addition to the test coupons, three wipe sampling procedural blanks and a single laboratory blank per material type were included. The procedural and laboratory blanks consisted of coupons of the same SE-related material type and dimensions as the associated test coupons. Each blank type is further described as follows:

- Procedural Blanks - SE-related material coupons that were not spiked but that were wipe-sampled and extracted in solvent alongside the test coupons.
- Laboratory Blanks - SE-related material coupons that were neither spiked nor wipe-sampled; the coupons were maintained outside the test hood until placed into extraction solvent.

Additionally, stainless-steel (SS) coupons of dimensions identical to the dimensions of the test coupons and other controls were included as evaporation controls during each test to quantify the amount of CWA lost to evaporation during the CWA dwell period. The SS evaporation controls were spiked and sampled during the test alongside the test coupons using the same equipment and procedures. Three SS evaporation controls were wipe-sampled with subsequent solvent extraction of the coupons, and three were extracted in solvent alone (no wipe sampling). SS procedural and laboratory blanks were included as well.

Three CWA challenge amount confirmation controls (spike controls) were included in each test to confirm the CWA challenge application amount. Spike controls consisted of a spike of equal amount of VX or HD directly into extraction solvent (see Section 2.3.2.2). When spiked, CWA was applied to the inside surface of the glass spike control sample jar (60 mL jar, see Section 2.3.5). Submersion of the syringe needle into the solvent was avoided.

Table 1 summarizes the coupon wipe sampling and wipe and coupon solvent extraction demonstration tests that were performed.

Table 1. Surface (Wipe) Sampling and Solvent Extraction Method Demonstration Tests

CWA	Sample Type	Material	Spiked	CWA Dwell Period	Wipe Sampled	Replicates
VX	Test Coupon (wiping)	ABS	Yes	60 min	Yes	3
	Test Coupon (extraction)		Yes	60 min	No	3
	Procedural Blank		No	NA*	Yes	1
	Laboratory Blank		No	NA	No	1
	Test Coupon (wiping)	Acrylic	Yes	60 min	Yes	3
	Test Coupon (extraction)		Yes	60 min	No	3
	Procedural Blank		No	NA	Yes	1
	Laboratory Blank		No	NA	No	1
	Test Coupon (wiping)	Aluminum	Yes	60 min	Yes	3
	Test Coupon (extraction)		Yes	60 min	No	3
	Procedural Blank		No	NA	Yes	1
	Laboratory Blank		No	NA	No	1
	Evaporation Control (wiping)	SS	Yes	60 min	Yes	3
	Evaporation Control (extraction)		Yes	60 min	No	3
	Procedural Blank		No	NA	Yes	1
	Laboratory Blank		No	NA	No	1
	Spike Control	NA	Yes	NA	NA	3
HD	Test Coupon (wiping)	ABS	Yes	60 min	Yes	3
	Test Coupon (extraction)		Yes	60 min	No	3
	Procedural Blank		No	NA	Yes	1
	Laboratory Blank		No	NA	No	1
	Test Coupon (wiping)	Acrylic	Yes	60 min	Yes	3
	Test Coupon (extraction)		Yes	60 min	No	3
	Procedural Blank		No	NA	Yes	1
	Laboratory Blank		No	NA	No	1
	Test Coupon (wiping)	Aluminum	Yes	60 min	Yes	3
	Test Coupon (extraction)		Yes	60 min	No	3
	Procedural Blank		No	NA	Yes	1
	Laboratory Blank		No	NA	No	1
	Evaporation Control (wiping)	SS	Yes	60 min	Yes	3
	Evaporation Control (extraction)		Yes	60 min	No	3
	Procedural Blank		No	NA	Yes	1
	Laboratory Blank		No	NA	No	1
	Spike Control	NA	Yes	NA	NA	3

* NA = Not Applicable

2.2.1.2. *Quench Evaluation Test Matrix*

Matrices for the quench method evaluations are provided in Table 2. As discussed in Section 2.1.1.2, two quench methods were evaluated: (1) extraction in organic solvent alone, and (2) addition of 3M STS to the wipe/coupon extraction solvent. Solvent extraction alone was evaluated initially as a quench for all six CWA/decontaminant combinations. A subsequent test was then conducted to evaluate the adequacy of 3M STS as a quench for those combinations that were not adequately quenched by solvent extraction alone.

Table 2. Quench Method Demonstration Tests

Test No.	Material Type	Quench Method	Decontaminant	Post-Spike CWA (Wipe and Coupon Extracts)	Replicates
1	Aluminum	Extraction in 25 mL of hexane only	EasyDECON DF200	VX	3
			Dahlgren Decon	VX	3
			$eClO_2$	VX	3
			None	VX	3
			EasyDECON DF200	HD	3
			Dahlgren Decon	HD	3
			$eClO_2$	HD	3
			None	HD	3
			EasyDECON DF200	None	1
			Dahlgren Decon	None	1
			$eClO_2$	None	1
			None	None	1
2	Aluminum	Extraction in 25 mL of hexane and 15 mL 3M STS	EasyDECON DF200	VX	3
			Dahlgren Decon	VX	3
			None	VX	3
			Dahlgren Decon	HD	3
			None	HD	3
			EasyDECON DF200	None	1
			Dahlgren Decon	None	1
			None	None	1

2.2.2 Decontamination Efficacy Test Matrix

The complete matrix for decontamination efficacy testing is provided in Table 3. The matrix was completed twice, once using VX as the challenge CWA and again using HD as the challenge CWA, for a total of 18 decontamination efficacy tests. During each test, environmental conditions (temperature and relative humidity (RH)) in the test hood were monitored and recorded but not explicitly controlled.

In addition to the test and positive control coupons identified in Table 3, procedural blanks, laboratory blanks, and spike control samples were incorporated into each test. Spike controls were generated as described in Section 2.2.1.1. Procedural and laboratory blank samples consisted of coupons of the same SE-related materials of the same dimensions as the test coupons to which they were associated and are further described as follows:

- Procedural Blanks - SE-related material coupons that were not spiked but that were decontaminated, wipe sampled and extracted in solvent alongside the test coupons using the same equipment and procedures.
- Laboratory Blanks - SE-related material coupons that were not spiked, decontaminated, or wipe sampled; the coupons were maintained outside the test hood until placed into extraction solvent.

Table 3. Decontamination Efficacy Test Matrix

Test	Sample Type	Material	Spiked	Decontamination Technology	Wipe Sampled	Replicates
1	Test Sample	ABS Molded Plastic	Yes	EasyDECON DF200	Yes	5
	Positive Control	ABS Molded Plastic	Yes	None	Yes	3
	Procedural Blank	ABS Molded Plastic	No	EasyDECON DF200	Yes	1 + 1*
	Laboratory Blank	ABS Molded Plastic	No	None	No	1
2	Test Sample	Acrylic	Yes	EasyDECON DF200	Yes	5
	Positive Control	Acrylic	Yes	None	Yes	3
	Procedural Blank	Acrylic	No	EasyDECON DF200	Yes	1 + 1*
	Laboratory Blank	Acrylic	No	None	No	1
3	Test Sample	Aluminum	Yes	EasyDECON DF200	Yes	5
	Positive Control	Aluminum	Yes	None	Yes	3
	Procedural Blank	Aluminum	No	EasyDECON DF200	Yes	1 + 1*
	Laboratory Blank	Aluminum	No	None	No	1
4	Test Sample	ABS Molded Plastic	Yes	Dahlgren Decon	Yes	5
	Positive Control	ABS Molded Plastic	Yes	None	Yes	3
	Procedural Blank	ABS Molded Plastic	No	Dahlgren Decon	Yes	1 + 1*
	Laboratory Blank	ABS Molded Plastic	No	None	No	1
5	Test Sample	Acrylic	Yes	Dahlgren Decon	Yes	5
	Positive Control	Acrylic	Yes	None	Yes	3
	Procedural Blank	Acrylic	No	Dahlgren Decon	Yes	1 + 1*
	Laboratory Blank	Acrylic	No	None	No	1
6	Test Sample	Aluminum	Yes	Dahlgren Decon	Yes	5
	Positive Control	Aluminum	Yes	None	Yes	3
	Procedural Blank	Aluminum	No	Dahlgren Decon	Yes	1 + 1*
	Laboratory Blank	Aluminum	No	None	No	1
7	Test Sample	ABS Molded Plastic	Yes	$eClO_2$	Yes	5
	Positive Control	ABS Molded Plastic	Yes	None	Yes	3
	Procedural Blank	ABS Molded Plastic	No	$eClO_2$	Yes	1 + 1*
	Laboratory Blank	ABS Molded Plastic	No	None	No	1
8	Test Sample	Acrylic	Yes	$eClO_2$	Yes	5
	Positive Control	Acrylic	Yes	None	Yes	3
	Procedural Blank	Acrylic	No	$eClO_2$	Yes	1 + 1*
	Laboratory Blank	Acrylic	No	None	No	1
9	Test Sample	Aluminum	Yes	$eClO_2$	Yes	5
	Positive Control	Aluminum	Yes	None	Yes	3
	Procedural Blank	Aluminum	No	$eClO_2$	Yes	1 + 1*
	Laboratory Blank	Aluminum	No	None	No	1
	Spike Controls	NA	Yes	NA	NA	3 per test

* "+1" refers to an additional procedural blank that was not wiped or extracted along with other test coupons and controls following the decontamination period, to assess the effect of extended decontaminant contact with the materials.

2.3 Experimental Methods and Materials

Experimental methods and materials used to conduct all testing are described in the subsections below. Prior to the experimental research, literature searches were performed to identify decontamination technologies and approaches that are simultaneously efficacious in decontamination of CWA and compatible with materials often used in construction of SE. Specific search criteria comprised of keyword lists and Boolean search strategies were developed for use to execute the searches. The criteria and strategies were then applied to multiple information repositories and scientific and technical literature databases to accumulate secondary

data and information related to the decontamination efficacy and materials compatibility characteristics of various decontamination technologies, methodologies, and approaches. For the purpose of focusing the search, secondary data and information related to decontamination of the CWAs VX and HD were prioritized.

In the context of this study, SE materials were identified by the EPA project team as materials associated with the housing or exterior of larger sensitive equipment items. While these materials by themselves may not be a sensitive material, they represent materials that would be used to house sensitive equipment. Degradation of such materials would lead to exposure of the interior components to these decontaminants with potential additional incompatibilities with highly sensitive items such as optical components or electrical contacts.

2.3.1 Coupon Materials

The method demonstration, decontamination efficacy testing, and material/decontaminant compatibility evaluations were conducted using the following types of SE-related materials: ABS molded plastic, acrylic, and aluminum. These materials were selected because they are frequently included in construction of SE. Test articles consisted of coupons of each of the SE-related materials selected for the evaluation. Coupons measured 2.5 centimeters x 4 centimeters (cm; 10 square centimeters [cm^2] contamination/decontamination surface area), with thickness dependent upon the specific material.

ABS molded plastic is a common thermoplastic made by polymerizing styrene and acrylonitrile in the presence of polybutadiene. The proportions of the three constituents can vary from 15 to 35% acrylonitrile, 5 to 30% butadiene, and 40 to 60% styrene. ABS is generally regarded as a strong, lightweight plastic used in several applications including automotive components, protective cases, and kitchen appliances. Regarding SE, ABS is used in several applications including construction of electrical enclosures, medical devices for blood access, keyboard keycaps, and others. ABS plastic coupons used during this work had a thickness of approximately 6.4 millimeters (mm; 0.25 inch), which is representative of the thickness of most common electrical/electronics enclosures. 24 x 24-inch sheets (8586K471, McMaster-Carr®, Cleveland, Ohio) were obtained and cut into individual coupons for use during testing.

Acrylic, or poly(methyl methacrylate) (PMMA), Plexiglas™, or Lucite™ by DuPont, is a transparent thermoplastic often used as a lightweight, shatter-resistant alternative to glass. Typical uses of acrylic include aircraft windows, hard contact lenses, and eyeglass lenses. Regarding SE, acrylic is used in construction of semiconductors, dosimeters, liquid crystal displays and optical media (compact discs and digital video discs). For this work, acrylic coupons had a thickness of approximately 1.6 mm (0.0625 inch), which is representative of the thickness of CDs and DVDs. The 24 x 24-inch sheets (8560K174, McMaster-Carr®, Cleveland, Ohio) were obtained and cut into individual coupons.

Aluminum is a generally soft, light (low-density), corrosion-resistant metal used widely in the aerospace, automotive, and building industries, as well as extensively in the construction of

many types of SE. Type 6061 aluminum was used during this testing. Type 6061 aluminum is comprised of 0.4% to 0.8% silicon, up to 0.7% iron, 0.15% to 0.4% copper, up to 0.15% manganese, 0.8% to 1.2% magnesium, 0.04% to 0.35% chromium, up to 0.25% zinc, up to 0.15% titanium, 95.85% to 98.56% aluminum, and no more than 0.05% of any other single element and no more than 0.15% total of other elements. The 6061 aluminum is one of the most common alloys of aluminum for general purpose use, including the construction of handheld electronic devices and mobile phones. Aluminum coupons used for this work had a thickness of approximately 2 mm (0.08 inch), which is consistent with the thickness of most personal computer PC case walls. The 24 x 24-inch sheets (9015T246, McMaster-Carr®, Cleveland, Ohio) were obtained and cut into individual coupons.

As described in Section 2.2.1.1, stainless steel was also used for the wipe sampling and solvent extraction method demonstration tests. Type 304 stainless steel (24-gauge, 0.5 mm thickness) coupons precut by the supplier to the required 4.0 cm length and 2.5 cm width (custom part, Adept Products, Inc., West Jefferson, Ohio) were obtained for use during testing.

All coupons were cut to a uniform length (4.0 cm) and width (2.5 cm), so the top surface area to which the CWA challenge and decontamination technologies were applied measured 10 cm^2. Thicknesses were dependent upon the material type, as specified above. These dimensions enabled the coupons to fit lying flat at the bottom of the 125-mL jars that were used for solvent extraction (see Section 2.3.5). Following cutting, coupons were cleaned using dry air to remove dust and debris prior to use in tests. Aluminum and stainless steel coupons were also wiped using isopropyl alcohol (IPA)-soaked wipes to remove any machining/cutting grease residue. All coupons were visually inspected prior to use during all phases of testing to confirm the integrity and representativeness of the material. Coupons with irregular edges and/or damaged areas were discarded.

Table 4 provides a summary of test coupon information, including the number of coupons of each type that were prepared for use during testing.

Table 4. SE-Related Materials

Material	Description	Supplier Location	Coupon Dimensions	Preparation	Coupon Quantity
ABS Molded Plastic	Black plastic; approximately 6.4 mm thickness (electrical/electronics enclosures)	McMaster-Carr Cleveland, OH	4.0 cm length 2.5 cm width 6.4 mm thick	Coupons cut from 61 x 61 cm (24 x 24 inch) sheet; cleaned using dry air to remove cutting debris	321
Acrylic	Clear plastic; approximately 1.6 mm thickness (CD/DVD thickness)	McMaster-Carr Cleveland, OH	4.0 cm length 2.5 cm width 1.6 mm thick	Coupons cut from 61 x 61 cm (24 x 24 inch) sheet; cleaned using dry air to remove cutting debris	308
Aluminum	6061 alloy aluminum; approximately 2 mm thickness (computer case)	McMaster-Carr Cleveland, OH	4.0 cm length 2.5 cm width 2 mm thick	Coupons cut from 61 x 61 cm (24 x 24 inch) sheet; cleaned using dry air to remove cutting debris, wiped with IPA wipe	378
Stainless Steel	Type 304 stainless steel; 24-gauge thickness (approximately 0.5 mm)	Adept Products, Inc. West Jefferson, OH	4.0 cm length 2.5 cm width 0.5 mm thick	Coupons cut from 41 x 41 cm (16 x 16 inch) sheet; cleaned using dry air to remove cutting debris, wiped with IPA wipe	125

2.3.2 CWA Application

2.3.2.1. *CWA*

All quantities of VX and HD used during this work were synthesized at Battelle's HMRC under Chemical Weapons Convention program guidelines. All VX and HD originated from the same synthesis lots. VX and HD were stored in the HMRC CWA vault until needed for testing in accordance with HMRC security and CWA storage policies. To preserve CWA purity, VX was stored in multiple sealed glass ampoules (one ampoule per test, based on the matrices provided in Sections 2.2.1 and 2.2.2, with the sealed volume based on the anticipated need for a particular test). As HD has been demonstrated to be much less susceptible to degradation when maintained in accordance with the HMRC controlled storage policies, HD was stored in a single capped vial from which quantities were drawn for use when needed. Table 5 provides purity information for VX and HD used during testing and identifies the tests during which each CWA was used.

Table 5. CWA Purity

CWA	Purity	Tests Used
HD	99.9%	All methods development and decontamination efficacy
VX	95.1%	Methods development
VX	95.0%	DF200/ABS plastic, DF200/acrylic
VX	94.9%	DF200/aluminum, Dahlgren Decon/ABS plastic
VX	95.1%	$eClO_2$/ABS plastic, $eClO_2$/acrylic, $eClO_2$/aluminum
VX	95.0%	Dahlgren Decon/acrylic, Dahlgren Decon/aluminum

2.3.2.2. *Coupon Spiking*

Test and positive control coupons were inspected visually prior to contamination with neat VX or HD, and any coupons with surface anomalies were not used. Neat VX or HD was applied to the center of each designated test coupon or positive control as a single 2 µL droplet (approximately 202 µg/cm^2 of VX, or 254 µg/cm^2 of HD, based on the 10 cm^2 coupon surface area) using a Hamilton® repeating dispenser (83700, Hamilton, Reno, NV, or equivalent) and 100 µL Hamilton Gastight® syringe (81085, Hamilton, Reno, NV, or equivalent). Spiked control samples were generated by delivering the same quantity of CWA (2 µL) directly into 25 mL of extraction solvent, rather than onto a coupon surface. Following spiking, spike controls were processed in a manner similar to wipe and coupon extracts (that is, spike controls were sonicated and aliquoted as described for wipe and coupon extracts in Section 2.3.5).

2.3.2.3. *CWA Dwell Period*

Following application of CWA, the contaminated coupons were allowed to remain undisturbed for a 60-minute CWA dwell period. During the dwell period, the coupons were subjected to the ambient atmosphere within the test hood. Each coupon was covered with a Petri dish or other loose cover to protect from air currents. Temperature and RH of the coupon environment within the hood were monitored and recorded but not controlled. Typical ambient laboratory temperature (and thus, temperature within the hood where the coupons were located) ranged from 17 °C (64 degrees Fahrenheit [°F]) to 24 °C (75 °F). Testing was not initiated if the ambient laboratory temperature was outside this range. Ambient RH in the laboratory was more variable and was dependent on outdoor weather conditions and time of year, but typically ranged from 5% to 70%. RH was not expected to have an impact on evaporation of the CWA. Temperature and RH conditions within the hood were measured and recorded using a HOBO UX100 Data Logger (UX100-003, Onset® Computer Corporation, Bourne, MA) on each day of testing. Environmental conditions for each test are provided as Appendix A.

2.3.3 Decontamination Technologies

2.3.3.1. *Dahlgren Decon*

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). Part A of Dahlgren Decon was obtained premixed and ready for use (normally Part A in solid form must be dissolved in water before mixing with Parts B1 and B2). Prior to each test, a 200-mL quantity of Dahlgren Decon was prepared for use by mixing the three parts in accordance with directions provided by the manufacturer. The decontaminant was then used (applied to designated coupons) within 30 minutes of preparation. According to the manufacturer, the unmixed components have a ten-year shelf life, and the decontaminant remains efficacious for at least 6 hours after the components are mixed.

Consistent with the manufacturer-recommended use instructions, a decontaminant to CWA ratio of 50:1 (by volume) was used during decontamination efficacy testing. Thus, 100 µL of decontaminant was applied to designated coupons according to procedures described in Section 2.3.3.4, following the CWA dwell period.

According to the manufacturer, Dahlgren Decon is capable of 100% neutralization of HD in two minutes and 95% neutralization of VX in 15 minutes. For this testing, a 60-minute decontamination contact period was used.

2.3.3.2. *EasyDECON DF200*

EasyDECON DF200 (200-5312, Intelagard, Lafayette, CO) is a commercial variant of Sandia National Laboratories' decontamination foam DF200. EasyDECON DF200 is a three-component decontaminant system containing water and water-soluble cationic surfactants (Part 1), hydrogen peroxide (8% H_2O_2, active ingredient, Part 2), and diacetin (CAS 25395-31-7, catalyst, Part 3). The decontaminant can be applied as a liquid or a foam (using compressed air systems that inject air into the pumped liquid decontaminant to create the foam). For this testing, EasyDECON DF200 was applied as a liquid. EasyDECON DF200 is not received premixed, but rather the three parts are received packaged separately and need to be mixed prior to use (by combining the complete volumes of all three parts).

EasyDECON DF200 was prepared daily (each day of testing) in small batches of 10 mL each by combining 4.9 mL of Part 1, 4.9 mL of Part 2, and 200 µL of Part 3 in the correct order and manner specified by the EasyDECON DF200 use instructions. According to the manufacturer, Part 2 of EasyDECON DF200 (hydrogen peroxide active ingredient) has a shelf life of up to five years when stored at ideal conditions. Following preparation (proper mixing of the components in accordance with manufacturer directions), the decontaminant then has a pot-life of eight hours (per the manufacturer). Manufacturer-recommended use procedures require that the contaminated surface to which the decontaminant is applied be kept wet for a period of no less than 10 minutes. For this testing, 100 µL of prepared decontaminant was applied to designated coupons (directly on top of the liquid CWA droplet in the case of test coupons). During previous EPA studies using EasyDECON DF200 to decontaminate HD from nonporous material coupons (sealed concrete, glass, galvanized metal ductwork) [2], 60 µL of EasyDECON DF200 decontaminant was applied to a 1 µL CWA challenge on coupons with 5.25 cm^2 surface area. Use of a 100 µL decontaminant volume is just less than the 60:1 decontaminant to CWA ratio used during that work and is consistent with the 50:1 ratio recommended by other decontaminant manufacturers. A decontaminant contact period of 60 minutes was used.

2.3.3.3. *TDA Research Inc. HDA*

The Handheld Decontamination Apparatus (HDA) by TDA Research, Inc. is a developmental (not yet commercially available) handheld sprayer system equipped with an electrode for electrochemical generation of aqueous chlorine dioxide ($eClO_2$). The HDA is intended for use in decontamination of CWAs and biological agents from hard nonporous surfaces. The system

consists of the electrode-equipped sprayer, an active ingredient salt package (Part A), and a surfactant package (Part B). The Part A salt package contains a mixture of sodium chlorite and sodium bromide as active ingredients. Prior to use, the total contents of the two packages were added to 1 liter (L) of distilled water in the sprayer system bottle. Following mixing and reassembly of the sprayer system, the sprayer pump was primed for 10 to 15 seconds until the sprayed solution turned a light shade of yellow (per manufacturer use instructions, this yellow color indicates that the HDA is fully primed and ready for use). During use, the system dispenses the prepared salt solution through the electrochemical cell, oxidizing the salts and generating chlorine dioxide and hypobromite ions. According to the manufacturer, the mixed decontaminant solution is stable for multiple months. The decontaminant is not "activated" until it is delivered through the electrode-equipped sprayer, following which it must be used as soon as possible as the concentration of the oxidant will decrease quickly.

TDA Research, Inc., recommended use instructions require that the HDA spray stream be held perpendicular to the contaminated surface during application at a distance of no more than 12 inches. Per the manufacturer, it is also acceptable for experimental purposes to spray the electrochemical decontaminant into a glass jar or beaker and aliquot the solution onto contaminated surfaces. This "collect and aliquot" method was used during the decontamination efficacy testing conducted during this work. Just prior to the required decontaminant application time (approximately 20 to 30 minutes before the 60-minute CWA dwell period described in Section 2.3.2.3 was complete), the HDA $eClO_2$ decontaminant was prepared. Then, just prior to use during testing, a sufficient quantity was collected from the sprayer into a new, clean, and unused glass jar for application to all designated coupons for the test being run. Immediately after collection, the decontaminant was aliquoted from the jar onto the coupons according to procedures described in Section 2.3.3.4, below. The manufacturer did not establish a requirement to keep the contaminated surface to which the decontaminant is applied wet for a specific period. To maintain consistency with the other decontaminants, 100 µL of activated $eClO_2$ solution was applied to the center of each required coupon (directly on top of the CWA contamination, if applicable), and a 60-minute decontaminant contact period was used.

2.3.3.4. *Decontaminant Application*

The decontaminants were applied as liquids to test and procedural blank coupons using a positive displacement pipette (M-250E [50-250 µL pipette] and CP250 [tip], Gilson Inc, Middleton, WI). Decontaminant (100 µL) was applied to designated coupons in such a manner that the decontaminant remained pooled/beaded on the coupon surface (did not run off the edges of the coupon). In the case of spiked coupons (e.g., test coupons), decontaminant was applied directly on top of the CWA challenge. Following application, the decontaminants were allowed to remain undisturbed on the coupons (to react with the CWA challenge, in the case of test coupons) for 60 minutes. Coupons were left uncovered during the decontamination contact period. The air flow across coupons was not directly measured. Decontaminants were not reapplied during the 60-minute contact period as the 100 µL application volume used for all three decontaminants was

sufficient to keep the coupon surfaces wetted for the duration, even in the presence of the higher air flow across the coupons. Following the decontaminant contact period, coupons were wipe-sampled, extracted with solvent, or both according to procedures described in Sections 2.3.4 and 2.3.5. Decontaminant application volumes, contact periods, and rationale for each are summarized in Table 6.

Table 6. Decontamination Technology Application Volumes and Contact Periods

Decontamination Technology	Application Volume	Decon Contact Period	Rationale
Dahlgren Decon	100 µL (50:1 decontaminant by volume to CWA)	60 minutes	Manufacturer recommended 50:1 decontaminant to contaminant ratio. Manufacturer claimed HD neutralization in two minutes and VX neutralization in 15 minutes.
EasyDECON DF200	100 µL (50:1 decontaminant by volume to CWA)	60 minutes	Application volume is consistent with the 50:1 decontaminant to CWA ratio recommended for other decontaminants. Manufacturer recommends that contaminated surface be kept wet with decontaminant for at least 10 minutes.
TDA Research, Inc. HDA	100 µL (50:1 decontaminant by volume to CWA)	60 minutes	Collection of decontaminant from the sprayer into glassware and subsequent application via pipette onto contaminated surfaces is a practice accepted by the manufacturer for experimental purposes. Manufacturer did not provide recommendation on the time that the surface should be kept wet with decontaminant.

2.3.4 Coupon Surface (Wipe) Sampling

The method for coupon surface wipe-sampling used during this work was evaluated prior to decontamination efficacy testing to ensure adequate recovery of VX and HD could be achieved from the SE-related materials included in testing (refer to Section 2.1.1.1). The method included the following details:

- Wipes used were lint-free 2 × 2-inch (5 × 5 cm) four-ply rayon/polyester blend (gauze) sponges (22-037-921, Fisher Scientific, Pittsburgh, PA).
- The wipe was initially folded, as necessary, for manageability during wiping. Each coupon was wiped using an established wipe pattern (four horizontal and four vertical strokes with no folding between changes in direction). Given the small surface area of the coupons, strokes were short (coupon length) and placed on top of each other.

No blotting or rinsing of any excess liquid decontaminant remaining on coupons was performed. The excess decontaminant was absorbed into the wipe during the wiping action. As previously described, adequate methods for quenching the decontaminant reactions were demonstrated prior to decontamination efficacy testing (Sections 2.1.1.2 and 2.2.1.2).

Wipes were extracted in the same manner as coupons, as described in Section 2.3.5, using the same solvent as used to wet the wipes. Wipe extracts were analyzed for VX or HD by GC/MS as described in Section 2.4.

2.3.5 Coupon and Wipe Solvent Extraction

All coupons and wipes were extracted by placing each into a separate 60 mL glass jar (05-719-257, Fisher Scientific, Pittsburgh, PA, or similar) containing 25 mL of hexane. Hexane with internal standard (IS; 2.5 µg/mL naphthalene-d_8, AC17496-0010, Fisher Scientific, Pittsburgh, PA) was prepared in 4-L batches prior to filling individual extraction jars to ensure a consistent internal standard (IS) concentration in each sample. A stabilizer (N,N'-diisopropylcarbodiimide (DIC), CAS 693-13-0, D125407-100G, Sigma Aldrich, St. Louis, MO) was also added to help improve the sensitivity of the GC/MS analysis of VX samples. During previous studies, the DIC stabilizer was demonstrated to provide the intended benefits to VX analysis (without drawbacks) while not affecting analysis for HD [1].

Using the dimensions provided in Table 4, coupons of the SE-related materials fit lying flat within the inside diameter of the extraction jars identified above. Extraction solvent (25 mL) reached a height within the jar of approximately 2 cm. This jar and volume of solvent were sufficient to submerge all coupon types fully. ABS plastic coupons were identified to float in the extraction solvent and so were placed into the jars with the CWA-exposed/decontaminated side facing downward (into the extraction solvent).

Following addition of wipes or coupons to the extraction solvent within each jar, the jars were swirled by hand for approximately 5-10 seconds and then placed into a sonicator. Extraction jars were sonicated at 40 - 60 kilohertz for 10 min. Within 30 minutes of completing this process, aliquots of at least 0.5 mL from each extraction jar were transferred to individual GC vials (21140 [vial], 24670 [cap], Fisher Scientific [Restek Corp.], Hanover Park, IL 60133) and sealed. Samples that were not analyzed the same day were stored at -20 ± 10 °C.

2.4 Analytical Methods

2.4.1 VX and HD Quantitative Analysis

Wipe and coupon extracts were analyzed to quantify the amount of VX or HD present and to semi-quantitatively assess the presence of degradation products[3] (see Sections 2.4.2 and 2.4.3) using GC/MS (6890 gas chromatograph and 5973 mass selective detector, Agilent Technologies, Santa Clara, CA). Each sample set was analyzed in full scan mode for compounds ranging from 40 to 500 atomic mass units (amu) to quantify VX or HD and to determine the presence of degradation products. VX was detected with ions 114, 72, 127, and 79. HD was detected with ions 158, 109, 160, and 111. GC/MS parameters used for analysis are provided in Table 7.

Table 7. GC/MS Conditions for VX and HD Analysis

CWA	Parameter	Description
VX	Instrument	Hewlett Packard Model HP 6890 Gas Chromatograph equipped with HP 5973A Mass Selective Detector and Model 7683 Automatic Sampler
	Data System	MSD ChemStation
	Column	Rxi-5Sil MS (cross-linked methylsilicone), 30 meters x 0.25 mm, 0.25 µm film thickness (Restek Cat. No. 13653)
	Liner Type	4 mm Split/Splitless
	Carrier Gas Flow Rate	1.2 mL/min
	Column Temperature	50 °C initial temperature, hold 1 min, 30 °C/min to 280 °C, hold 0 min
	Injection Volume	1.0 µL
	Injection Temperature	250 ºC
	MS Quad Temperature	150 °C
	MS Source Temperature	230 ºC
	Solvent Delay	3 min
HD	Instrument	Hewlett Packard Model HP 6890 Gas Chromatograph equipped with HP 5973A Mass Selective Detector and Model 7683 Automatic Sampler
	Data System	MSD ChemStation
	Column	Rxi-5Sil MS, 30.0 meters × 0.25 mm, 0.25 µm film thickness
	Liner Type	4 mm Split/Splitless
	Carrier Gas Flow Rate	1.2 mL/min
	Column Temperature	40 °C initial temperature, hold 2.0 min, 30 °C/min to 310 °C, hold 0 min
	Injection Volume	2.0 µL
	Injection Temperature	250 °C
	MS Quad Temperature	150 °C
	MS Source Temperature	230 ºC
	Solvent Delay	3 min

See Section 4.2.2 for GC/MS calibration details. Samples with quantification results that fell below the low standard were reported as the method minimum quantifiable limit (MQL). All data were reported to two significant figures. Generally, accurate quantification of VX or HD was prioritized over qualitative assessment of degradation products when selecting GC/MS method parameters (i.e., quantitative VX or HD analysis capability was not sacrificed for increased capability to qualitatively assess degradation products).

2.4.2 VX Byproduct Qualitative Analysis

Extracts were also analyzed to semi-quantitatively estimate the amount of diethyl methanephosphonate and diethyl dimethylpyrophosphonate present. Diethyl methanephosphonate and diethyl dimethylpyrophosphonate are degradation products of ethyl methylphosphonic acid (EMPA) and sometimes impurities associated with VX [3]. EMPA is a hydrolysis product of VX. During preliminary/unpublished testing, 10 µg/mL of EMPA in hexane with naphthalene-d_8 (IS) and DIC and in acetone with naphthalene-d_8 (IS) and DIC was not directly detected via GC/MS as described in Section 2.4.1 for the analysis of VX. The EMPA may have reacted or degraded in the hot inlet of the GC; however, degradation products of EMPA (diethyl methanephosphonate and diethyl dimethylpyrophosphonate) were detected.

Thus, analyses for diethyl methanephosphonate and diethyl dimethylpyrophosphonate were performed concurrently with analyses for VX during the full scan analysis of each sample set. Diethyl methanephosphonate was detected with ions 79, 97, and 125, and diethyl dimethylpyrophosphonate was detected with ions 203, 143, and 175.

An EMPA standard at 81 µg/mL (equivalent to the maximum response from each EMPA degradant if all the VX on a particular coupon were to degrade into EMPA) was included during VX analytical runs (along with the VX calibration curve and the VX continuing calibration verification [CCV] standards described in Section 4.2.2). An intermediate EMPA standard at 3 milligrams (mg)/mL was prepared first by addition of neat EMPA (98% purity, 386561-1G, Sigma-Aldrich, St. Louis, MO) to hexane containing naphthalene-d_8 (IS) and DIC. The intermediate was then diluted to the 81 µg/mL concentration standard that was included in the analytical runs. The EMPA standard served as a single "calibration point" that was compared to any EMPA-associated peaks in the test and control samples. Ratios of peak area response for EMPA-associated byproducts in the test samples to the peak area response of EMPA-associated byproducts in the single "calibration point" were reported for each test sample.

Note: the toxic byproduct of VX degradation, EA-2192, cannot be identified by GC/MS. Analysis for EA-2192 requires the use of liquid chromatography (LC)/MS, which was outside the scope of this testing. Thus, degradation of VX into EA-2192 was not evaluated during this work.

2.4.3 HD Byproduct Qualitative Analysis

bis(beta-Chloroethyl) sulfone (mustard sulfone, CAS 471-03-4) and thiodiglycol (TDG, CAS 111-48-8) were the target degradation byproducts of interest during HD analysis runs. Semi-quantitative analyses for mustard sulfone and TDG were accomplished in the same manner as the semi-quantitative analyses of diethyl methanephosphonate and diethyl dimethylpyrophosphonate during analyses for VX (Section 2.4.2). Analyses for mustard sulfone and TDG were performed concurrently with analyses for HD during the full scan runs of each sample set. Mustard sulfone was detected with ions 63, 65, 92, and 127, and TDG was detected with ions 61, 45, 91 and 104.

A single "calibration point" standard each of mustard sulfone and TDG, both at 102 µg/mL (equivalent to the maximum response from the byproduct if all the HD on a particular coupon were to degrade), was included in each full scan GC/MS run for analysis of HD. Intermediate standards for each byproduct at 3 mg/mL were prepared first by addition of mustard sulfone (S741930-100MG, Sigma-Aldrich, St. Louis, MO) or TDG (1 mg/mL solution in methanol, ERT-053-1.2ML, Sigma-Aldrich, St. Louis, MO) to hexane containing naphthalene-d_8 (IS) and DIC. The intermediates were then diluted to the 102 µg/mL concentration standards that were included in the analytical runs.

Ratios of peak area response for mustard sulfone and TDG in the test samples to the peak area response of mustard sulfone and TDG in the single "calibration point" were reported for each test sample.

2.5 Calculations

Test, control, and blank coupon and wipe extract concentrations were provided in units of µg of VX or HD per mL of extract by the GC/MS ChemStation software through comparison of analyte peak areas to the calibration curve. Results less than the GC/MS MQL were set to the MQL for the sake of decontamination efficacy calculations. Mass recovered from the coupons/wipes via extraction was determined according to Equation 1:

$$Mass_{Rec} = Conc_{Ext} \times Vol_{Ext} \quad (1)$$

where: $Mass_{Rec}$ = CWA mass recovered from the coupon/wipe (µg)

$Conc_{Ext}$ = Coupon/wipe extract concentration provided by the GC/MS software (µg/mL)

Vol_{Ext} = Volume of extraction solvent (mL)

Total mass recovered from the test, control, or blank coupons was the sum of the masses recovered from the wipe sample taken from the coupon and from extraction of the coupon in solvent, according to Equation 2:

$$Mass_{Tot} = Mass_{Rec\ (wipe)} + Mass_{Rec\ (coupon)} \quad (2)$$

where: $Mass_{Tot}$ = Total CWA mass recovered (from wipe and coupon; µg)

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

$Mass_{Rec\ (coupon)}$ = CWA mass recovered from the coupon (µg)

Residual CWA contamination for each coupon was determined using the calculated total mass recovered (wipe and coupon) and the coupon contamination/decontamination surface area, according to Equation 3:

$$Cont_{Res} = \frac{Mass_{Tot}}{A_{Coupon}} \quad (3)$$

where: $Cont_{Res}$ = Residual coupon contamination (µg/cm^2)

$Mass_{Tot}$ = Total CWA mass recovered (from wipe and coupon; µg)

A_{Coupon} = Contamination/decontamination surface area of the coupon (cm^2)

Percent efficacy was then calculated for each individual test coupon according to Equation 4:

$$Efficacy = \left(\frac{Cont_{ResPos} - Cont_{ResTest}}{Cont_{ResPos}}\right) \times 100\% \quad (4)$$

where: $Cont_{ResTest}$ = Residual test coupon contamination (µg/cm^2)

$Cont_{ResPos}$ = Residual positive control coupon contamination (µg/cm^2)

For each CWA/SE-related material/decontamination technology combination, the mean of the efficacy values was determined. Thus, the primary result from testing was a matrix table in which each entry provided the mean and percent relative standard deviation (RSD) of efficacy results for each combination.

2.6 Analysis of Variance

For each CWA/decontamination technology/SE-related material type combination, arithmetic mean and percent RSD of the CWA recovery from test coupon and positive control sample sets were calculated, and test coupon CWA recovery means were compared to associated positive control means to determine if statistically significant decontamination of CWA occurred. F-tests were used to determine if the variances of results sets are equal or not. The null hypothesis that the variances of two sets were equal was rejected if the F-test p-value was ≤ 0.05. One-tailed, two-sample Student's t-tests (homoscedastic or heteroscedastic based on the F-test result) were then used to determine if the means of the test results were significantly less than the positive controls or not. The null hypothesis that the sample set means were equal was rejected if the t-test p-Value was ≤ 0.05.

Results were tested to determine if the data were reasonably bell-shaped and normally distributed. A natural logarithmic transformation of total mass recovery was performed to generally improve adherence to the statistical assumptions of normality and constant variance. A one-way analysis of variance (ANOVA) model was fitted separately for each CWA (HD or VX) and material (ABS plastic, acrylic, or aluminum) combination to the log transformed test sample total mass recovery with an effect for decontaminant (Dahlgren Decon, DF200, and $eClO_2$) to determine if there were significant performance differences among the different decontaminants. The geometric means from the ANOVA model were presented for each CWA and material combination. Tukey's multiple comparisons procedure was performed for each CWA/material combination to determine which pairs of decontaminants had geometric mean total mass recoveries that were significantly different from each other (however, the results are presented only if significant differences were identified).

Within each CWA/material/decontaminant test, the characteristics of CWA application to the positive controls are assumed to be the same as the characteristics of application to the test coupons with regard to variability from coupon to coupon and in the average amount of CWA applied. Acceptance criteria for the spike control results (average within 80% to 120% of theoretical, corrected for CWA purity; <30% RSD) are intended to support this assumption. For accurate comparison of the performance of the decontaminants within each CWA/material combination as described above, the amount of CWA applied to the test coupons must be consistent across all eighteen CWA/material/decontaminant combinations. To evaluate consistency of CWA application across the three tests (one test per decontaminant) of each CWA/material combination, the comparisons described above for the test samples were repeated using the geometric mean total recoveries from the three positive control sets associated with each CWA/material/decontaminant combination.

3. RESULTS

3.1 Method Demonstration

3.1.1 Wipe Sampling and Solvent Extraction Method Development Results

As described in Section 2.1.1.1, the methods for coupon surface wipe sampling and coupon solvent extraction were tested concurrently during two tests including both methods (one test per CWA). Hexane was demonstrated in each test as the wipe wetting and wipe and coupon extraction solvent. Also, as discussed in Section 2.1.1.1, stainless steel evaporation control coupons were included in the wipe sampling and coupon solvent extraction method development test matrices. Inclusion of the SS evaporation controls to account for the degree of evaporative losses of CWA during the 60-minute CWA dwell period was assumed to allow the measured recoveries from the test coupons to be attributed to the efficiency of the wipe sampling and solvent extraction methods.

As defined in Section 2.1.1.1, successful demonstration of the wipe sampling and coupon solvent extraction methods using hexane as the wipe wetting and wipe and coupon extraction solvent was defined as average CWA recovery from the test samples within 70% to 120% of the average of the stainless steel controls, with ≤ 30% RSD between replicates. These criteria were achieved for all three SE-related material types tested, successfully demonstrating the methods for use during decontamination efficacy testing. Average total VX recoveries from ABS plastic, acrylic, and aluminum measured 107%, 120%, and 119%, respectively. Average total recoveries of 75% from ABS plastic, 97% from acrylic, and 97% from aluminum were measured for HD.

No VX or HD was detected in any procedural or laboratory blank sample included in the wipe sampling and coupon solvent extraction method development tests. No degradation products for either CWA (as identified in Sections 2.4.2 and 2.4.3) were detected in any of the test samples.

Table 8 provides the average masses, standard deviations, and percent RSD for each sample type included during method development testing. Table 9 provides percent recoveries for each. As indicated, spike control recoveries were based on the theoretical target values, corrected for CWA percent purity (see Section 2.3.2).

Table 8. Wipe Sampling and Coupon Solvent Extraction Average Mass Recoveries

Sample Description	VX		HD	
	Avg. Mass Recovery (µg)	Std. Dev.	Avg. Mass Recovery (µg)	Std. Dev.
Spike Controls	2034	193	2699	233
Stainless Steel (extraction only)	1983	117	2698	123
Stainless Steel (wipe)	1621	124	2772	131
Stainless Steel (extraction following wiping)	8.3	6.3	8.2	9.9
Stainless Steel (total mass; wipe and ext.)	1629	127	2780	136
ABS Plastic (extraction only)	1944	182	1178	139
ABS Plastic (wipe)	1736	195	1392	248
ABS Plastic (extraction following wiping)	4.4	3.2	687	123
ABS Plastic (total mass; wipe and ext.)	1740	195	2079	144
Acrylic (extraction only)	2119	183	2646	204
Acrylic (wipe)	1943	63	2700	30
Acrylic (extraction following wiping)	3.9	1.2	2.9	0.76
Acrylic (total mass; wipe and ext.)	1947	62	2703	29
Aluminum (extraction only)	2209	206	2682	113
Aluminum (wipe)	1932	31	2699	168
Aluminum (extraction following wiping)	3.2	0.70	2.5	0.00
Aluminum (total mass; wipe and ext.)	1936	31	2702	168

Table 9. Wipe Sampling and Coupon Solvent Extraction Percent Recoveries

Sample Description	VX		HD		Percent recovery determined vs
	% Recovery	RSD	% Recovery	RSD	
Spike Controls	106%	9.5%	106%	8.6%	Theoretical
Stainless Steel (extraction only)	97%	5.9%	100%	4.6%	Spike controls
Stainless Steel (wipe)	82%	7.6%	103%	4.7%	Stainless steel (ext. only)
Stainless Steel (extraction following wiping)	0.42%	76%	0.31%	121%	Stainless steel (ext. only)
Stainless Steel (total mass; wipe and ext.)	82%	7.8%	103%	4.9%	Stainless steel (ext. only)
ABS Plastic (extraction only)	98%	9.3%	44%	12%	Stainless steel (ext. only)
ABS Plastic (wipe)	107%	11%	50%	18%	Stainless steel (total)
ABS Plastic (extraction following wiping)	0.27%	73%	25%	18%	Stainless steel (total)
ABS Plastic (total mass; wipe and ext.)	107%	11%	75%	6.9%	Stainless steel (total)
Acrylic (extraction only)	107%	8.7%	98%	7.7%	Stainless steel (ext. only)
Acrylic (wipe)	119%	3.2%	97%	1.1%	Stainless steel (total)
Acrylic (extraction following wiping)	0.24%	31%	0.11%	26%	Stainless steel (total)
Acrylic (total mass; wipe and ext.)	120%	3.2%	97%	1.1%	Stainless steel (total)
Aluminum (extraction only)	111%	9.3%	99%	4.2%	Stainless steel (ext. only)
Aluminum (wipe)	119%	1.6%	97%	6.2%	Stainless steel (total)
Aluminum (extraction following wiping)	0.20%	22%	0.09%	0.00%	Stainless steel (total)
Aluminum (total mass; wipe and ext.)	119%	1.6%	97%	6.2%	Stainless steel (total)

Figures 1 and 2 summarize average mass recovery from each SE-related material type for each CWA. As indicated in the figures, the upper and lower limit bars correspond to 120% and 70% (respectively) of the average total mass recovery from the stainless steel evaporation controls associated with the test samples.

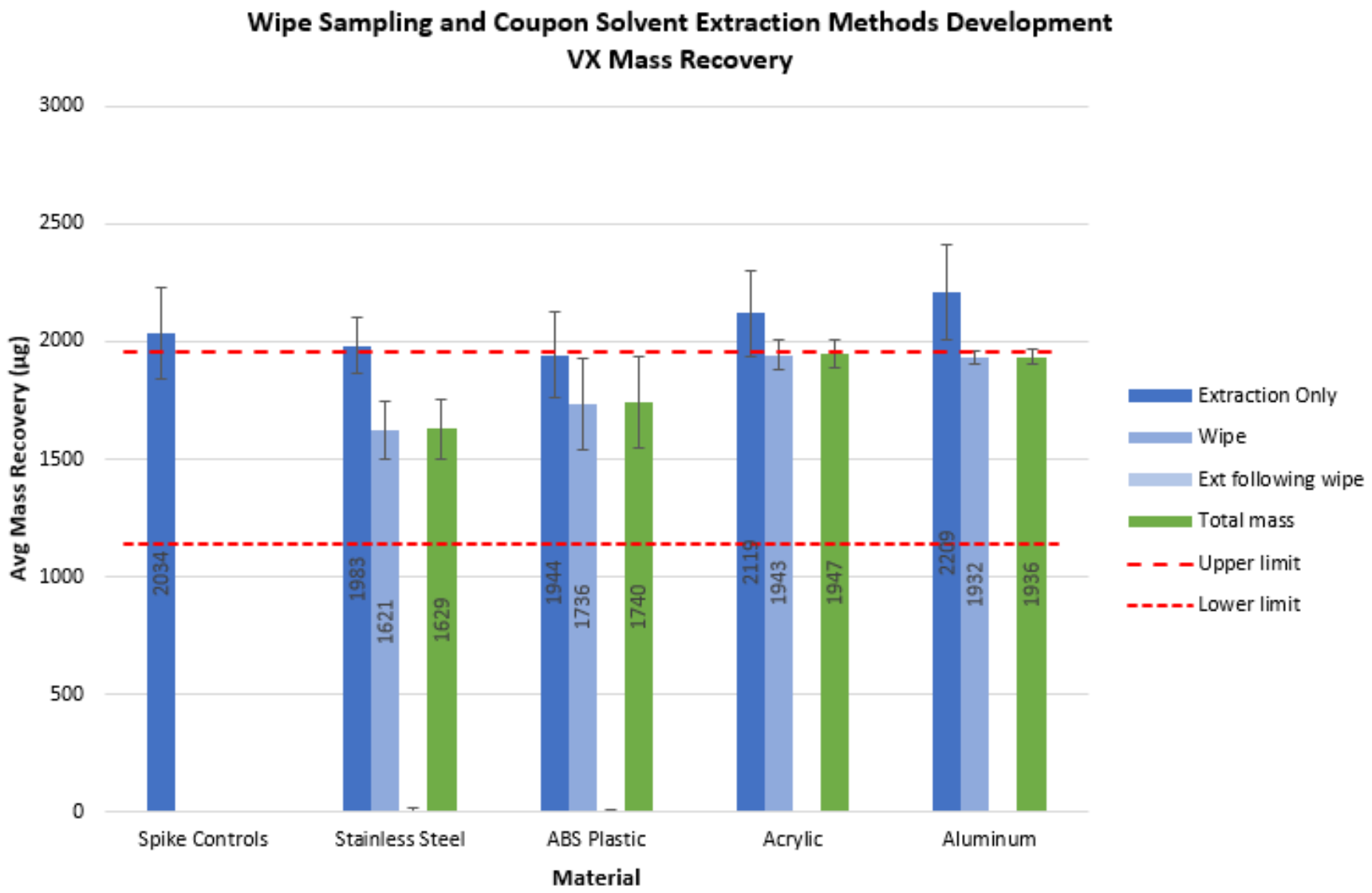


Figure 1. Wipe Sampling and Coupon Solvent Extraction, VX Mass Recovery
(Error bars equal ± one standard deviation; upper limit equals 120% of mean total mass recovery from stainless steel, lower limit equals 70% of mean total mass recovery from stainless steel; Ext is abbreviation for Extraction)

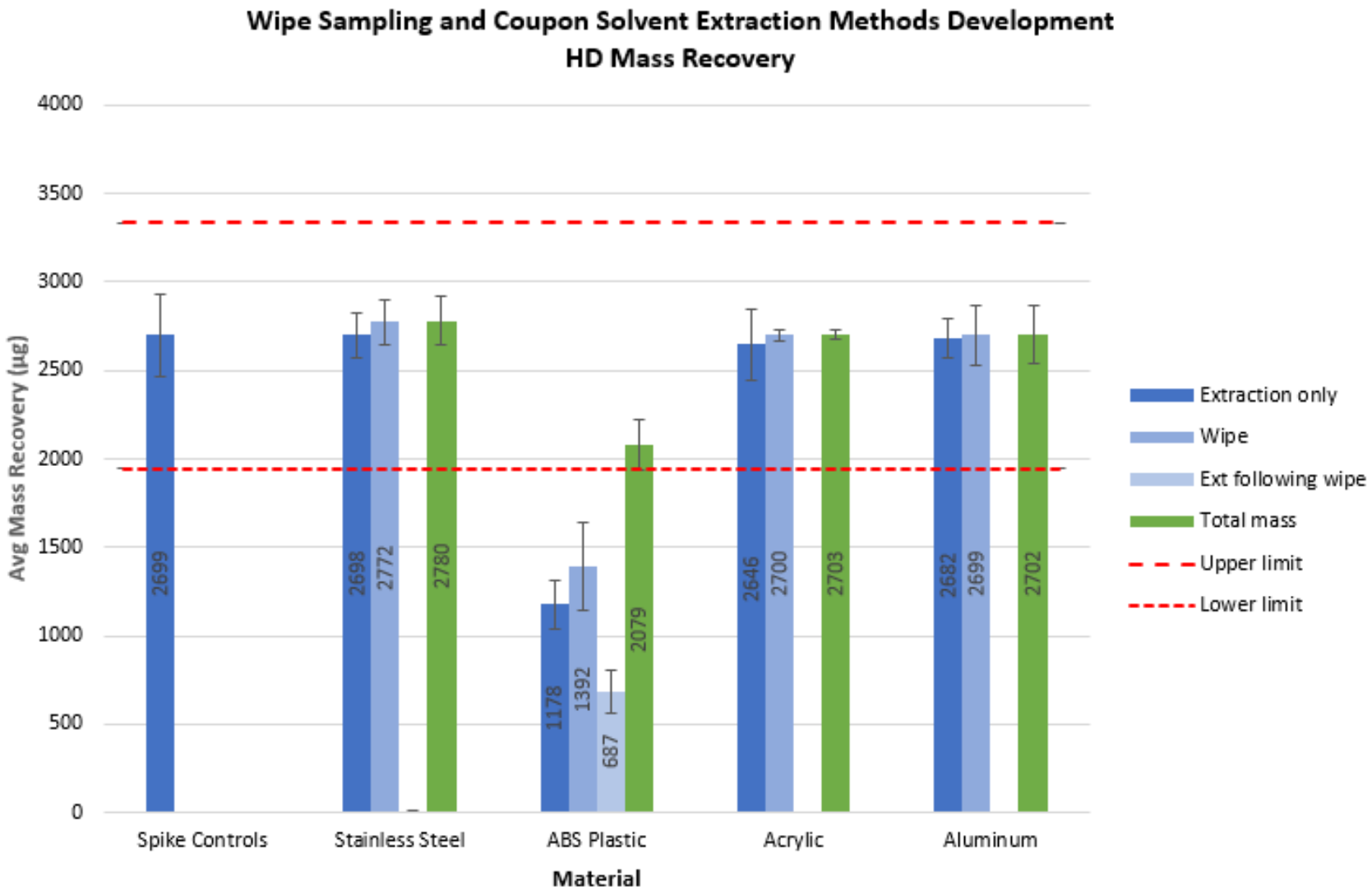


Figure 2. Wipe Sampling and Coupon Solvent Extraction, HD Mass Recovery
(Error bars equal ± one standard deviation; upper limit equals 120% of mean total mass recovery from stainless steel, lower limit equals 70% of mean total mass recovery from stainless steel; Ext is abbreviation for Extraction)

3.1.2 Quench Method Development Test Results

Based on method development data from previous EPA studies evaluating decontamination of HD and also considering that the three decontamination technologies selected for evaluation during this work are aqueous-based (refer to Section 2.3.3), extraction of wipes and coupons in organic solvent alone (hexane) was anticipated to be sufficient to halt the decontamination reaction via separation of any residual CWA from the decontaminant and preserve any residual VX and/or HD following the decontaminant contact period.

Average mass recoveries obtained during the first quench method test evaluating extraction in hexane alone are provided in Tables 10 and 11 and summarized in Figure 3. No VX or HD was detected in any blank sample included in the test. No degradation products for either CWA (see Sections 2.4.2 and 2.4.3) were detected in any of the samples.

As defined in Section 2.1.1.1, extraction in hexane alone would be considered adequate to quench the reactions of the decontaminants if the amounts of CWA recovered from post-spiked extracts containing decontaminant were at least 70% of the mean amount of CWA recovered from post-spiked extracts that did not contain decontaminant.

As described in Section 2.3.4, residual decontaminant still present on the surface of coupons after the 60-minute decontaminant contact period was absorbed into the wipe during the act of wipe sampling (i.e., decontaminant was not poured or rinsed off, or removed by some other means). The results provided in Tables 10 and 11 and in Figure 3 suggest that this practice led to residual decontaminant being nearly completely collected in the wipe sample. For decontaminants that were not quenched by extraction in hexane alone, recoveries of both CWAs from coupon extracts still ranged as high as 85% to 102% of the associated positive controls. Low recoveries were obtained only from the post-spiked wipe extracts for those samples. This suggests that unquenched decontaminant was only present in the wipe extracts and not in the coupon extracts.

Table 10. Quench Method Test 1, Solvent Extraction Alone, Average Mass Recoveries

CWA	Quench Method	Sample Description	Initial Analysis		Three- Day Storage at 20 °C	
			Avg. Mass Recovery (µg)	Std. Dev.	Avg. Mass Recovery (µg)	Std. Dev.
VX	Extraction in 25 mL of hexane	Spike Controls	109	43	109	41
		Positive Control Wipe Extracts	134	1.3	136	2.7
		Positive Control Coupon Extracts	135	1.5	137	3.8
		DF200 Wipe Extracts	45	24	53	24
		DF200 Coupon Extracts	114	7.6	125	7.4
		Dahlgren Decon Wipe Extracts	2.6	0.00	2.6	0.00
		Dahlgren Decon Coupon Extracts	131	6.6	129	8.7
		$eClO_2$ Wipe Extracts	135	2.1	137	6.3
		$eClO_2$ Coupon Extracts	131	8.0	132	7.2
HD	Extraction in 25 mL of hexane	Spike Controls	135	0.96	135	1.7
		Positive Control Wipe Extracts	138	5.3	137	4.5
		Positive Control Coupon Extracts	138	2.3	137	3.3
		DF200 Wipe Extracts	108	11	106	11
		DF200 Coupon Extracts	136	4.6	135	4.2
		Dahlgren Decon Wipe Extracts	2.6	0.00	2.6	0.00
		Dahlgren Decon Coupon Extracts	123	21	124	22
		$eClO_2$ Wipe Extracts	133	1.5	134	1.6
		$eClO_2$ Coupon Extracts	138	0.88	137	1.0

Table 11. Quench Method Test 1, Solvent Extraction Alone, Percent Recoveries

CWA	Quench Method	Sample Description	Initial Analysis		3 Day Storage at 20 °C		Percent recovery determined vs
			% Recovery	RSD	% Recovery	RSD	
VX	Extraction in 25 mL of hexane	Spike Controls	81%	39%	82%	38%	Theoretical
		Positive Control Wipe Extracts	123%	0.97%	125%	2.0%	Spike controls
		Positive Control Coupon Extracts	124%	1.1%	125%	2.8%	Spike controls
		DF200 Wipe Extracts	34%	52%	39%	45%	PC wipe extracts
		DF200 Coupon Extracts	85%	6.7%	92%	5.9%	PC coupon extracts
		Dahlgren Decon Wipe Extracts	2.0%	0.00%	1.9%	0.00%	PC wipe extracts
		Dahlgren Decon Coupon Extracts	97%	5.1%	94%	6.7%	PC coupon extracts
		$eClO_2$ Wipe Extracts	101%	1.6%	101%	4.6%	PC wipe extracts
		$eClO_2$ Coupon Extracts	97%	6.1%	96%	5.5%	PC coupon extracts
HD	Extraction in 25 mL of hexane	Spike Controls	101%	0.71%	101%	1.2%	Theoretical
		Positive Control Wipe Extracts	102%	3.8%	101%	3.3%	Spike controls
		Positive Control Coupon Extracts	102%	1.7%	102%	2.4%	Spike controls
		DF200 Wipe Extracts	78%	9.8%	77%	10%	PC wipe extracts
		DF200 Coupon Extracts	98%	3.4%	99%	3.1%	PC coupon extracts
		Dahlgren Decon Wipe Extracts	1.9%	0.00%	1.9%	0.00%	PC wipe extracts
		Dahlgren Decon Coupon Extracts	89%	17%	90%	18%	PC coupon extracts
		$eClO_2$ Wipe Extracts	97%	1.1%	98%	1.2%	PC wipe extracts
		$eClO_2$ Coupon Extracts	100%	0.64%	100%	0.73%	PC coupon extracts

PC = Positive Control

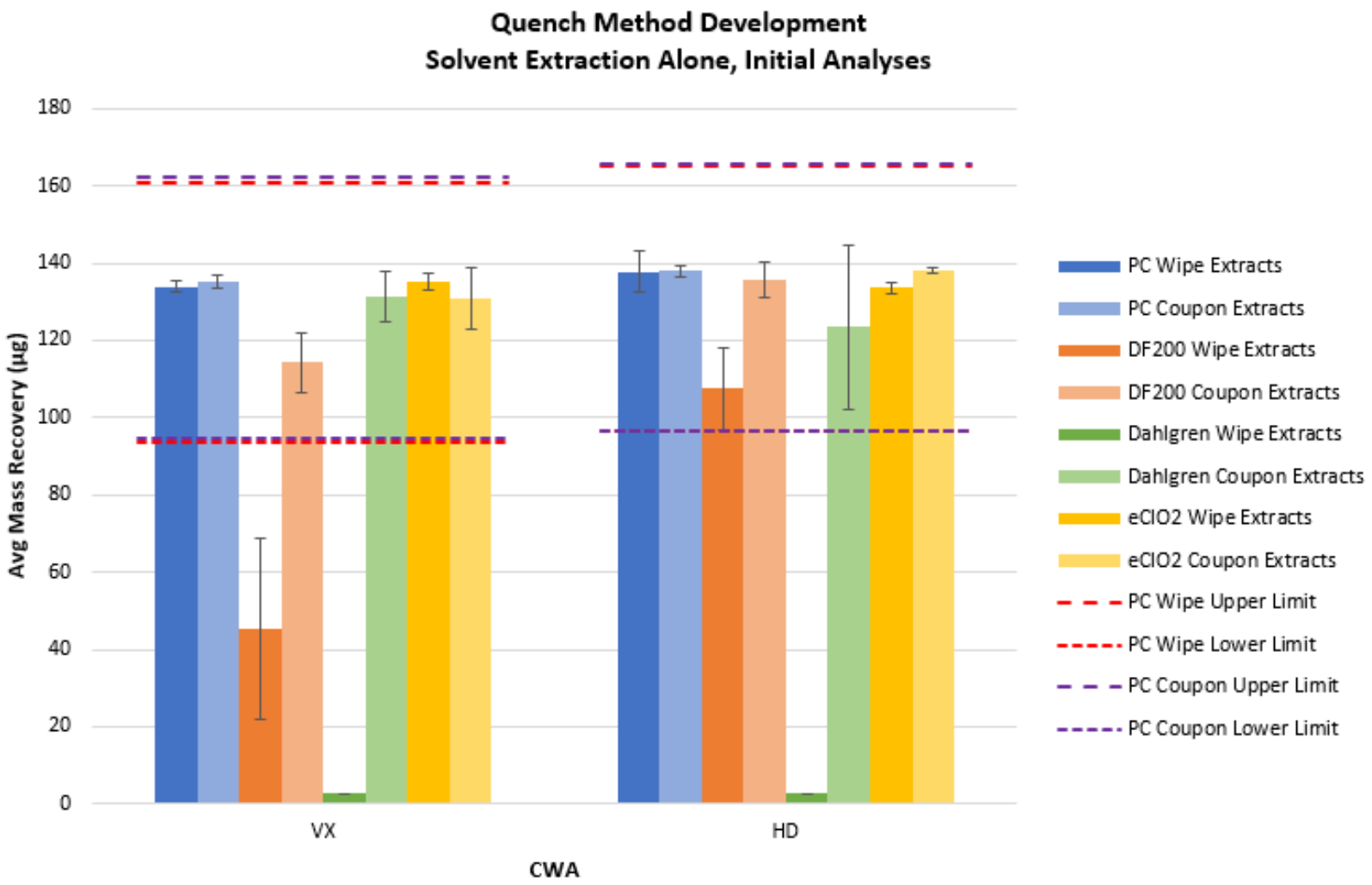


Figure 3. Quench Method Test 1, Solvent Extraction Alone, Average Mass Recoveries
(Error bars equal ± one standard deviation; upper and lower limit bars correspond to 70% and 120%, respectively, of the average mass recovery from positive wipe sample extracts [red] and positive coupon sample extracts [purple])

The data show that solvent extraction alone sufficiently quenched decontamination of VX and HD by the TDA $eClO_2$ decontaminant (post-spiked VX and HD recoveries ranging from 96% to 101% from wipe and coupon extracts of decontaminated coupons, both initially and after three days), as well as decontamination of HD by EasyDECON DF200 (post-spiked HD recoveries ranging from 77% to 99% from wipe and coupon extracts of decontaminated coupons, both initially and after three days). Conversely, extraction in hexane alone was not sufficient to prevent decontamination of post-spiked VX by DF200 or of either post-spiked VX or HD by Dahlgren Decon.

Based on these results, a second test was performed to evaluate the adequacy of a 3M solution of STS as a quenching agent to prevent decontamination of VX by DF200 and of VX and HD by Dahlgren Decon. As described in Section 2.1.1.2, the second test was performed in the same manner as the first using the same procedures and equipment, with the only exception being that 15 mL of 3M STS was added with the 25 mL of hexane to each wipe/coupon extraction jar.

Average mass recoveries obtained during the second quench method test evaluating 3M STS as an adequate quench are provided in Tables 12 and 13 and are summarized in Figure 4. No VX or HD was detected in any blank sample included in the test. No degradation products for either CWA (as identified in Sections 2.4.2 and 2.4.3) were detected in any of the samples.

Table 12. Quench Method Test 2, 3M STS, Average Mass Recoveries

CWA	Quench Method	Sample Description	Initial Analysis			Three- Day Storage at 20 °C		
			Avg. Mass Recovery (µg)	Std. Dev.	RSD (%)	Avg. Mass Recovery (µg)	Std. Dev.	RSD (%)
VX	Extraction in 25 mL of hexane with 15 mL 3M STS	Spike Controls	145	17	12%	140	5.5	4.0%
		Positive Control Wipe Extracts	159	4.8	3.0%	144	1.3	0.87%
		Positive Control Coupon Extracts	157	1.4	0.87%	144	1.5	1.0%
		DF200 Wipe Extracts	142	3.5	2.4%	127	2.8	2.2%
		DF200 Coupon Extracts	143	2.4	1.7%	141	2.4	1.7%
		Dahlgren Decon Wipe Extracts	161	10	6.1%	150	7.2	4.8%
		Dahlgren Decon Coupon Extracts	159	7.8	4.9%	146	0.65	0.44%
HD	Extraction in 25 mL of hexane with 15 mL 3M STS	Spike Controls	134	1.0	0.77%	135	0.46	0.34%
		Positive Control Wipe Extracts	135	1.1	0.84%	137	2.0	1.5%
		Positive Control Coupon Extracts	136	0.94	0.69%	138	2.6	1.9%
		Dahlgren Decon Wipe Extracts	135	3.1	2.3%	136	3.0	2.2%
		Dahlgren Decon Coupon Extracts	136	0.54	0.40%	137	1.1	0.83%

Table 13. Quench Method Test 2, 3M STS, Percent Recoveries

CWA	Quench Method	Sample Description	Initial Analysis		Three-Day Storage at 20 °C		Vs
			% Recovery	RSD	% Recovery	RSD	
VX	Extraction in 25 mL of hexane with 15 mL 3M STS	Spike Controls	108%	12%	105%	4.0%	Theoretical
		Positive Control Wipe Extracts	110%	3.0%	103%	0.87%	Spike controls
		Positive Control Coupon Extracts	109%	0.87%	103%	1.0%	Spike controls
		DF200 Wipe Extracts	89%	2.4%	88%	2.2%	PC wipe extracts
		DF200 Coupon Extracts	91%	1.7%	98%	1.7%	PC coupon extracts
		Dahlgren Decon Wipe Extracts	101%	6.1%	104%	4.8%	PC wipe extracts
		Dahlgren Decon Coupon Extracts	101%	4.9%	102%	0.44%	PC coupon extracts
HD	Extraction in 25 mL of hexane with 15 mL 3M STS	Spike Controls	101%	0.77%	101%	0.34%	Theoretical
		Positive Control Wipe Extracts	101%	0.84%	102%	1.5%	Spike controls
		Positive Control Coupon Extracts	102%	0.69%	103%	1.9%	Spike controls
		Dahlgren Decon Wipe Extracts	100%	2.3%	99%	2.2%	PC wipe extracts
		Dahlgren Decon Coupon Extracts	100%	0.40%	99%	0.83%	PC coupon extracts

PC = Positive Control

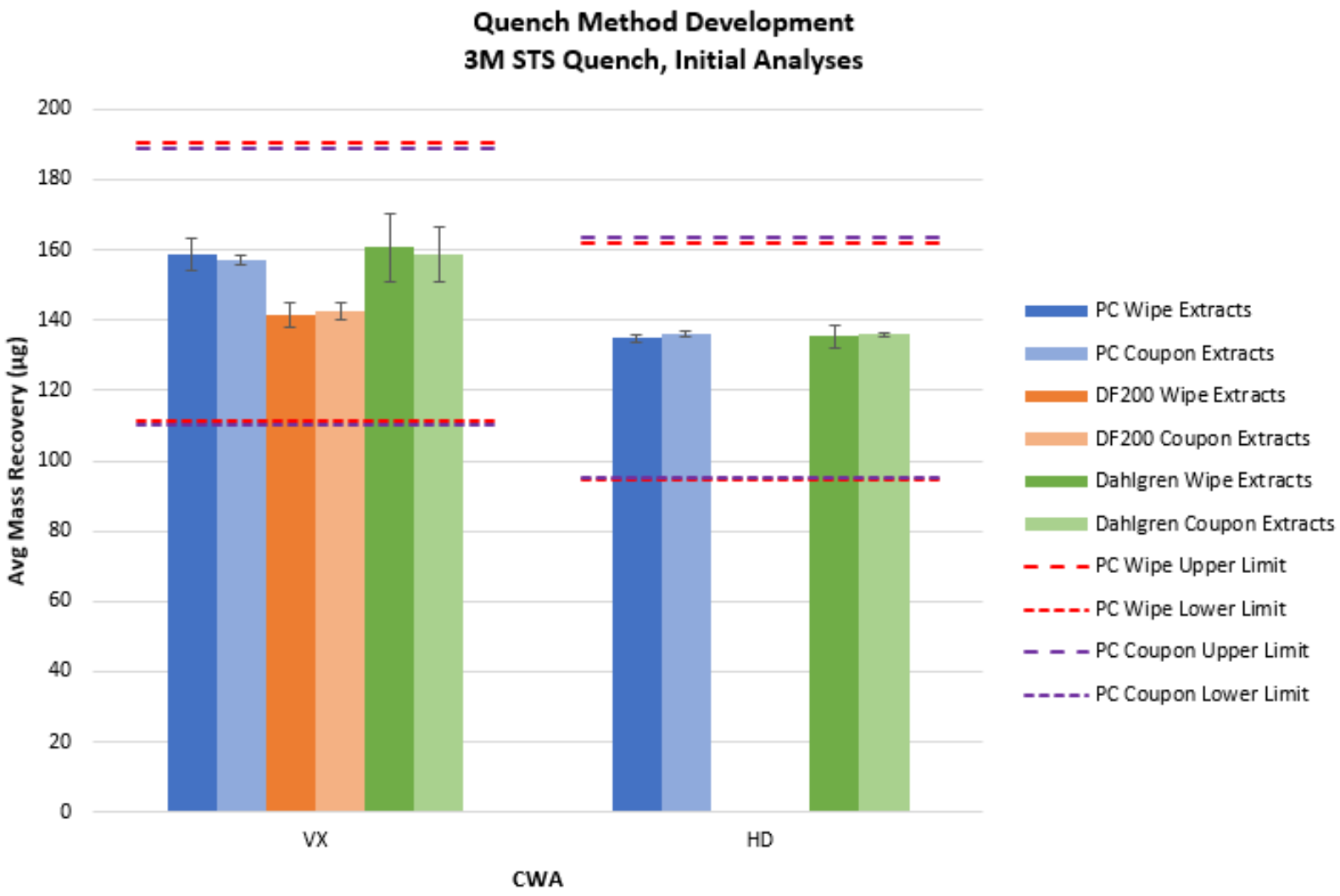


Figure 4. Quench Method Test 2, 3M STS, Average Mass Recoveries
(Error bars equal ± one standard deviation; upper and lower limit bars correspond to 70% and 120%, respectively, of the average mass recovery from positive wipe sample extracts [red] and positive coupon sample extracts [purple])

As demonstrated by the data, 3M STS was found to adequately quench decontamination of VX by EasyDECON DF200 (post-spiked VX recoveries ranging from 88% to 98% from wipe and coupon extracts of decontaminated coupons, both initially and after three days) and decontamination of VX and HD by Dahlgren Decon (post-spiked VX and HD recoveries ranging from 99% to 104% from wipe and coupon extracts of decontaminated coupons, both initially and after three days). Thus, the quench methods used for each CWA/decontaminant combination during decontamination efficacy testing were as follows:

- HD by EasyDECON DF200: Extraction in hexane alone (78% recovery of post-spiked HD in wipe extracts, 98% in coupon extracts)
- HD by Dahlgren Decon: 3M STS added to extraction solvent (100% recovery of post-spiked HD in wipe extracts, 100% in coupon extracts)
- HD by TDA $eClO_2$: Extraction in hexane alone (97% recovery of post-spiked HD in wipe extracts, 100% in coupon extracts)
- VX by EasyDECON DF200: 3M STS added to extraction solvent (89% recovery of post-spiked HD in wipe extracts, 91% in coupon extracts)
- VX by Dahlgren Decon: 3M STS added to extraction solvent (101% recovery of post-spiked HD in wipe extracts, 101% in coupon extracts)
- VX by TDA $eClO_2$: Extraction in hexane alone (101% recovery of post-spiked HD in wipe extracts, 97% in coupon extracts)

As described in Section 2.1.1.2, following the initial analyses of the samples generated during each quench method test, samples were stored at -20 °C for three days, and then reanalyzed to determine if decontamination of VX or HD in the samples occurred during storage. Tables 14 and 15 provide the wipe and coupon sample mass recoveries for the individual replicates included in each test, both from the initial analyses performed immediately after generation of the samples as well as during reanalyses of the samples following storage for three days at -20 °C. Calculated percent difference in mass recovery between the initial analysis and reanalysis is provided as well for each sample. Results suggest that continued decontamination of VX and HD in the samples did not occur.

Table 14. Quench Sample Reanalyses Percent Differences, 1st Test

Sample	Wipe Sample Mass Recovered			Coupon Mass Recovered		
	Initial (µg)	Reanalysis (µg)	% Diff	Initial (µg)	Reanalysis (µg)	% Diff
VX Positive 1	135	134	-1.4%	137	134	-1.6%
VX Positive 2	133	139	4.4%	134	135	1.0%
VX Positive 3	133	136	2.2%	136	141	4.1%
VX DF200 1	38	48	28%	109	120	10%
VX DF200 2	72	78	9.1%	123	134	8.8%
VX DF200 3	27	32	19%	111	122	10%
VX DD 1	2.6	2.6	0.00%	128	126	-1.4%
VX DD 2	2.6	2.6	0.00%	128	122	-4.0%
VX DD 3	2.6	2.6	0.00%	139	139	-0.14%
VX $eClO_2$ 1	138	144	4.5%	133	136	2.5%
VX $eClO_2$ 2	133	136	2.1%	137	136	-1.0%
VX $eClO_2$ 3	135	131	-2.7%	122	124	1.5%
HD Positive 1	144	142	-1.2%	139	140	0.65%
HD Positive 2	136	134	-2.0%	135	134	-1.2%
HD Positive 3	133	134	0.72%	139	137	-1.7%
HD DF200 1	104	106	1.6%	131	131	-0.31%
HD DF200 2	99	94	-4.7%	136	136	0.11%
HD DF200 3	119	116	-2.6%	140	139	-1.0%
HD DD 1	2.6	2.6	0.00%	136	137	0.88%
HD DD 2	2.6	2.6	0.00%	99	98	-0.75%
HD DD 3	2.6	2.6	0.00%	136	136	0.10%
HD $eClO_2$ 1	135	135	0.44%	137	138	0.71%
HD $eClO_2$ 2	132	133	0.45%	138	136	-1.3%
HD $eClO_2$ 3	134	133	-0.59%	139	136	-1.8%

Table 15. Quench Sample Reanalyses Percent Differences, 2nd Test

Sample	Wipe Sample Mass Recovered			Coupon Mass Recovered		
	Initial (µg)	Reanalysis (µg)	% Diff	Initial (µg)	Reanalysis (µg)	% Diff
VX Positive 1	164	145	-12%	159	145	-8.4%
VX Positive 2	156	143	-8.6%	156	143	-8.6%
VX Positive 3	156	144	-7.5%	157	145	-7.7%
VX DF200 1	141	128	-8.8%	140	138	-1.0%
VX DF200 2	145	129	-11%	143	142	-1.0%
VX DF200 3	139	124	-11%	145	143	-1.0%
VX DD 1	149	143	-4.6%	151	146	-3.3%
VX DD 2	166	157	-5.3%	159	146	-7.9%
VX DD 3	167	152	-9.0%	167	147	-12%
HD Positive 1	136	140	2.6%	136	137	0.63%
HD Positive 2	134	136	1.3%	137	141	2.7%
HD Positive 3	134	136	1.7%	136	136	0.61%
HD DD 1	138	139	0.92%	136	136	0.23%
HD DD 2	132	133	1.0%	136	138	1.2%
HD DD 3	136	136	-0.41%	136	137	1.3%

3.2 Decontamination Efficacy

3.2.1 VX Residual Contamination

Throughout all of the VX decontamination efficacy testing, no VX was detected in any procedural or laboratory blank samples. Spike control samples were within specification for all tests with one exception: during the test evaluating $eClO_2$ decontamination of VX from ABS plastic, two spike control sample recoveries were measured at 122% and 123% of the theoretical target value. The mean VX recovery value for spike controls during this test was 122% of theoretical, with 1.0% RSD. Neither EMPA-associated VX degradant (diethyl methanephosphonate or diethyl dimethylpyrophosphonate) was detected in any sample. Frequencies of detection of VX in wipe and coupon sample extracts are provided in Table 16.

Table 16. VX Frequency of Detection

Material	Sample Description	EasyDECON DF200		Dahlgren Decon		TDA $eClO_2$	
		Wipes	Coupons	Wipes	Coupons	Wipes	Coupons
ABS Plastic	Positive Controls	3/3	2/3	3/3	3/3	3/3	3/3
	Test Coupons	5/5	4/5	1/5	0/5	5/5	5/5
Acrylic	Positive Controls	3/3	3/3	3/3	3/3	3/3	3/3
	Test Coupons	5/5	2/5	5/5	1/5	5/5	5/5
Aluminum	Positive Controls	3/3	3/3	3/3	3/3	3/3	3/3
	Test Coupons	4/5	1/5	5/5	0/5	5/5	4/5

Frequency of detection = samples with detection above the GC/MS MQL/total replicate samples

For positive control coupon sets, a frequency of detection less than 3/3 indicates that all CWA was recovered in the wipe sample for one or more replicates.

Average mass recoveries from wipe samples and coupon extractions and calculated average residual VX contamination for each decontaminant/SE-related material combination included during testing are provided in Table 17 and summarized in Figure 5. Generally, the least VX was recovered from test coupons decontaminated with Dahlgren Decon, with average residual contamination values of 1.5 $\mu g/cm^2$ (ABS plastic), 2.4 $\mu g/cm^2$ (acrylic), and 3.2 $\mu g/cm^2$ (aluminum). Recoveries from coupons decontaminated with EasyDECON DF200 were slightly higher, with average residual contamination values of 30 $\mu g/cm^2$ (ABS plastic), 3.3 $\mu g/cm^2$ (acrylic), and 5.3 $\mu g/cm^2$ (aluminum). Both are in contrast to the markedly higher recoveries from test coupons decontaminated with TDA's $eClO_2$ decontaminant, with average residual contamination values of 191 $\mu g/cm^2$ (ABS plastic) and 139 $\mu g/cm^2$ (acrylic and aluminum).

Table 17. VX Average Recoveries and Residual Contamination

Decontaminant	Material	Sample Description	Average Recovery									Avg Residual Contamination		
			Wipe			Coupon			Total Mass					
			Mass (µg)	St. Dev. (µg)	RSD (%)	Mass (µg)	St. Dev. (µg)	RSD (%)	Mass (µg)	St. Dev. (µg)	RSD (%)	Avg. (µg/cm²)	St. Dev. (µg/cm²)	RSD (%)
EasyDECON DF200	ABS Plastic	Positive Controls	1790	76	4.2%	3.3*	0.73	22%	1793	76	4.2%	179	7.6	4.2%
		Test Coupons	295	188	64%	8.4*	9.0	107%	304	190	62%	30	19	62%
	Acrylic	Positive Controls	1817	51	2.8%	12	9.1	76%	1829	56	3.1%	183	5.6	3.1%
		Test Coupons	26	17	67%	7.2*	9.3	129%	33	25	75%	3.3	2.5	75%
	Aluminum	Positive Controls	1832	151	8.3%	36	28	77%	1868	124	6.6%	187	12	6.6%
		Test Coupons	50*	72	145%	2.8*	0.76	27%	53	73	138%	5.3	7.3	138%
Dahlgren Decon	ABS Plastic	Positive Controls	1823	102	5.6%	40	9.8	24%	1863	110	5.9%	186	11	5.9%
		Test Coupons	13*	23	177%	2.5*	0.0	0.00%	15	23	148%	1.5	2.3	148%
	Acrylic	Positive Controls	1971	67	3.4%	13	11	81%	1984	70	3.5%	198	7.0	3.5%
		Test Coupons	20	18	90%	4.2*	3.7	90%	24	19	78%	2.4	1.9	78%
	Aluminum	Positive Controls	1964	159	8.1%	41	9.2	22%	2005	154	7.7%	201	15	7.7%
		Test Coupons	30	45	152%	2.5*	0.0	0.00%	32	45	140%	3.2	4.5	140%
TDA $eClO_2$	ABS Plastic	Positive Controls	1965	178	9.1%	28	18	63%	1993	172	8.6%	199	17	8.6%
		Test Coupons	1852	139	7.5%	54	25	46%	1906	141	7.4%	191	14	7.4%
	Acrylic	Positive Controls	1941	45	2.3%	19	20	105%	1960	44	2.3%	196	4.4	2.3%
		Test Coupons	1364	105	7.7%	29	23	79%	1393	94	6.7%	139	9.4	6.7%
	Aluminum	Positive Controls	1924	44	2.3%	55	15	27%	1979	58	2.9%	198	5.8	2.9%
		Test Coupons	1340	267	20%	50*	63	126%	1390	211	15%	139	21	15%

*: Average recovery value includes non-detects (see Table 16) that were set at the MQL value.

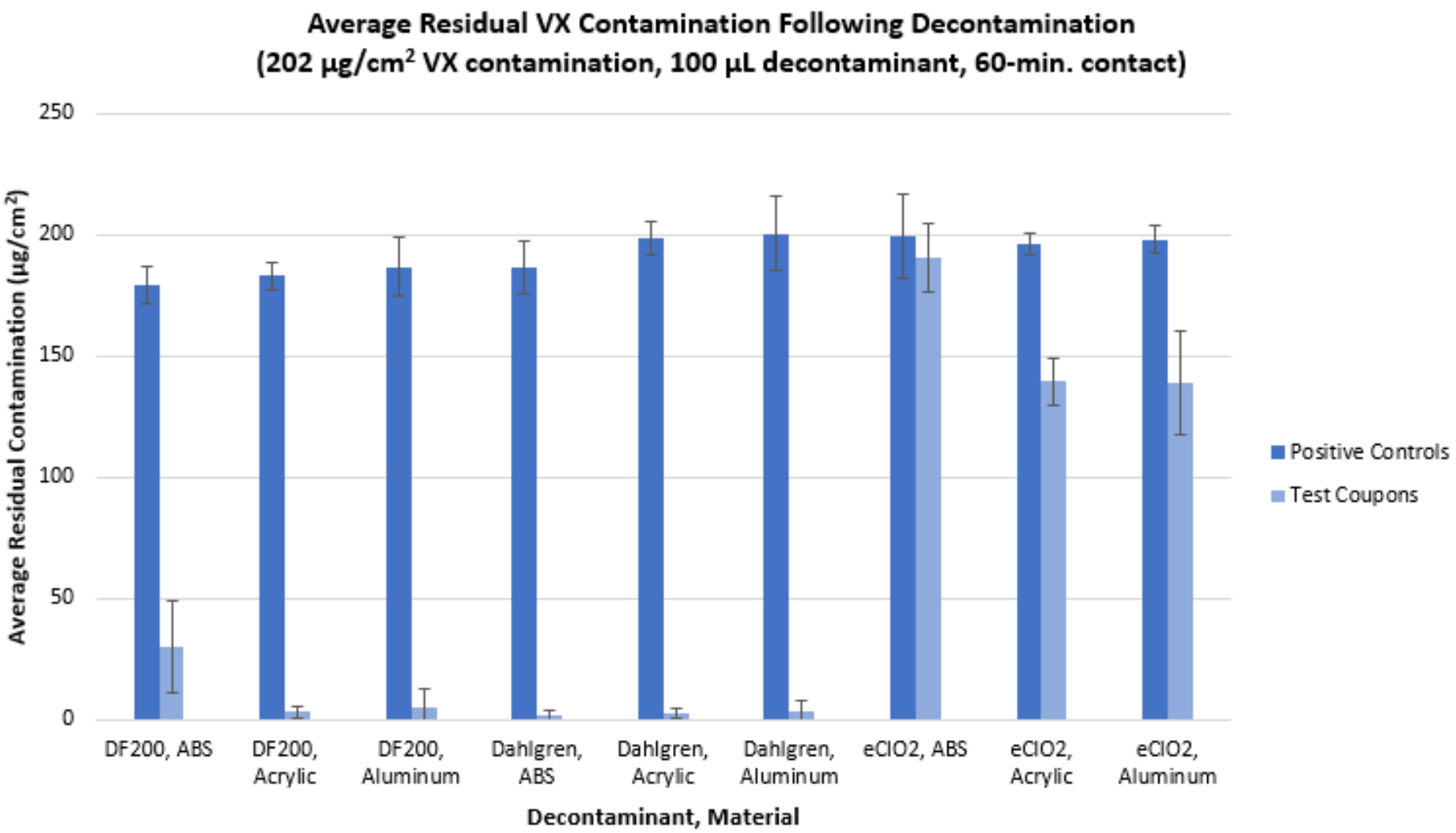


Figure 5. Average Residual VX Contamination
(Error bars equal ± one standard deviation)

3.2.2 HD Residual Contamination

Throughout all of HD decontamination efficacy testing, no HD was detected in any procedural or laboratory blank sample. Spike control samples were within specification for all tests. Frequency of detection of HD in wipe and coupon sample extracts is provided in Table 18.

Table 18. HD Frequency of Detection

Material	Sample Description	EasyDECON DF200		Dahlgren Decon		TDA $eClO_2$	
		Wipes	Coupons	Wipes	Coupons	Wipes	Coupons
ABS Plastic	Positive Controls	3/3	3/3	3/3	3/3	3/3	3/3
	Test Coupons	5/5	5/5	5/5	5/5	5/5	5/5
Acrylic	Positive Controls	3/3	2/3	3/3	3/3	3/3	2/3
	Test Coupons	5/5	5/5	5/5	3/5	5/5	4/5
Aluminum	Positive Controls	3/3	3/3	3/3	1/3	3/3	3/3
	Test Coupons	5/5	5/5	5/5	2/5	5/5	1/5

Frequency of detection = samples with detections above the GC/MS MQL/total replicate samples

For positive control coupon sets, a frequency of detection less than 3/3 indicates that all CWA was recovered in the wipe sample for one or more replicates.

No TDG was detected in any sample. However, mustard sulfone was detected in several samples. Table 19 lists samples in which mustard sulfone was detected and provides the peak area response ratio for each. Since the HD sulfone standard concentration was equivalent to the maximum response from this byproduct if all HD were to degrade to HD sulfone, these semi-quantitative ratios can be interpreted by first approximation as the percent conversion of HD into HD sulfone.

Table 19. Mustard Sulfone Detection

CWA	Material	Decontaminant	Sample Description	HD Sulfone *	
				Wipe Extract	Coupon Extract
HD	Aluminum	Dahlgren Decon	Test Coupon 1	-	-
			Test Coupon 2	0.29%	-
			Test Coupon 3	0.67%	-
			Test Coupon 4	0.12%	-
			Test Coupon 5	0.53%	-
HD	ABS Plastic	Dahlgren Decon	Test Coupon 1	0.43%	-
			Test Coupon 2	-	-
			Test Coupon 3	-	-
			Test Coupon 4	-	-
			Test Coupon 5	-	-
HD	ABS Plastic	$eClO_2$	Test Coupon 1	1.2%	0.40%
			Test Coupon 2	1.4%	0.46%
			Test Coupon 3	1.3%	0.64%
			Test Coupon 4	1.1%	0.61%
			Test Coupon 5	2.5%	0.76%
HD	Acrylic	$eClO_2$	Test Coupon 1	6.3%	1.0%
			Test Coupon 2	7.9%	-
			Test Coupon 3	8.7%	0.41%
			Test Coupon 4	8.2%	0.15%
			Test Coupon 5	8.7%	-
HD	Aluminum	$eClO_2$	Test Coupon 1	8.9%	-
			Test Coupon 2	9.9%	-
			Test Coupon 3	9.2%	-
			Test Coupon 4	6.9%	-
			Test Coupon 5	14%	-

* Percentage = Sample HD sulfone peak area response/HD sulfone standard peak area response (see Section 2.4.3).

Average mass recoveries from wipe samples and coupon extractions and calculated average residual HD contamination for each decontaminant/SE-related material combination included during testing are provided in Table 20 and summarized in Figure 6. HD recoveries from positive controls were lower across all materials than recoveries recorded during the wipe sampling and solvent extraction method development (Table 8). However, method development recoveries were for a 60 min dwell time of the agent on the surface versus a 120 min contact time in the decontamination study. The highest test coupon recoveries of HD came from acrylic and aluminum decontaminated with DF200 (average residual contamination values of 177 $\mu g/cm^2$ and 210 $\mu g/cm^2$, respectively). The remaining decontaminant combinations ranged from 92 $\mu g/cm^2$ (acrylic/$eClO_2$) to 137 $\mu g/cm^2$ (aluminum/$eClO_2$).

Table 20. HD Average Recoveries and Residual Contamination

Decontaminant	Material	Sample Description	Average Recovery									Avg. Residual Contamination		
			Wipe			Coupon			Total Mass					
			Mass (µg)	St. Dev. (µg)	RSD (%)	Mass (µg)	St. Dev. (µg)	RSD (%)	Mass (µg)	St. Dev. (µg)	RSD (%)	Avg. (µg/cm^2)	St. Dev. (µg/cm^2)	RSD (%)
EasyDECON DF200	ABS Plastic	Positive Controls	1027	81	7.9%	545	88	16%	1572	37	2.3%	157	3.7	2.3%
		Test Coupons	570	215	38%	538	102	19%	1108	305	28%	111	31	28%
	Acrylic	Positive Controls	2162	112	5.2%	7.9	8.9	113%	2170	112	5.2%	217	11	5.2%
		Test Coupons	1712	198	12%	55	76	138%	1767	155	8.7%	177	15	8.7%
	Aluminum	Positive Controls	2301	47	2.0%	13	6.7	53%	2314	48	2.1%	231	4.8	2.1%
		Test Coupons	2055	63	3.1%	42	65	155%	2097	70	3.3%	210	7.0	3.3%
Dahlgren Decon	ABS Plastic	Positive Controls	921	202	22%	607	59	9.7%	1528	259	17%	153	26	17%
		Test Coupons	548	178	32%	394	134	34%	942	133	14%	94	13	14%
	Acrylic	Positive Controls	2374	117	4.9%	6.1	4.6	76%	2380	122	5.1%	238	12	5.1%
		Test Coupons	1087	272	25%	23	41	179%	1110	255	23%	111	25	23%
	Aluminum	Positive Controls	2193	458	21%	5.7	5.5	97%	2199	462	21%	220	46	21%
		Test Coupons	1011	248	25%	6.6	8.8	134%	1017	250	25%	102	25	25%
TDA $eClO_2$	ABS Plastic	Positive Controls	903	178	20%	620	93	15%	1524	241	16%	152	24	16%
		Test Coupons	320	38	12%	647	125	19%	967	120	12%	97	12	12%
	Acrylic	Positive Controls	2354	77	3.3%	37	37	101%	2390	46	1.9%	239	4.6	1.9%
		Test Coupons	889	185	21%	32	31	96%	921	172	19%	92	17	19%
	Aluminum	Positive Controls	2203	121	5.5%	24	23	96%	2227	125	5.6%	223	13	5.6%
		Test Coupons	1366	176	13%	2.6	0.22	8.3%	1369	177	13%	137	18	13%

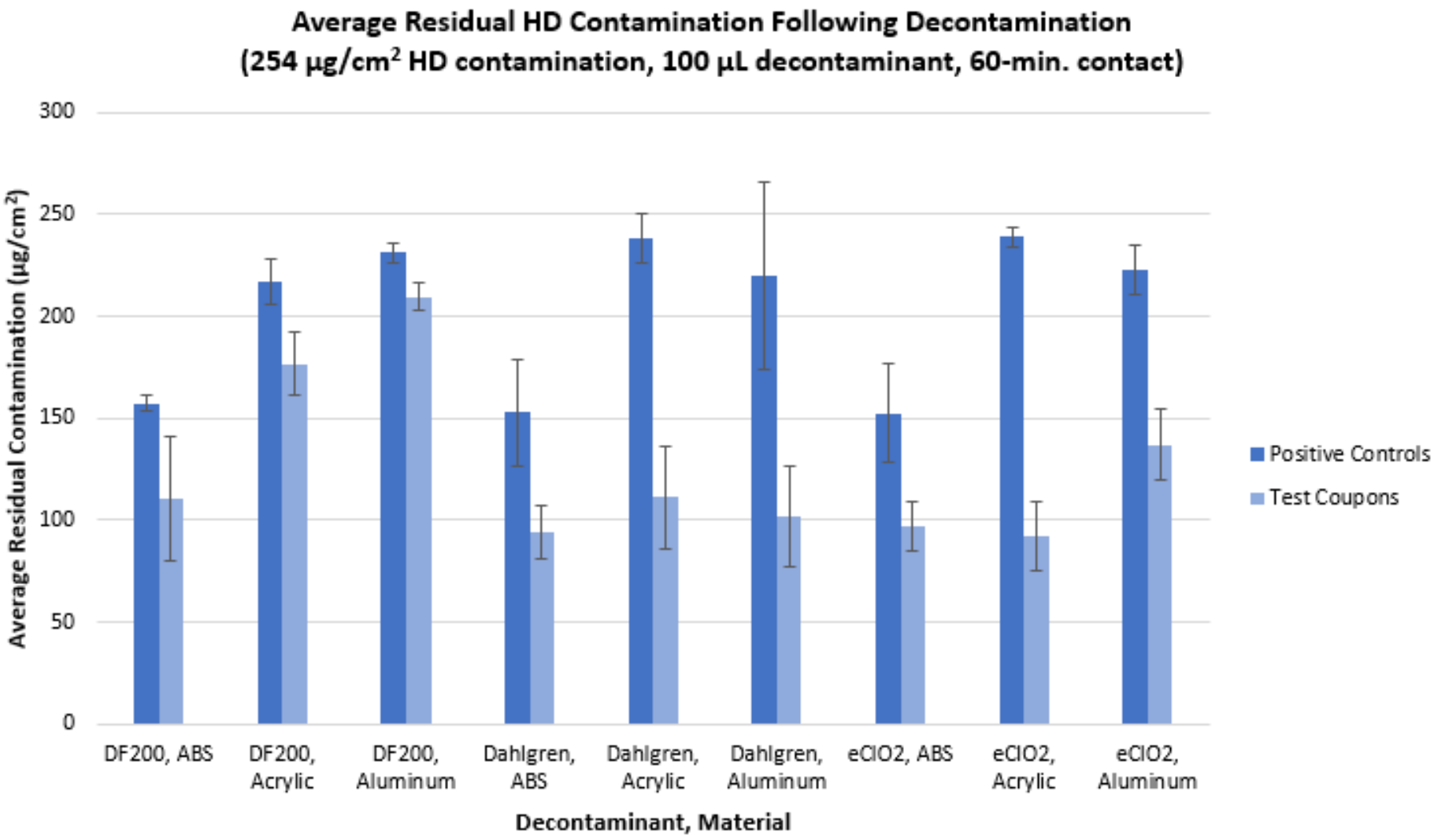


Figure 6. Average Residual HD Contamination
(Error bars equal ± one standard deviation)

3.2.3 VX and HD Decontamination Efficacy

Average percent decontamination efficacies for each CWA/decontaminant/SE-related material combination are provided in Table 21.

Table 21. Average Percent Decontamination Efficacies

Decontaminant	Material	Avg. VX Decontamination Efficacy (%)	Avg. HD Decontamination Efficacy (%)
EasyDECON DF200	ABS Plastic	83%	29%
	Acrylic	98%	19%
	Aluminum	97%	9.4%
Dahlgren Decon	ABS Plastic	99%	38%
	Acrylic	99%	53%
	Aluminum	98%	54%
TDA $eClO_2$	ABS Plastic	4.4%	37%
	Acrylic	29%	61%
	Aluminum	30%	39%

Dahlgren Decon achieved the highest efficacy for VX, with efficacy values of 99% from ABS plastic and acrylic and 98% from aluminum. EasyDECON DF200 VX decontamination efficacy was only slightly lower at 98% from acrylic, 97% from aluminum, and 83% from ABS plastic. The TDA $eClO_2$ decontaminant demonstrated lower VX decontamination efficacy, measuring 30% from aluminum, 29% from acrylic, and only 4.4% from ABS plastic. VX decontamination efficacies are summarized in Figure 7.

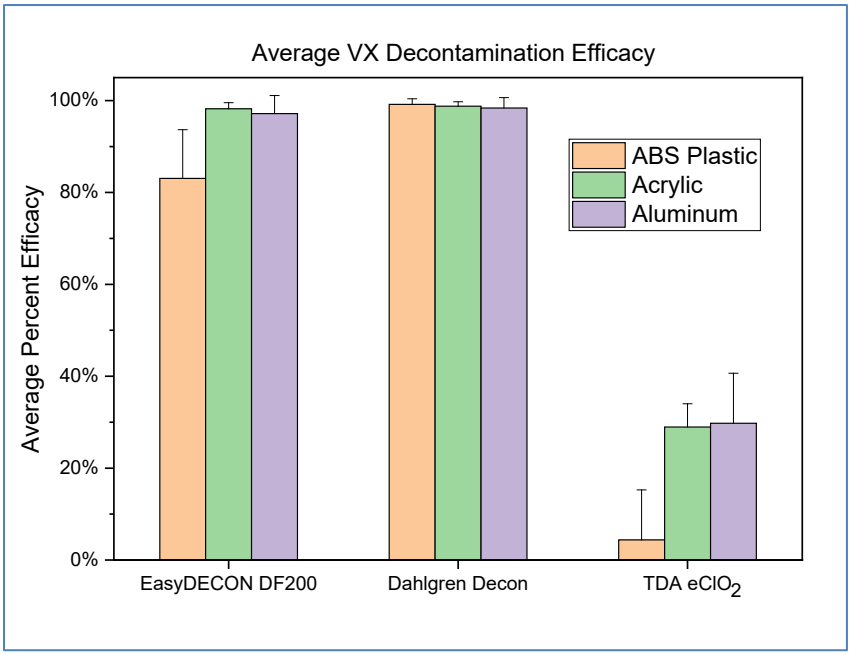


Figure 7. Average VX Decontamination Efficacy

EasyDECON DF200 and Dahlgren Decon demonstrated lower efficacy in decontamination of HD, ranging from only 54% (Dahlgren Decon decontamination of HD from aluminum) to only 9.4% (EasyDECON DF200 decontamination of HD from aluminum). Conversely, the TDA $eClO_2$ decontaminant demonstrated generally higher efficacy in decontamination of HD than of VX. HD decontamination efficacy for the $eClO_2$ decontaminant ranged from 37% from ABS plastic to 61% from acrylic, which notably was the highest measured HD decontamination efficacy. HD decontamination efficacies are summarized in Figure 8.

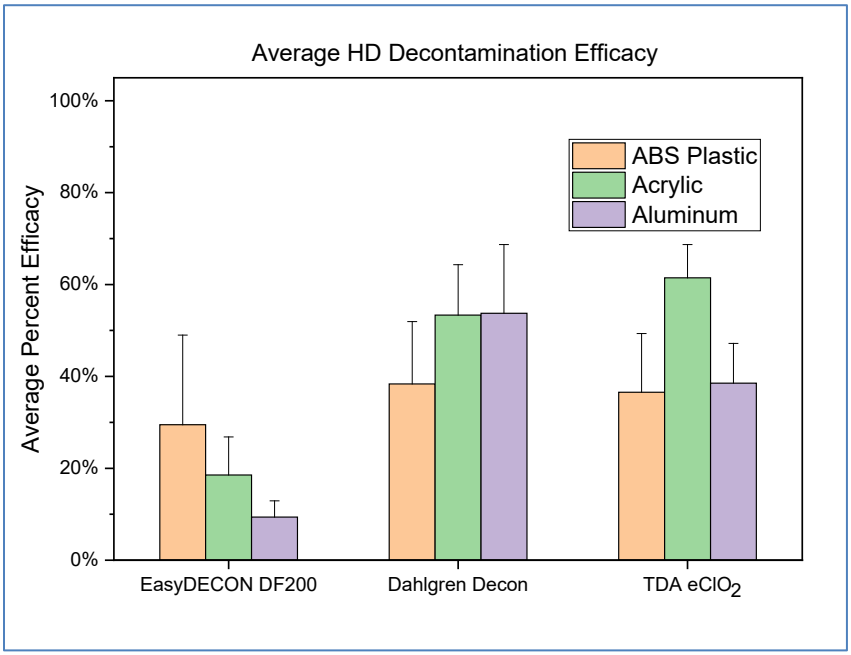


Figure 8. Average HD Decontamination Efficacy

3.3 ANOVA Results

3.3.1 Comparison of Positive Control Results

Tables 22 through 27 present the geometric mean total recoveries of the positive controls for each CWA/material/decontaminant combination and any significant differences in geometric means identified from Tukey-adjusted comparisons based on ANOVA models. The character in the "Similarity Designation" column indicates the statistical similarity of the geometric mean total mass recovery of a decontaminant to the geometric mean total mass recovery of the other decontaminants within the material/CWA combination (e.g., in Table 26, DF200 and $eClO_2$ are statistically similar and thus both designated "A", $eClO_2$ and Dahlgren Decon are statistically similar and thus both designated "B", DF200 and Dahlgren Decon are not statistically similar).

There was only one CWA/material combination with a significant pairwise comparison: for VX/acrylic, the positive control geometric mean total mass recovery for DF200 was significantly less than the positive control geometric mean total mass recovery for Dahlgren Decon. Considering that the random probability of measuring a significant difference when none truly exists is 0.05, one significant difference out of 18 pairwise comparisons (three comparisons for each of the six CWA/material combinations) is not enough evidence to say there was a difference between the sets of positive controls within each CWA/material combination. In this case, the range of standard deviations for positive control total mass recovery for the three VX/acrylic tests was smaller than the ranges for any other CWA/material combinations tested (44 to 70 µg; the next narrowest range was 46 to 122 µg for HD/acrylic). This smaller variability in geometric mean total mass recovery for the three positive control sets for VX/acrylic makes the comparisons more sensitive, and smaller differences in the geometric means of the three sets are thus identified as statistically significant.

While the statistical comparisons that were performed identified that a significantly lesser amount of VX (subjectively small, but nonetheless statistically significant based on the analysis) was applied to the DF200 positive controls than to the Dahlgren Decon positive controls (i.e., more VX was applied to the Dahlgren Decon positive controls), average VX decontamination efficacy for Dahlgren Decon from acrylic was calculated at 99%, while average VX decontamination efficacy for DF200 from acrylic was calculated at 98%. Comparison of the geometric mean total mass recoveries for the VX/acrylic/DF200 and VX/acrylic/Dahlgren Decon test coupon sets did not determine that the sets are significantly different (suggesting no performance difference between the two decontaminants for VX/acrylic). However, given the considerations discussed above, it is not anticipated that a significant difference in performance between the two decontaminants would have been identified if the positive control geometric mean total mass recoveries were *not* identified as different.

Table 22. ANOVA Results for ABS Plastic with HD (Positives)

Decontaminant	Similarity Designation	Positive Control Geometric Mean Total Mass Recovery (µg)	Tukey-Adjusted p-Value *
$eClO_2$	A	1511	No significant differences.
Dahlgren Decon	A	1513	
DF200	A	1571	

* There were no significant differences between any pairs of decontaminants.

Table 23. ANOVA Results for Acrylic with HD (Positives)

Decontaminant	Similarity Designation	Positive Control Geometric Mean Total Mass Recovery (µg)	Tukey-Adjusted p-Value *
DF200	A	2168	No significant differences.
Dahlgren Decon	A	2378	
$eClO_2$	A	2390	

* There were no significant differences between any pairs of decontaminants.

Table 24. ANOVA Results for Aluminum with HD (Positives)

Decontaminant	Similarity Designation	Positive Control Geometric Mean Total Mass Recovery (µg)	Tukey-Adjusted p-Value *
Dahlgren Decon	A	2164	No significant differences.
$eClO_2$	A	2225	
DF200	A	2313	

* There were no significant differences between any pairs of decontaminants.

Table 25. ANOVA Results for ABS Plastic with VX (Positives)

Decontaminant	Similarity Designation	Positive Control Geometric Mean Total Mass Recovery (µg)	Tukey-Adjusted p-Value *
DF200	A	1792	No significant differences.
Dahlgren Decon	A	1861	
$eClO_2$	A	1988	

* There were no significant differences between any pairs of decontaminants.

Table 26. ANOVA Results for Acrylic with VX (Positives)

Decontaminant	Similarity Designation	Positive Control Geometric Mean Total Mass Recovery (µg)	Tukey-Adjusted p-Value *
DF200	A	1828	0.0374 (DF200 < Dahlgren Decon)
$eClO_2$	AB	1960	
Dahlgren Decon	B	1983	

* Pairwise comparisons that were significant at the 0.05 level. The format within each cell is: (1) the Tukey-adjusted p-value, and (2) the relationship between the corresponding pair of decontaminants shown in parentheses.

Table 27. ANOVA Results for Aluminum with VX (Positives)

Decontaminant	Similarity Designation	Positive Control Geometric Mean Total Mass Recovery (µg)	Tukey-Adjusted p-Value *
DF200	A	1865	No significant differences.
$eClO_2$	A	1978	
Dahlgren Decon	A	2001	

* There were no significant differences between any pairs of decontaminants.

3.3.2 Comparison of Test Sample Results

Tables 28 through 33 present the geometric means of the ANOVA models for the decontaminants for each material/CWA combination ordered from lowest to highest, along with the significant Tukey-adjusted comparisons. As in Tables 22 through 27, the character in the "Similarity Designation" column indicates the statistical similarity of the geometric mean total mass recovery of a decontaminant to the geometric mean total mass recovery of the other decontaminants within the material/CWA combination. Simply based on the ordering, DF200 consistently had the largest geometric mean total mass recovery across all three materials for HD. For VX, Dahlgren Decon consistently had the smallest geometric mean total mass recovery while $eClO_2$ consistently had the largest geometric mean total mass recovery across all three materials.

There were no significant differences between decontaminants for ABS plastic and acrylic materials with HD. For aluminum with HD and ABS plastic with VX, the geometric mean total mass recovery for Dahlgren Decon was significantly less than the geometric mean total mass recovery for DF200. For ABS plastic, acrylic, and aluminum with VX, the geometric mean total mass recovery for Dahlgren Decon was significantly less than the geometric mean total mass recovery for $eClO_2$. For acrylic and aluminum with VX, the geometric mean total mass recovery for DF200 was significantly less than the geometric mean total mass recovery for $eClO_2$.

Table 28. ANOVA Results for ABS Plastic with HD (Test Samples)

Decontaminant	Similarity Designation	Geometric Mean Total Mass Recovery (µg)	Tukey-Adjusted p-Value *
$eClO_2$	A	891	No significant differences.
Dahlgren Decon	A	985	
DF200	A	1207	

* There were no significant differences between any pairs of decontaminants.

Table 29. ANOVA Results for Acrylic with HD (Test Samples)

Decontaminant	Similarity Designation	Geometric Mean Total Mass Recovery (µg)	Tukey-Adjusted p-Value *
$eClO_2$	A	1039	No significant differences.
Dahlgren Decon	A	1179	
DF200	A	1682	

* There were no significant differences between any pairs of decontaminants.

Table 30. ANOVA Results for Aluminum with HD (Test Samples)

Decontaminant	Similarity Designation	Geometric Mean Total Mass Recovery (µg)	Tukey-Adjusted p-Value *
Dahlgren Decon	A	1127	0.0345 (Dahlgren Decon < DF200)
$eClO_2$	AB	1469	
DF200	B	2055	

* Pairwise comparisons that were significant at the 0.05 level. The format within each cell is: (1) the Tukey-adjusted p-value, and (2) the relationship between the corresponding pair of decontaminants shown in parentheses.

Table 31. ANOVA Results for ABS Plastic with VX (Test Samples)

Decontaminant	Similarity Designation	Geometric Mean Total Mass Recovery (µg)	Tukey-Adjusted p-Value *
Dahlgren Decon	A	14	<0.0001 (Dahlgren Decon < $eClO_2$) 0.0060 (Dahlgren Decon < DF200)
DF200	B	286	
$eClO_2$	B	1435	

* Pairwise comparisons that were significant at the 0.05 level. The format within each cell is: (1) the Tukey-adjusted p-value, and (2) the relationship between the corresponding pair of decontaminants shown in parentheses.

Table 32. ANOVA Results for Acrylic with VX (Test Samples)

Decontaminant	Similarity Designation	Geometric Mean Total Mass Recovery (µg)	Tukey-Adjusted p-Value *
Dahlgren Decon	A	17	0.0015 (Dahlgren Decon < $eClO_2$) 0.0399 (DF200 < $eClO_2$)
DF200	A	43	
$eClO_2$	B	635	

* Pairwise comparisons that were significant at the 0.05 level. The format within each cell is: (1) the Tukey-adjusted p-value, and (2) the relationship between the corresponding pair of decontaminants shown in parentheses.

Table 33. ANOVA Results for Aluminum with VX (Test Samples)

Decontaminant	Similarity Designation	Geometric Mean Total Mass Recovery (µg)	Tukey-Adjusted p-Value *
Dahlgren Decon	A	14	0.0005 (Dahlgren Decon < $eClO_2$) 0.0049 (DF200 < $eClO_2$)
DF200	A	20	
$eClO_2$	B	842	

* Pairwise comparisons that were significant at the 0.05 level. The format within each cell is: (1) the Tukey-adjusted p-value, and (2) the relationship between the corresponding pair of decontaminants shown in parentheses.

3.4 Material Compatibility

As described in Section 2.1.3, compatibility of the decontaminants with the SE-related materials was assessed qualitatively (visually) during decontamination efficacy testing. Test coupons and procedural blanks were visually inspected and compared to other coupons of the same SE-related material types that were not exposed to the decontamination technologies.

Also, as described in Section 2.1.3, additional procedural blanks were included during decontamination efficacy tests to which decontaminants were applied, but the blanks were not wiped or extracted following the 60-minute decontaminant dwell period. Rather, the decontaminant was allowed to remain on the coupon surface for one week to assess the effect of extended decontaminant contact with the SE-related materials. Tables 34 through 36 depict the additional procedural blanks at the time of decontaminant application, after one day of contact with the decontaminants, one week of contact with the decontaminants, and then following

wiping of the coupon surface with hexane-soaked and/or water-soaked wipes to assess the possibility of and effort involved with removal of any remaining decontaminant or residue.

Acrylic appeared to be unaffected by extended contact with any of the three decontaminants. After one week, Dahlgren Decon was still visibly wet/puddled on the surface of the acrylic coupon, while DF200 and the TDA $eClO_2$ decontaminant had evaporated and left a white, crystallized residue. In all three cases, however, remaining decontaminant/residue was completely removed via wiping with a hexane-soaked wipe, leaving no observable damage or deterioration.

Results similar to the results obtained for acrylic were observed for the ABS plastic blanks. Liquid Dahlgren Decon was still visible on the coupon surface following one week, while DF200 and $eClO_2$ had evaporated, leaving a "crusty" white residue. Residual decontaminant and dried residue were easily removed from the surface of all three material types using a hexane-soaked wipe, but all three decontaminants were found to have slightly discolored the ABS plastic coupon.

Aluminum was generally affected the most by extended contact with the three decontaminants. Like the ABS plastic, only very slight discoloration of aluminum was observed following one week of contact with Dahlgren Decon. DF200 and the TDA $eClO_2$ decontaminant did not demonstrate the same degree of compatibility, however. Both DF200 and $eClO_2$ left residues on aluminum that were not easily removed using either hexane or water-soaked wipes, and the $eClO_2$ even left the surface of the aluminum coupon visibly discolored and pitted.

On all three materials, Dahlgren Decon remained wet even after the one-week dwell period. Some evaporation may have occurred, as the Dahlgren Decon remaining on the surface of the coupons seemed thicker/tackier than when initially applied, but this observation is in contrast to the other two decontaminants that had both completely evaporated from all three materials after one week, leaving behind dried residues.

In summary, Dahlgren Decon appeared to demonstrate the highest degree of compatibility with the three SE-related materials included in this evaluation. Residual decontaminant was easily wiped from the surface of all three material types, leaving no lasting observable effects on acrylic and only very slight discoloration of ABS plastic and aluminum. In contrast, DF200 and $eClO_2$ discolored ABS plastic to a greater degree and left residues on aluminum that were not easily removed and/or actual physical damage to/deterioration of the aluminum coupon surface ($eClO_2$).

Table 34. Dahlgren Decon Material Compatibility

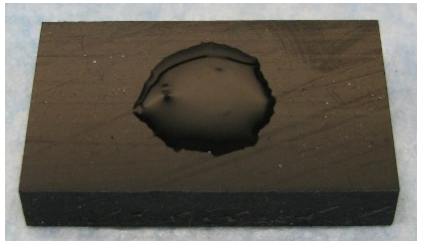
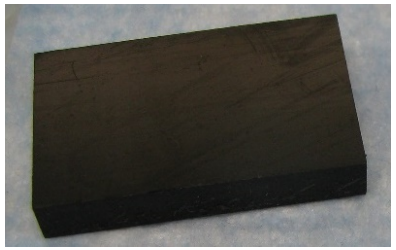
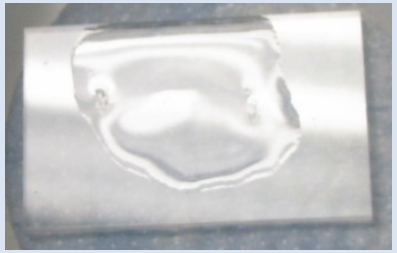
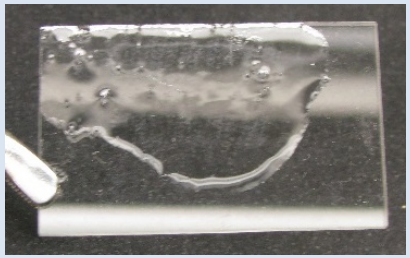
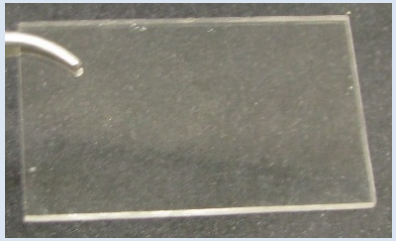
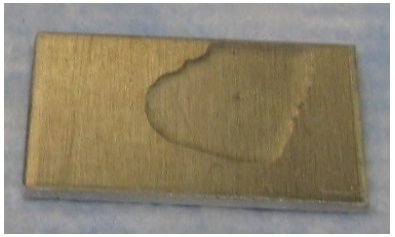
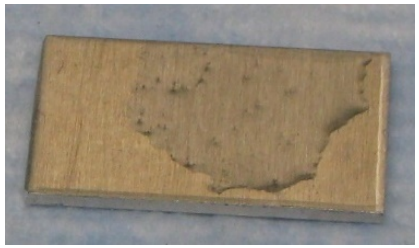
Material	Following Application	After One Day	After One Week	Following Wiping/Removal
ABS Plastic	Decontaminant pooled on coupon.	Decontaminant still appeared wet.	 Decontaminant still wet but appeared thicker/tackier when wiped.	 Decontaminant easily removed with hexane-soaked wipe. **Very slight discoloration where decon dwelled.**
Acrylic	Decontaminant pooled on coupon.	 Decontaminant still appeared wet	 Decontaminant still wet but appeared thicker/tackier when wiped.	 Decontaminant easily removed with hexane-soaked wipe. **No deterioration of material observed.**
Aluminum	Decontaminant pooled on coupon.	 Decontaminant still appeared wet.	 Decontaminant still wet but appeared thicker/tackier when wiped.	Decontaminant easily removed with hexane wipe. **Very slight discoloration where decon dwelled.**

Table 35. EasyDECON DF200 Material Compatibility

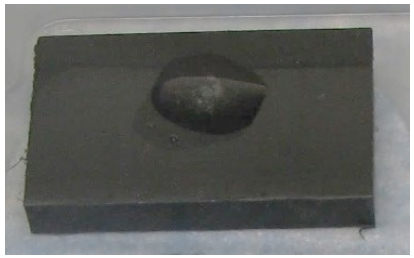
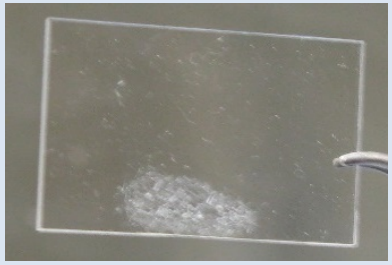
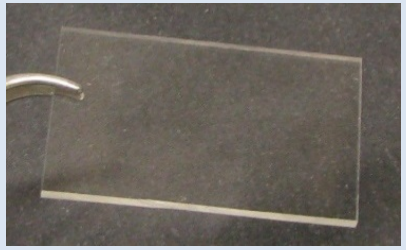
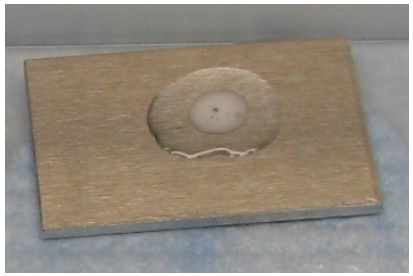
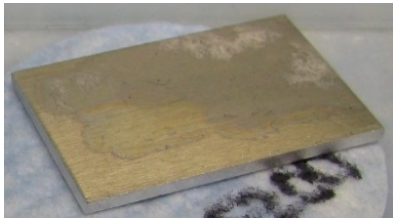
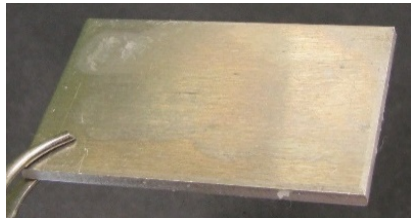
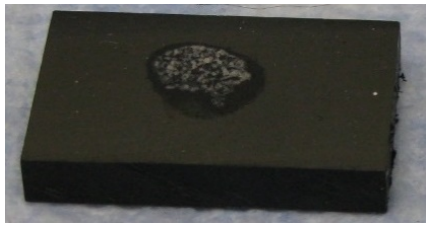
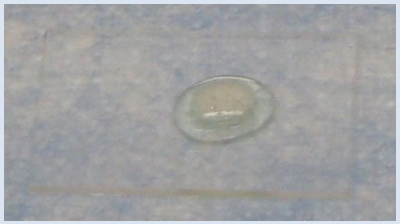
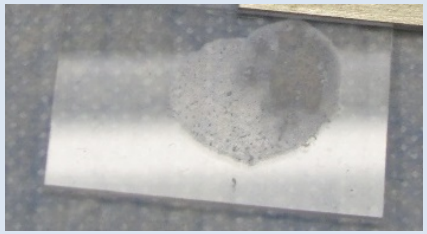
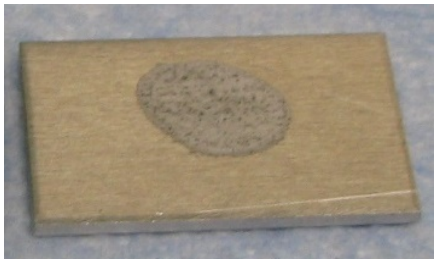
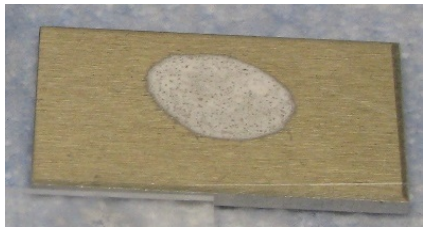
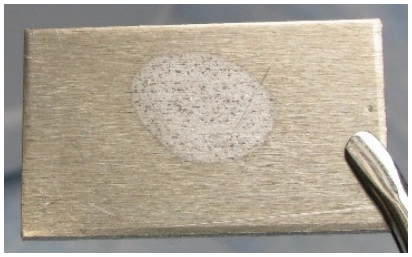
Material	Following Application	After One Day	After One Week	Following Wiping/Removal
ABS Plastic	 Decontaminant pooled on coupon.	Decontaminant appeared slightly wet/crystallized.	Crystallized/crusty residue remaining.	Residue easily removed with hexane-soaked wipe. **Discoloration where decontaminant dwelled.**
Acrylic	Decontaminant pooled on coupon.	Decontaminant spread and appeared slightly wet/crystallized.	 Crystallized/crusty residue remaining.	 Residue easily removed with hexane-soaked wipe. **No deterioration of material observed.**
Aluminum	 Decontaminant pooled on coupon.	Decontaminant spread and appeared dry/crystallized.	 Crystallized/crusty residue remaining.	 Residue not easily removed with either hexane-soaked or water-soaked wipe. **Discoloration/white residue present where decontaminant dwelled.**

Table 36. TDA Research HDA eClO$_2$ Material Compatibility

Material	Following Application	After One Day	After One Week	Following Wiping/Removal
ABS Plastic	Decontaminant pooled on coupon.	Decontaminant still appeared wet.	 White residue remaining.	Residue easily removed with hexane-soaked wipe. **Very slight discoloration where decontaminant dwelled.**
Acrylic	 Decontaminant pooled on coupon.	Decontaminant still appeared wet.	 White residue remaining.	Residue easily removed with hexane-soaked wipe. **No deterioration of material observed.**
Aluminum	Decontaminant pooled on coupon.	 Decontaminant appeared slightly wet/crystallized.	 White residue remaining.	 Residue not easily removed with either hexane-soaked or water-soaked wipe. **Discoloration/possible pitting present where decontaminant dwelled.**

Damage to coupons due to contact with the CWAs was also assessed visually. Neither VX nor HD appeared to damage or degrade the materials following the 60-minute CWA dwell period, with one exception: HD was observed to damage/deteriorate the surface of ABS plastic coupons. Figures 9 through 11 depict damage to ABS plastic coupons caused by contact with neat HD. The pictures below depict ABS plastic coupons included in the HD wipe sampling and coupon solvent extraction methods development test (procedures described in Section 2.1.1.1).

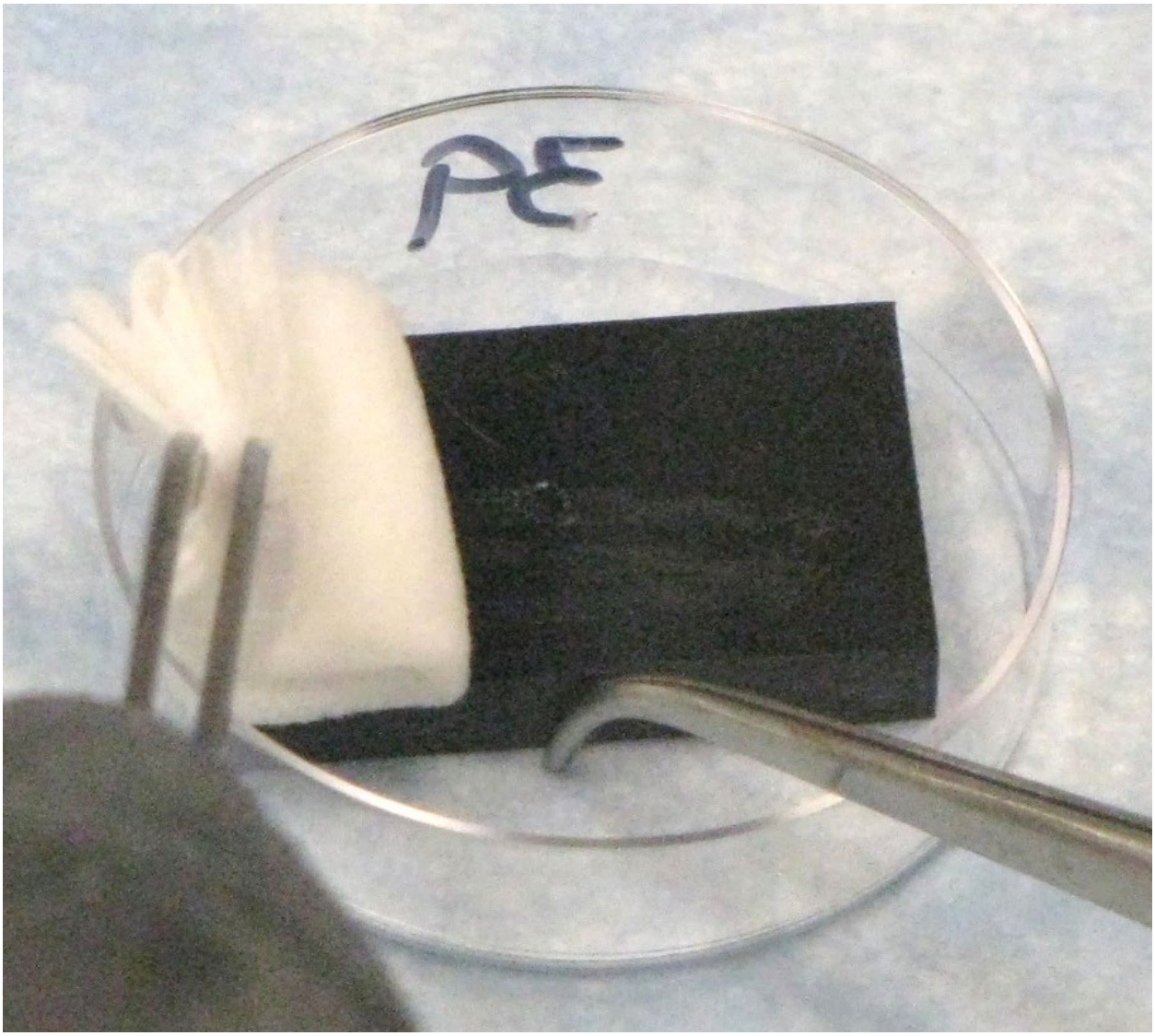


Figure 9. Damage to ABS plastic caused by HD (1).

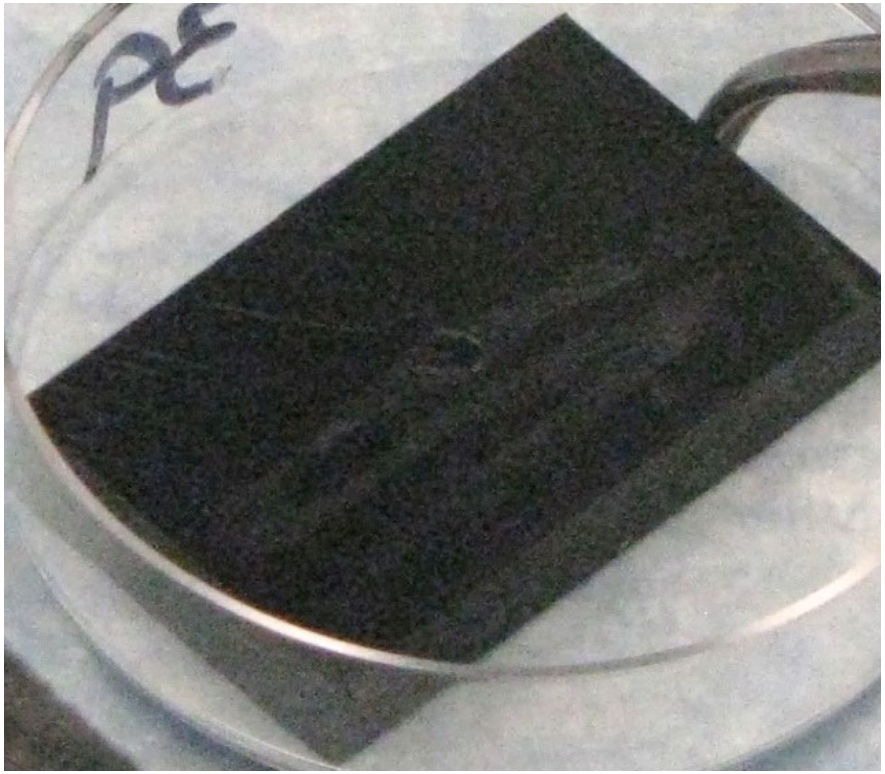


Figure 10. Damage to ABS plastic caused by HD (2).

Figure 11. Damage to ABS plastic caused by HD (3).

Figures 12 and 13 depict an ABS plastic coupon and wipe sample included in the decontamination efficacy test evaluating decontamination of HD by TDA's $eClO_2$ HDA decontaminant. As the pictures show, the $eClO_2$ decontaminant does not appear to have affected the coupon material, but a prominent pit is present in the coupon surface where the HD was applied. Additionally, ABS plastic debris can be seen on the wipe sample taken from the coupon (Figure 13).

Figure 12. Damage to ABS plastic caused by HD (4).

Figure 13. ABS plastic debris on wipe sample.

As discussed in Section 3.1.1, during the HD wipe sampling and coupon solvent extraction method development test, total HD mass recoveries from ABS coupons were generally lower than the total HD mass recoveries from acrylic and aluminum (75% average total mass recovery from ABS plastic, versus 97% from both acrylic and aluminum). Furthermore, approximately two-thirds of the HD recovered from ABS coupons was recovered from the wipe, with the remaining HD obtained from extraction of the coupons. This recovery scheme is in contrast to the acrylic and aluminum coupons, which demonstrated recovery of nearly all HD in the wipe sample with low or no recovery from the coupon solvent extraction (only one acrylic coupon extraction sample was above the GC/MS detection limit, with the other two samples less than the detection limit; no measurable HD was recovered from extraction of the aluminum coupons).

ABS plastic coupons that were extracted in solvent only (no prior wiping) demonstrated only 44% average recovery during the wipe sampling and solvent extraction method development test, versus the 75% average total mass recovery from ABS plastic coupons that were sampled via both wiping and coupon solvent extraction. This scenario possibly suggests that HD is absorbed into the ABS plastic material, becoming unrecoverable by solvent extraction alone. The act of wiping then disrupts the damaged ABS plastic coupon surface, allowing absorbed HD to be accessed and recovered by wiping and solvent extraction.

4. QUALITY ASSURANCE/QUALITY CONTROL

4.1 Control of Monitoring and Measuring Devices

Quality control requirements and results are provided in Table 37. In general, the data quality indicator results were acceptable, including check of the measurement methods for temperature, RH, time, volume, IS response, and VX recovery from spike controls and blank samples. Attainment of these data quality indicator results limited the amount of error introduced into the investigation results.

Table 37. Quality Control Requirements and Results

Parameter	Measurement Method	Data Quality Indicators	Results
Temperature (°C)	Calibrated HOBO UX100 Data Logger	Compare against NIST-traceable calibrated thermometer once before testing; agree ±1 °C through one hour.	The HOBO UX100 Data logger reading remained within 0.6 °C of the NIST-traceable calibrated reference through one hour.
Relative Humidity (%)	Calibrated HOBO UX100 Data Logger	Compare against NIST-traceable calibrated hygrometer once before testing; agree ±10% through one hour.	The HOBO UX100 Datalogger reading remained within 5.4% of the NIST-traceable calibrated reference through one hour.
Time (sec)	Timer	Compare to time provided at NIST.time.gov once before testing; agree ±2 seconds/hour.	No difference was observed between the timer and NIST.time.gov after one hour.
Volume (mL, µL)	Syringe (CWA delivery) Calibrated pipette (decon delivery)	Syringes/pipettes were checked for accuracy and repeatability one time before use by determining the mass of water delivered. The syringe/pipette was acceptable if the range of observed masses for five droplets was ±10% of expected.	100 µL syringe verification – Percent difference across five measurements ranged from 0.24% to 5.25%. Pipette verification - Percent difference across five measurements ranged from 0.57% to 1.07%.
CWA amount	Extraction, GC/MS	Calibration curve with linear or quadratic regression (coefficient of determination [r2] ≥ 0.990)	Calibration curves were created at the beginning of each batch of test samples. Curves that did not pass criteria were re-run.
Naphthalene-d_8 IS Recovery	Extraction, GC/MS	The mean of the IS included with each day of testing will be within 50% to 120% of the expected mass.	IS response of each sample was compared to the mid-point standard. Anything outside the specification was flagged and re-run.

4.2 Equipment Calibrations

4.2.1 Calibration Procedures and Schedules

Instrumentation was maintained and operated according to the quality requirements and documentation of Battelle's HMRC. All equipment was calibrated with appropriate standards. Table 38 provides calibration schedules for instruments that were used during the evaluation.

Table 38. Equipment Calibration Schedule

Equipment	Frequency
Calibrated pipette and repeating dispenser/syringe	Prior to the investigation. Calibration/accuracy was verified as described in Table 37.
Calibrated UX100 HOBO Hygrometer/Thermometer	Calibrated by the manufacturer, and calibration was verified against a separate, NIST-traceable calibrated instrument once before use during testing as described in Table 37.
Timer	Calibrated by the manufacturer, and calibration was verified against NIST.time.gov once before use during testing as described in Table 37.
GC/MS	Beginning of each batch of test samples (calibration curve) and a calibration verification standard after every five samples and at the end of a batch of samples (see Section 4.2.2).

4.2.2 GC/MS Calibration

Neat VX or HD (concentrations corrected for percent purity; see Section 2.3.2.1) was used to create calibration standards encompassing the appropriate analysis range. Calibration standards were kept and used for no longer than six months from the date of creation.

GC/MS calibration ranged from 0.1 µg/mL to 125 µg/mL. To cover the entire analysis range, two overlapping five-point calibration curves were used (a "low curve" from 0.1 µg/mL to 10 µg/mL, and a "high curve" from 5.0 µg/mL to 125 µg/mL). A linear or quadratic regression (coefficient of determination [r^2] ≥ 0.990) curve fit was applied to the calibration data. The GC/MS was recalibrated if the r^2 from the regression analysis of these standards was less than 0.990. Limits were also placed on the percent bias (Equation 5) observed in the standards.

$$Bias = \left(\frac{E_V - O_V}{E_V}\right) \times 100\% \qquad (5)$$

where: E_V = expected value from calibration curve

O_V = observed value from standard

The percent bias for the low standard had to be less than or equal to 25%, and the percent bias for the remaining standards had to be less than or equal to 15%.

The GC/MS was tuned initially and then as needed following manufacturer's guidelines. A tune check was performed before running each set of samples using decafluorotriphenylphosphine (DFTPP). A 12-hour tune time was not employed.

Following analysis of the calibration standards at the beginning of each analytical run, a solvent blank sample was analyzed to confirm that no VX or HD carryover was occurring. Solvent blank sample analysis results had to be below the value of the lowest calibration standard.

Independently prepared CCV standards were analyzed prior to sample analysis, following every five test/control samples and at the end of each set of samples. Two CCV concentrations were used, one equal to the low calibration standard and the other within the calibration range (5.0 µg/mL for the low curve and 50 µg/mL for the high curve). CCV response had to be within 35%

of the nominal concentration for the low level CCV and within 20% of the nominal concentration for the higher level CCV for VX or HD to be acceptable. Samples analyzed prior to or following CCVs that were outside acceptance limits were re-analyzed (either within the same analytical run or during a separate run for which a new calibration curve was established; results from reanalysis were considered valid and reportable if analysis quality control objectives, as described above for calibration curve standards and bracketing CCVs, were met). CCV standards were kept and used for no longer than one month from the date of creation. GC analysis performance parameter and acceptance criteria are provided in Table 39.

Table 39. GC Performance Parameters and Acceptance Criteria

Parameter	Criterion
Coefficient of determination (r^2)	≥ 0.990
% bias for the lowest calibration standard	≤ 25%
% bias for remaining calibration standards (except lowest standard)	≤ 15%
Solvent blank sample	< lowest calibration standard
% bias for the lowest CCV	≤ 35%
% bias for remaining CCVs (except lowest CCV)	≤ 20%

4.3 Technical Systems Audit

The Quality Assurance (QA) Officer performed a technical systems audit (TSA) at the HMRC facility in West Jefferson, Ohio, during decontamination efficacy testing on February 20, 2018. The purpose of the TSA was to ensure that testing was performed in accordance with the quality assurance test plan. The QA Officer reviewed the investigation methods, compared test procedures to those specified in the quality assurance test plan, and reviewed data acquisition and handling procedures. The QA Officer did not identify any findings that required corrective action.

4.4 Performance Evaluation Audit

Performance evaluation (PE) audits, provided in Table 40 with results, addressed those reference measurements that factored into the data used in quantitative analysis during the evaluation, including volume and time measurements and GC/MS calibration and performance. The volume of VX or HD dispensed correlated directly to the mass of each CWA on the coupons. The measured times that CWA and the decontamination technologies were allowed to remain in contact with the coupons directly influenced efficacy of the decontaminants. Calibration of the GC/MS and IS recovery provided confidence that the analysis system was providing accurate data.

Temperature and RH were monitored and recorded on each day of testing, but not controlled. Therefore, no PE audit of these parameters was performed.

Table 40. Performance Evaluation Audit Results

Parameter	Audit Procedure	Required Tolerance	Results
Volume (mL, µL)	Syringes/pipettes were checked for accuracy and repeatability one time before use by determining the mass of water delivered.	The syringe/pipette will be acceptable if the range of observed masses for five droplets is ±10% of expected.	100 µL syringe verification – Percent difference across five measurements ranged from 0.24% to 5.25%. Pipette verification - Percent difference across five measurements ranged from 0.57% to 1.07%.
Time (sec)	Compared to time provided at NIST.time.gov once before testing.	±2 sec/hour	No difference was observed between the timer and NIST.time.gov after one hour.
CWA in Spike Control Extracts (µg/mL)	Used GC/MS to determine mass of CWA delivered as 2 µL droplet into 25 mL of extraction solvent and compared to target application level.	≥80% of spike target ≤ 120% of spike target ≤ 30% CoV	Spike control means throughout testing were within specification except for two instances (refer to Table 37).
GC/MS VX and HD Calibration Standards (%)	Verified all standards and CCVs used to calibrate and confirm calibration of the GC/MS system used for analysis during the project fell within the requirements provided in Section 4.2.2.	Refer to Table 39	All standards and CCVs were within specification for all reported data.
Naphthalene-d_8 IS Recovery	Used GC/MS to measure from a secondary source and compare to the primary source one time.	±10% relative percent difference	0.6% relative percent difference

4.5 Data Quality Audit

The QA Manager audited at least 10% of the investigation data and traced the data from initial acquisition, through reduction and statistical comparisons, to final reporting. All data analysis calculations were checked. The QA Officer did not identify any findings that required corrective action.

5. SUMMARY

The primary objective of this project was to quantitatively evaluate efficacy of three candidate decontamination technologies to decontaminate CWAs from select SE-related materials and to concurrently evaluate qualitatively the effects of the decontaminants on the integrity of the materials to which they were applied. Bench scale decontamination efficacy tests using neat VX and HD and the SE-related materials ABS molded plastic, acrylic, and aluminum were performed. The decontaminants evaluated were Dahlgren Decon from First Line Technology, EasyDECON DF200 from Intelagard®, and the TDA HDA $eClO_2$ decontaminant. During the decontamination efficacy evaluation, compatibility of the decontamination technologies with the SE-related materials was evaluated through visual assessment of the decontaminated materials and documentation of any visible deterioration or damage caused to the materials by application of the decontamination technologies.

Prior to evaluation of decontamination efficacy, method demonstration testing was conducted to evaluate the effectiveness of the coupon surface wipe-sampling and coupon solvent extraction methods planned for use during testing. Using hexane as the wipe-wetting and wipe and coupon extraction solvent, the requirement that recovery of each CWA from the three SE-related materials fall within the range of 70% to 120% with ≤ 30% RSD was achieved. VX recoveries from ABS plastic, acrylic, and aluminum measured 107%, 120%, and 119%, respectively. Recoveries of 75% from ABS plastic, 97% from acrylic, and 97% from aluminum were measured for HD. To improve analytical sensitivity, a VX stabilizer, N,N'-diisopropylcarbodiimide, was added to the wipe and coupon extraction solvent. Naphthalene-d_8 was used as the IS.

Additionally, methods for halting the decontamination reactions after a predefined interval (quenching) were evaluated. The quench methods tested included: (1) solvent extraction in hexane alone and (2) extraction in hexane with a 3M solution of STS added. The quench methods selected for each CWA/decontaminant combination for use during decontamination efficacy testing were:

- HD by EasyDECON DF200: Extraction in hexane alone (78% recovery of post-spiked HD in wipe extracts, 98% in coupon extracts)
- HD by Dahlgren Decon: 3M STS added to extraction solvent (100% recovery of post-spiked HD in wipe extracts, 100% in coupon extracts)
- HD by TDA $eClO_2$: Extraction in hexane alone (97% recovery of post-spiked HD in wipe extracts, 100% in coupon extracts)
- VX by EasyDECON DF200: 3M STS added to extraction solvent (89% recovery of post-spiked HD in wipe extracts, 91% in coupon extracts)
- VX by Dahlgren Decon: 3M STS added to extraction solvent (101% recovery of post-spiked HD in wipe extracts, 101% in coupon extracts)

- VX by TDA $eClO_2$: Extraction in hexane alone (101% recovery of post-spiked HD in wipe extracts, 97% in coupon extracts).

Coupons of each of the SE-related materials measuring 4.0 cm by 2.5 cm (10 cm^2 surface area) were each spiked with 2 µL of VX or HD. After a 60-minute CWA dwell period, 100 µL of one of the three test decontaminants was applied to the coupon surface, on top of the CWA contamination. The decontaminant was then allowed to react with CWA on the coupon surfaces for 60 minutes. Following the decontamination period, coupons were wipe-sampled and subsequently extracted in solvent. Wipe and coupon extracts were then analyzed by GC/MS to quantify residual CWA on the coupon surface following decontamination. Associated quantitation limits were 0.25 µg/cm^2 (2.5 µg residual CWA mass per coupon). Positive controls were included that consisted of SE-related material coupons that were spiked with CWA using the same equipment and procedures as the test coupons, but to which no decontaminants were applied.

Percent efficacy of each decontaminant was calculated as:

$$Efficacy = \left(\frac{Cont_{ResPos} - Cont_{ResTest}}{Cont_{ResPos}}\right) \times 100\%$$

where: $Cont_{ResTest}$ = Residual test coupon contamination (µg/cm^2)

$Cont_{ResPos}$ = Residual positive control coupon contamination (µg/cm^2)

Average residual CWA contamination results are provided in Table 41 and average percent decontamination efficacies are summarized in Figure 14.

Table 41. Average Residual Contamination Summary

Decontaminant	Material	Sample Description	Avg Residual Contamination					
			VX			HD		
			Avg. (µg/cm^2)	St. Dev. (µg/cm^2)	RSD (%)	Avg. (µg/cm^2)	St. Dev. (µg/cm^2)	RSD (%)
EasyDECON DF200	ABS Plastic	Positive Controls	179	7.6	4.2%	157	3.7	2.3%
		Test Coupons	30	19	62%	111	31	28%
	Acrylic	Positive Controls	183	5.6	3.1%	217	11	5.2%
		Test Coupons	3.3	2.5	75%	177	15	8.7%
	Aluminum	Positive Controls	187	12	6.6%	231	4.8	2.1%
		Test Coupons	5.3	7.3	138%	210	7.0	3.3%
Dahlgren Decon	ABS Plastic	Positive Controls	186	11	5.9%	153	26	17%
		Test Coupons	1.5	2.3	148%	94	13	14%
	Acrylic	Positive Controls	198	7.0	3.5%	238	12	5.1%
		Test Coupons	2.4	1.9	78%	111	25	23%
	Aluminum	Positive Controls	201	15	7.7%	220	46	21%
		Test Coupons	3.2	4.5	140%	102	25	25%
TDA $eClO_2$	ABS Plastic	Positive Controls	199	17	8.6%	152	24	16%
		Test Coupons	191	14	7.4%	97	12	12%
	Acrylic	Positive Controls	196	4.4	2.3%	239	4.6	1.9%
		Test Coupons	139	9.4	6.7%	92	17	19%
	Aluminum	Positive Controls	198	5.8	2.9%	223	13	5.6%
		Test Coupons	139	21	15%	137	18	13%

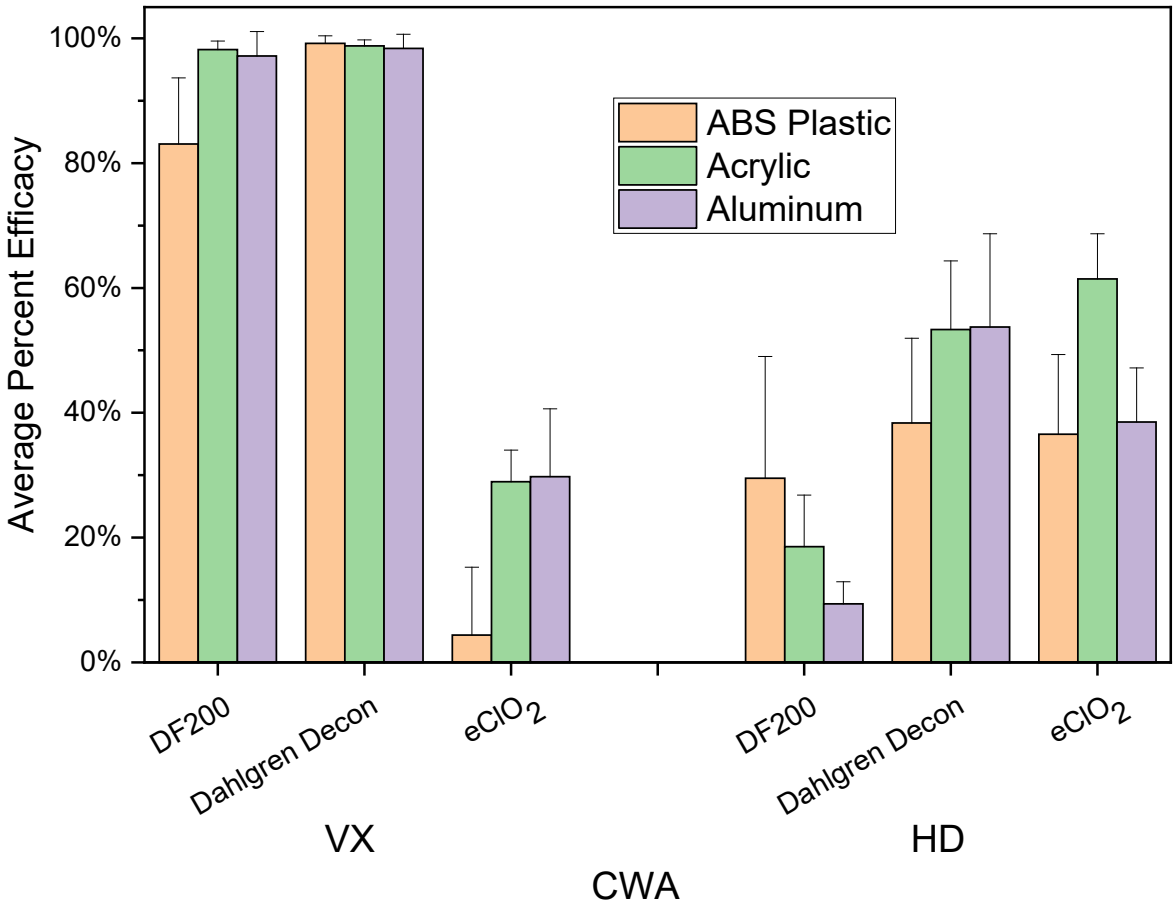


Figure 14. Average Percent Decontamination Efficacy by CWA/Decontaminant/Material

Numerically, Dahlgren Decon demonstrated the highest efficacy for decontamination of VX from all three SE-related material types, achieving 99% efficacy on ABS plastic and acrylic and 98% on aluminum. DF200 demonstrated similarly high efficacy in decontaminating VX from acrylic (98%) and aluminum (97%), though DF200 decontamination of VX from ABS plastic was slightly lower at 83%. A statistical comparison showed that the recovered amounts for Dahlgren Decon and DF200 for the ABS Plastic and aluminum were not significantly different ($p<0.05$). The TDA $eClO_2$ decontaminant demonstrated the lowest VX decontamination efficacies, measuring 30% from aluminum, 29% from acrylic, and only 4.4% from ABS plastic.

Conversely, $eClO_2$ demonstrated generally higher efficacies for decontamination of HD in comparison to the other two decontaminants. Efficacy of $eClO_2$ against HD was 37% from ABS plastic, 39% from aluminum, and 61% from acrylic (which was the highest HD decontamination efficacy measured during this work). Dahlgren Decon measured 54% HD decontamination efficacy from aluminum, 53% from acrylic, and 38% from ABS plastic. A statistical comparison showed that recovered amounts on ABS plastic and acrylic were not significantly different among all three decontaminants.

In contrast to its VX decontamination efficacy, DF200 demonstrated only 29% efficacy against HD on ABS plastic, 19% on acrylic, and 9.4% on aluminum. In summary, all three decontaminants evaluated demonstrated some degree of efficacy for decontamination of both VX and HD from all three SE-related materials. Generally, Dahlgren Decon and DF200 were much more efficacious in decontamination of VX than of HD. Conversely, $eClO_2$ demonstrated greater efficacy in decontamination of HD than in decontamination of VX. Considering the measurable amount of VX and HD remaining on the surface following any of the decontamination solution applications, additional efforts may be required to further degrade the residual agent. Such may be accomplished through an extended dwell time beyond 1 h or a reapplication of the decontaminant. Neither approach was part of the test matrix and was not investigated as part of this study.

With regard to VX and HD degradation/decontamination byproducts, neither EMPA-associated VX degradant (diethyl methanephosphonate or diethyl dimethylpyrophosphonate) was detected in any sample. No TDG was detected in any sample. However, mustard sulfone was detected in several samples, including four of five wipe sample extracts taken from HD-contaminated aluminum coupons decontaminated with Dahlgren Decon, and all wipe extracts taken from all three SE-related materials decontaminated with the $eClO_2$ decontaminant. Mustard sulfone was also detected in extracts of HD-contaminated ABS plastic and acrylic coupons decontaminated with the $eClO_2$ decontaminant. Detection of mustard sulfone was minor, ranging only from 0.12% to 14% of the peak area response of the mustard sulfone standard included in the analytical runs (see Section 2.4.3). The toxic byproduct of VX degradation, EA-2192, cannot be identified by GC/MS. Analysis for EA-2192 requires the use of LC/MS, which was outside the scope of this testing. Thus, degradation of VX into EA-2192 was not evaluated during this work.

Generally, Dahlgren Decon appeared to demonstrate the highest degree of compatibility with the three SE-related materials included in this evaluation. Residual decontaminant was easily wiped from the surface of all three material types, leaving no lasting observable effects on acrylic and only very slight discoloration of ABS plastic and aluminum. In contrast, DF200 and $eClO_2$ discolored ABS plastic to a greater degree and left residues on aluminum that were not easily removed and/or actual physical damage to/deterioration of the aluminum coupon surface ($eClO_2$).

6. REFERENCES

[1] EPA, 2016. Fate and Transport of Chemical Warfare Agents VX and HD Across a Permeable Layer into Porous Subsurfaces. EPA/600/R-16/173. U.S. Environmental Protection Agency (EPA), Office of Research and Development, National Homeland Security Research Center.

[2] Stone, H.; See, D.; Smiley, A.; Ellingson, A.; Schimmoeller, J.; Oudejans, L. Surface decontamination for blister agents Lewisite, sulfur mustard and agent yellow, a Lewisite and sulfur mustard mixture. *Journal of Hazardous Materials*. 2016, 314, pp 59-66.

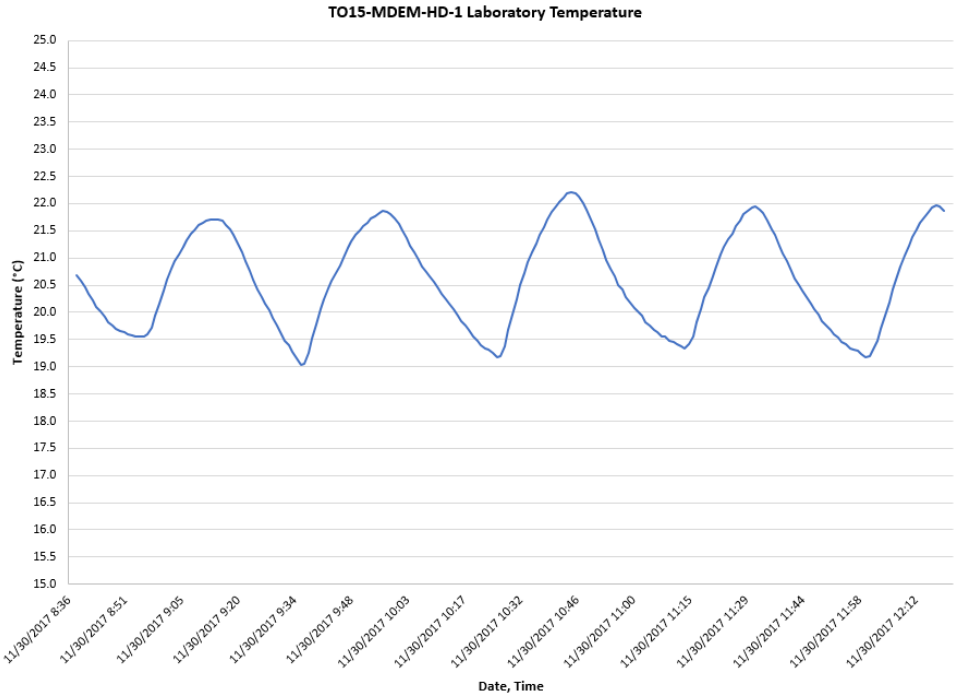
[3] Munro, N.B.; Talmage, S.S.; Griffin, G.D.; Waters, L.C.; Watson, A.P.; King, J.F.; Hauschild, V. The sources, fate, and toxicity of chemical warfare agent degradation products. *Environmental Health Perspectives*. 1999, 107(12), pp 933-974.

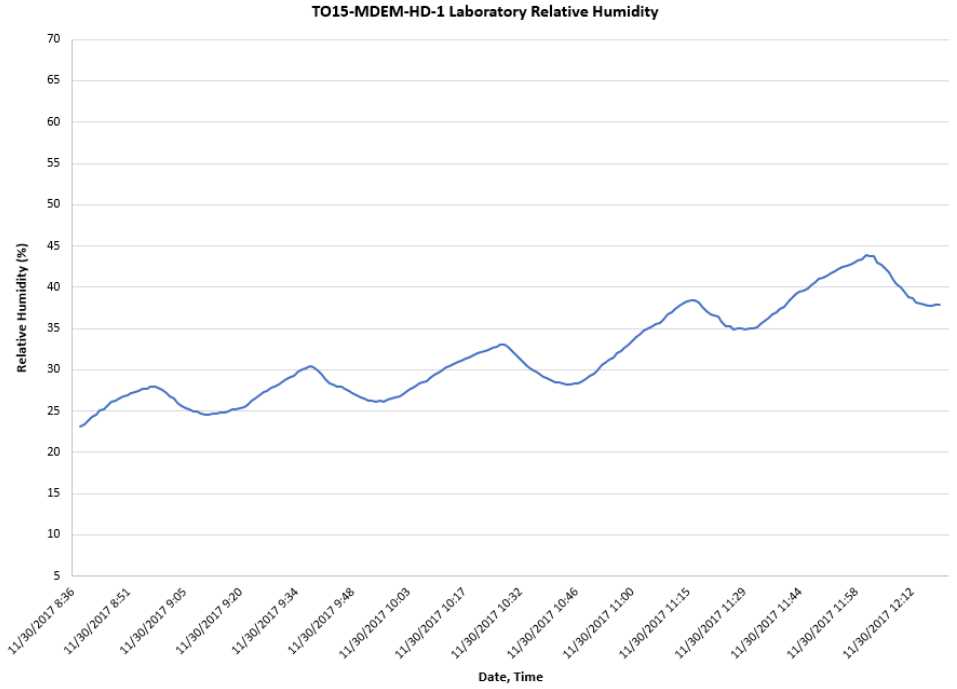
Attachment A – Environmental Data

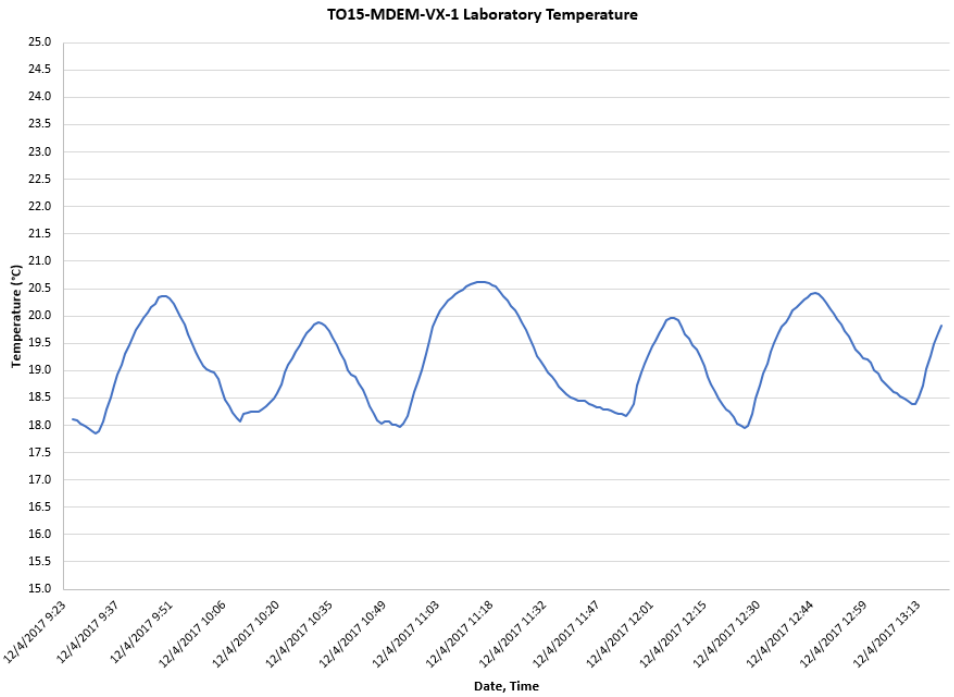
Figures TO15-MDEM-HD-1:
Temperature and RH during HD method development
Figures TO15-MDEM-VX-1:
Temperature and RH during VX method development
Figures TO15-QUENCH-1:
Temperature and RH during 1st quenching study
Figures TO15-QUENCH-2:
Temperature and RH during 2nd quenching study

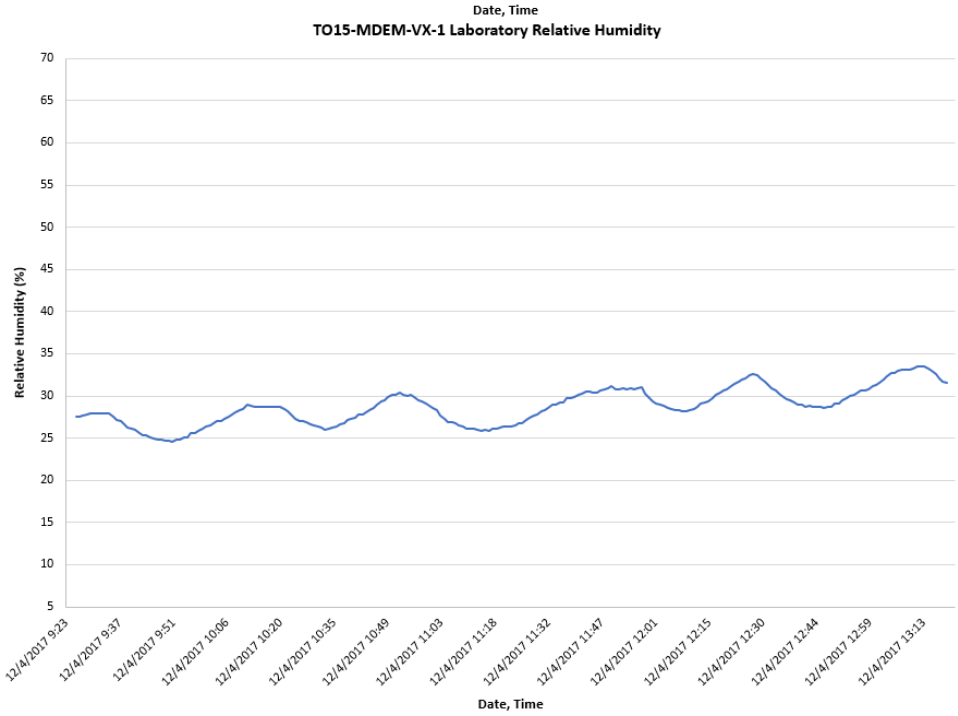
Figures ABS, ACRY DF200 VX:
Temperature and RH during DF200 decon test with VX on ABS and acrylic
Figures ALUM DF200, ABS DD VX:
Temperature and RH during DF200 decon test with VX on aluminum and DD decon test with VX on ABS
Figures ACRY, ALUM DD VX:
Temperature and RH during DD decon test with VX on acrylic and aluminum
Figures ABS, ACRY $eClO_2$ VX:
Temperature and RH during $eClO_2$ decon test with VX on ABS and acrylic
Figures ALU $eClO_2$ VX:
Temperature and RH during $eClO_2$ decon test with VX on aluminum

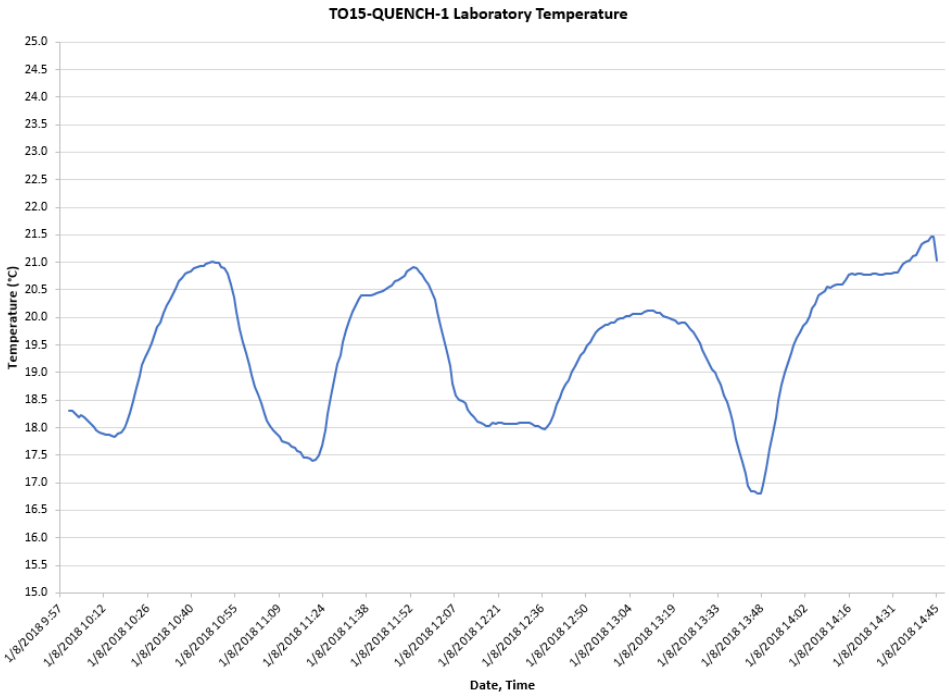
Figures ABS DF200 HD:
Temperature and RH during DF200 decon test with HD on ABS
Figures ACRY, ALUM DF200 HD:
Temperature and RH during DF200 decon test with HD acrylic and aluminum
Figures ABS DD HD:
Temperature and RH during DD decon test with HD on ABS
Figures ACRY, ALUM DD HD:
Temperature and RH during DD decon test with HD on acrylic and aluminum
Figures ABS, ACRY $eClO_2$ HD:
Temperature and RH during $eClO_2$ decon test with HD on ABS and acrylic
Figures ALUM $eClO_2$ HD:
Temperature and RH during $eClO_2$ decon test with HD on aluminum

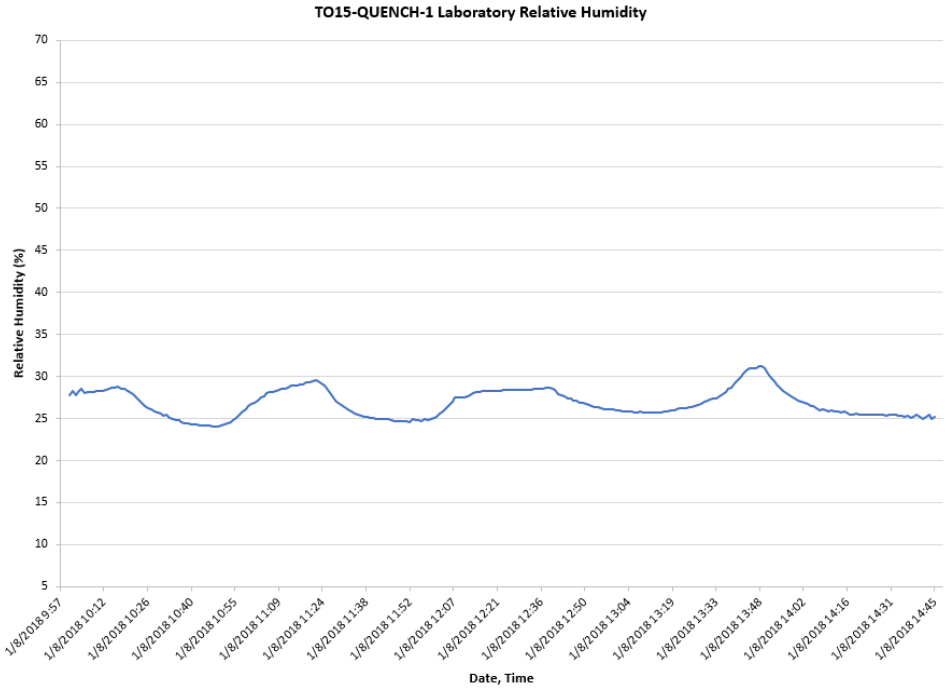

TO15-MDEM-HD-1 Laboratory Temperature
Temperature (°C)
25.0
24.5
24.0
23.5
23.0
22.5
22.0
21.5
21.0
20.5
20.0
19.5
19.0
18.5
18.0
17.5
17.0
16.5
16.0
15.5
15.0
11/30/2017 8:36
11/30/2017 8:51
11/30/2017 9:05
11/30/2017 9:20
11/30/2017 9:34
11/30/2017 9:48
11/30/2017 10:03
11/30/2017 10:17
11/30/2017 10:32
11/30/2017 10:46
11/30/2017 11:00
11/30/2017 11:15
11/30/2017 11:29
11/30/2017 11:44
11/30/2017 11:58
11/30/2017 12:12
Date, Time

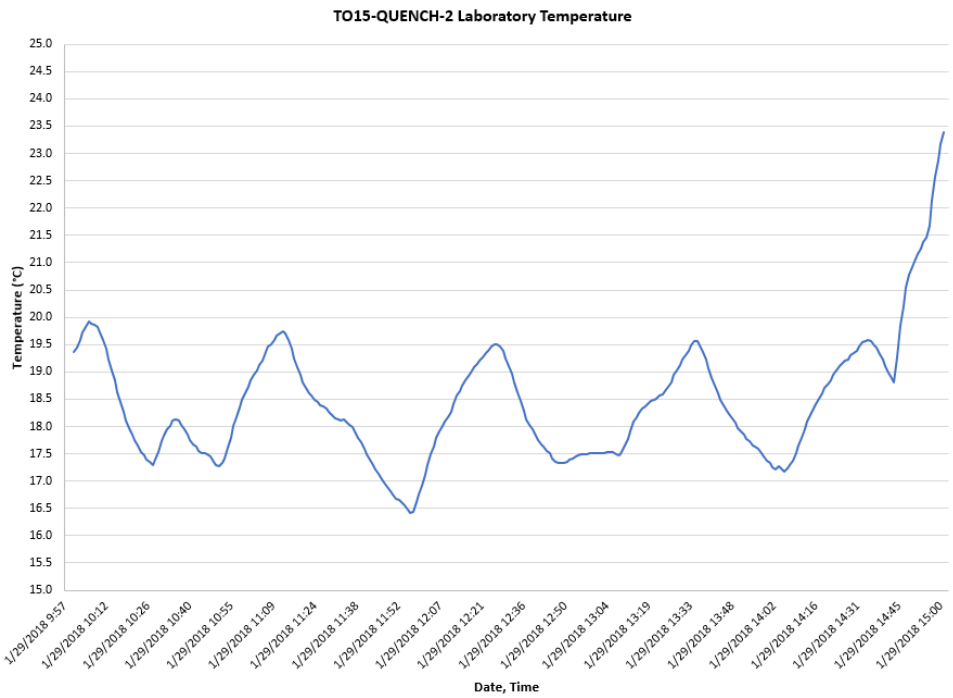

TO15-MDEM-HD-1 Laboratory Relative Humidity
Relative Humidity (%)
70
65
60
55
50
45
40
35
30
25
20
15
10
5
11/30/2017 8:36
11/30/2017 8:51
11/30/2017 9:05
11/30/2017 9:20
11/30/2017 9:34
11/30/2017 9:48
11/30/2017 10:03
11/30/2017 10:17
11/30/2017 10:32
11/30/2017 10:46
11/30/2017 11:00
11/30/2017 11:15
11/30/2017 11:29
11/30/2017 11:44
11/30/2017 11:58
11/30/2017 12:12
Date, Time

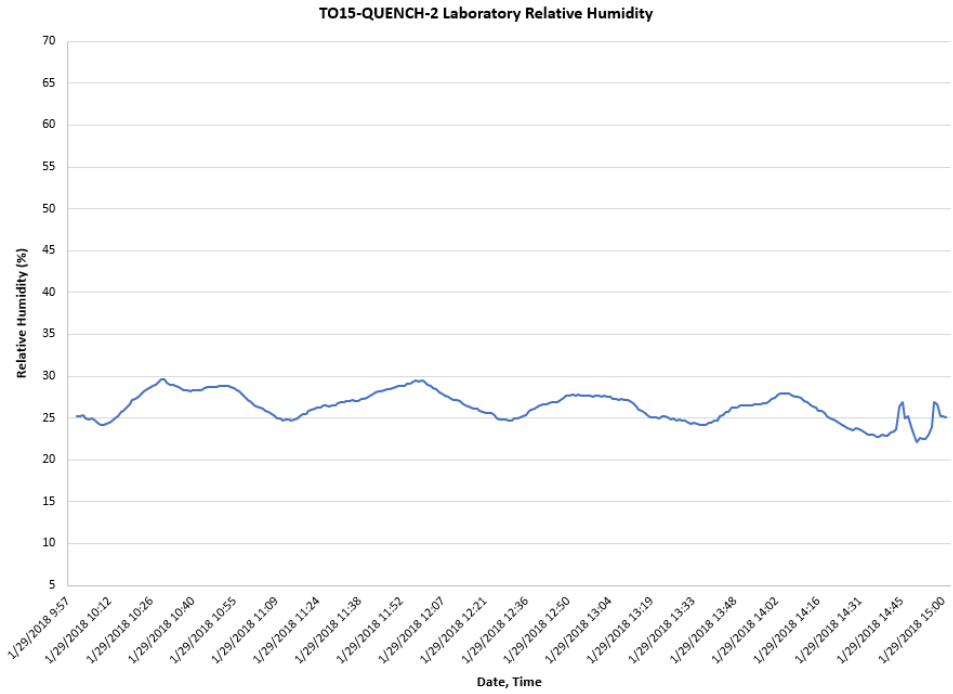

TO15-MDEM-VX-1 Laboratory Temperature
Temperature (°C)
25.0
24.5
24.0
23.5
23.0
22.5
22.0
21.5
21.0
20.5
20.0
19.5
19.0
18.5
18.0
17.5
17.0
16.5
16.0
15.5
15.0
12/4/2017 9:23
12/4/2017 9:37
12/4/2017 9:51
12/4/2017 10:06
12/4/2017 10:20
12/4/2017 10:35
12/4/2017 10:49
12/4/2017 11:03
12/4/2017 11:18
12/4/2017 11:32
12/4/2017 11:47
12/4/2017 12:01
12/4/2017 12:15
12/4/2017 12:30
12/4/2017 12:44
12/4/2017 12:59
12/4/2017 13:13
Date, Time

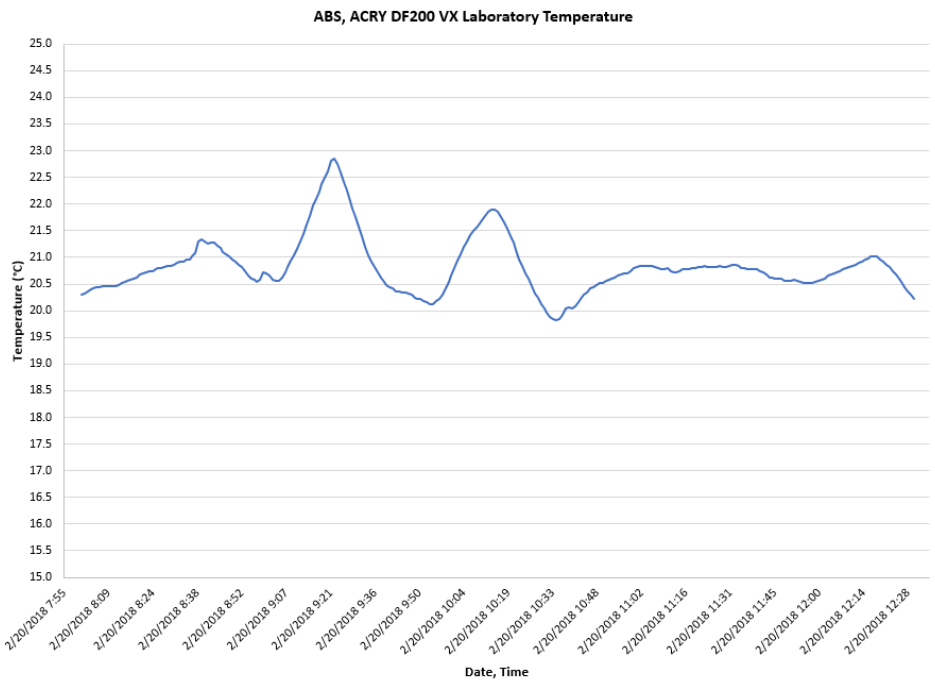

TO15-MDEM-VX-1 Laboratory Relative Humidity
Relative Humidity (%)
70
65
60
55
50
45
40
35
30
25
20
15
10
5
12/4/2017 9:23
12/4/2017 9:37
12/4/2017 9:51
12/4/2017 10:06
12/4/2017 10:20
12/4/2017 10:35
12/4/2017 10:49
12/4/2017 11:03
12/4/2017 11:18
12/4/2017 11:32
12/4/2017 11:47
12/4/2017 12:01
12/4/2017 12:15
12/4/2017 12:30
12/4/2017 12:44
12/4/2017 12:59
12/4/2017 13:13
Date, Time

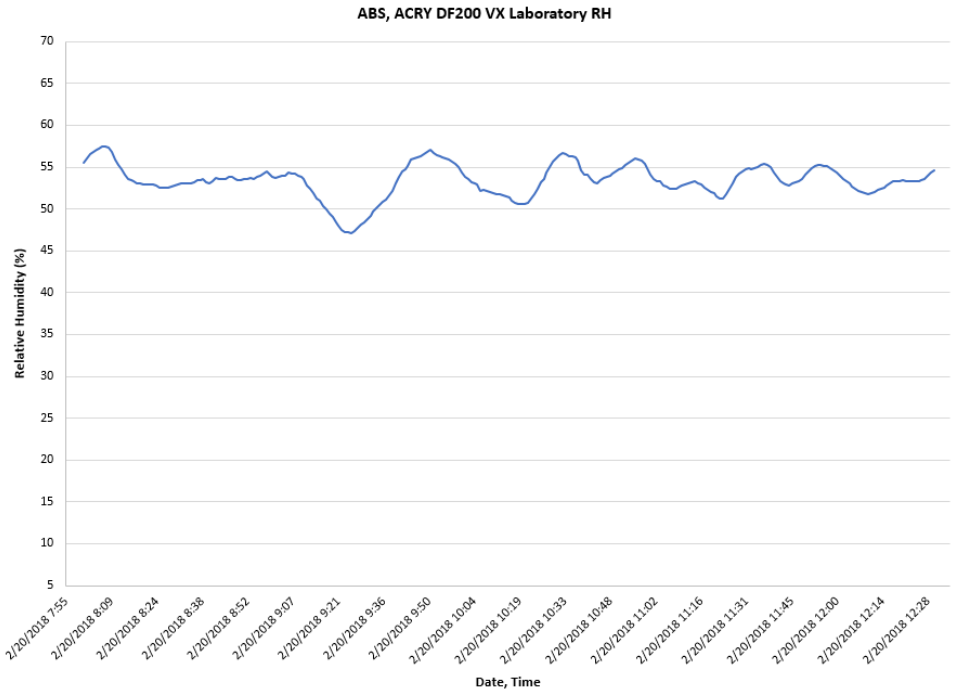

TO15-QUENCH-1 Laboratory Temperature
Temperature (°C)
25.0
24.5
24.0
23.5
23.0
22.5
22.0
21.5
21.0
20.5
20.0
19.5
19.0
18.5
18.0
17.5
17.0
16.5
16.0
15.5
15.0
1/8/2018 9:57
1/8/2018 10:12
1/8/2018 10:26
1/8/2018 10:40
1/8/2018 10:55
1/8/2018 11:09
1/8/2018 11:24
1/8/2018 11:38
1/8/2018 11:52
1/8/2018 12:07
1/8/2018 12:21
1/8/2018 12:36
1/8/2018 12:50
1/8/2018 13:04
1/8/2018 13:19
1/8/2018 13:33
1/8/2018 13:48
1/8/2018 14:02
1/8/2018 14:16
1/8/2018 14:31
1/8/2018 14:45
Date, Time

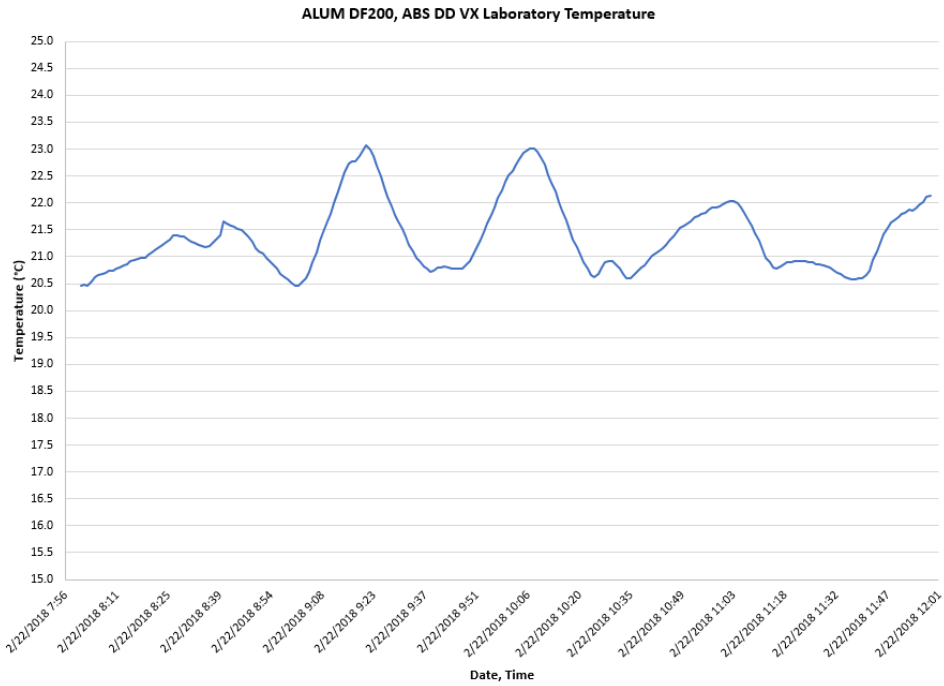

TO15-QUENCH-1 Laboratory Relative Humidity
Relative Humidity (%)
70
65
60
55
50
45
40
35
30
25
20
15
10
5
1/8/2018 9:57
1/8/2018 10:12
1/8/2018 10:26
1/8/2018 10:40
1/8/2018 10:55
1/8/2018 11:09
1/8/2018 11:24
1/8/2018 11:38
1/8/2018 11:52
1/8/2018 12:07
1/8/2018 12:21
1/8/2018 12:36
1/8/2018 12:50
1/8/2018 13:04
1/8/2018 13:19
1/8/2018 13:33
1/8/2018 13:48
1/8/2018 14:02
1/8/2018 14:16
1/8/2018 14:31
1/8/2018 14:45
Date, Time

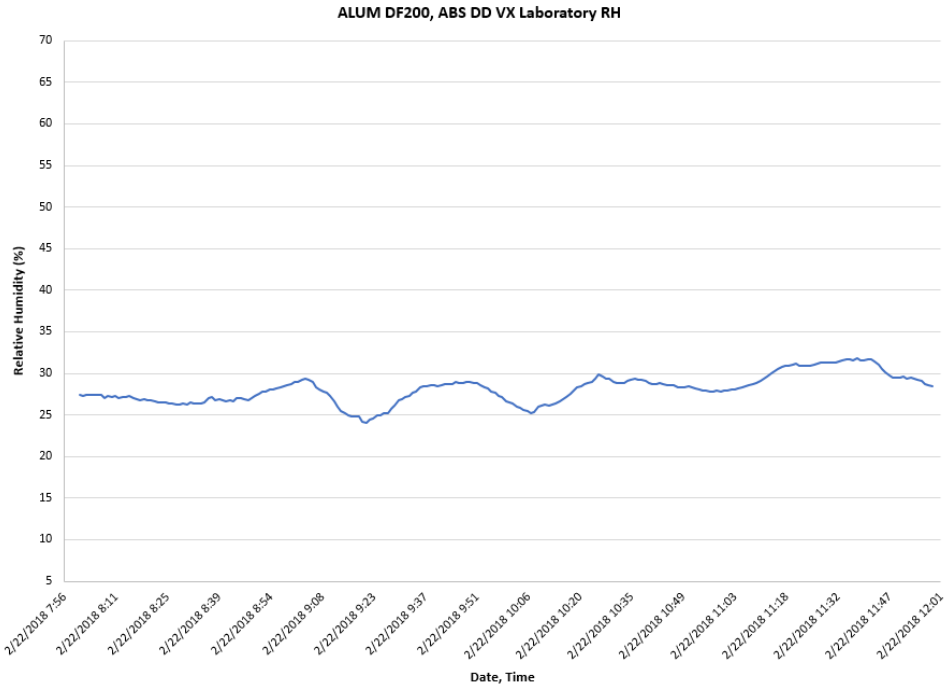

TO15-QUENCH-2 Laboratory Temperature
Temperature (°C)
25.0
24.5
24.0
23.5
23.0
22.5
22.0
21.5
21.0
20.5
20.0
19.5
19.0
18.5
18.0
17.5
17.0
16.5
16.0
15.5
15.0
1/29/2018 9:57
1/29/2018 10:12
1/29/2018 10:26
1/29/2018 10:40
1/29/2018 10:55
1/29/2018 11:09
1/29/2018 11:24
1/29/2018 11:38
1/29/2018 11:52
1/29/2018 12:07
1/29/2018 12:21
1/29/2018 12:36
1/29/2018 12:50
1/29/2018 13:04
1/29/2018 13:19
1/29/2018 13:33
1/29/2018 13:48
1/29/2018 14:02
1/29/2018 14:16
1/29/2018 14:31
1/29/2018 14:45
1/29/2018 15:00
Date, Time

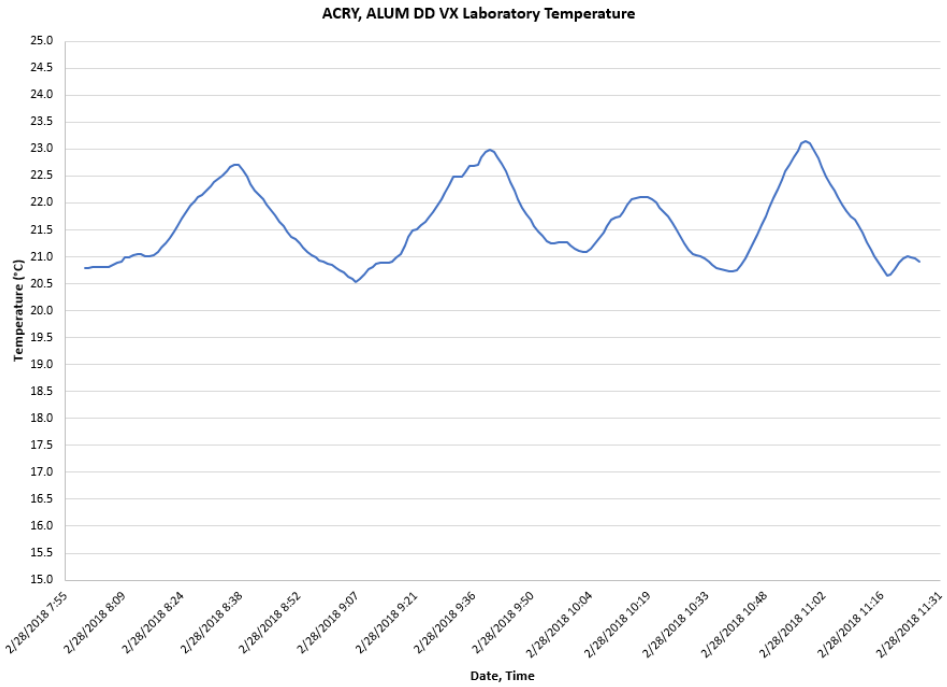

TO15-QUENCH-2 Laboratory Relative Humidity
Relative Humidity (%)
70
65
60
55
50
45
40
35
30
25
20
15
10
5
1/29/2018 9:57
1/29/2018 10:12
1/29/2018 10:26
1/29/2018 10:40
1/29/2018 10:55
1/29/2018 11:09
1/29/2018 11:24
1/29/2018 11:38
1/29/2018 11:52
1/29/2018 12:07
1/29/2018 12:21
1/29/2018 12:36
1/29/2018 12:50
1/29/2018 13:04
1/29/2018 13:19
1/29/2018 13:33
1/29/2018 13:48
1/29/2018 14:02
1/29/2018 14:16
1/29/2018 14:31
1/29/2018 14:45
1/29/2018 15:00
Date, Time

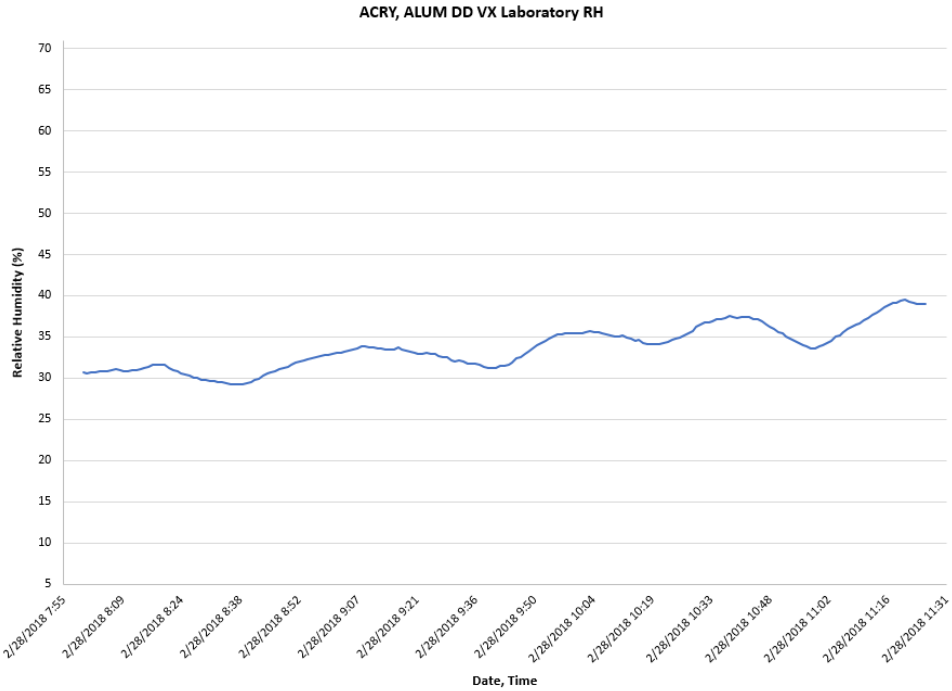

ABS, ACRY DF200 VX Laboratory Temperature
Temperature (°C)
25.0
24.5
24.0
23.5
23.0
22.5
22.0
21.5
21.0
20.5
20.0
19.5
19.0
18.5
18.0
17.5
17.0
16.5
16.0
15.5
15.0
2/20/2018 7:55
2/20/2018 8:09
2/20/2018 8:24
2/20/2018 8:38
2/20/2018 8:52
2/20/2018 9:07
2/20/2018 9:21
2/20/2018 9:36
2/20/2018 9:50
2/20/2018 10:04
2/20/2018 10:19
2/20/2018 10:33
2/20/2018 10:48
2/20/2018 11:02
2/20/2018 11:16
2/20/2018 11:31
2/20/2018 11:45
2/20/2018 12:00
2/20/2018 12:14
2/20/2018 12:28
Date, Time

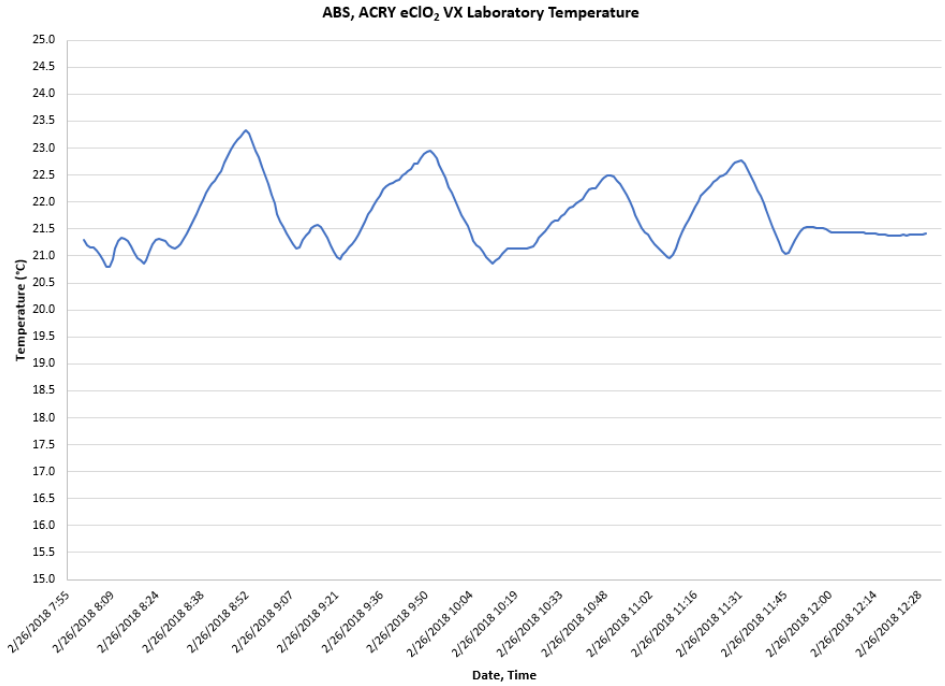

ABS, ACRY DF200 VX Laboratory RH
Relative Humidity (%)
70
65
60
55
50
45
40
35
30
25
20
15
10
5
2/20/2018 7:55
2/20/2018 8:09
2/20/2018 8:24
2/20/2018 8:38
2/20/2018 8:52
2/20/2018 9:07
2/20/2018 9:21
2/20/2018 9:36
2/20/2018 9:50
2/20/2018 10:04
2/20/2018 10:19
2/20/2018 10:33
2/20/2018 10:48
2/20/2018 11:02
2/20/2018 11:16
2/20/2018 11:31
2/20/2018 11:45
2/20/2018 12:00
2/20/2018 12:14
2/20/2018 12:28
Date, Time

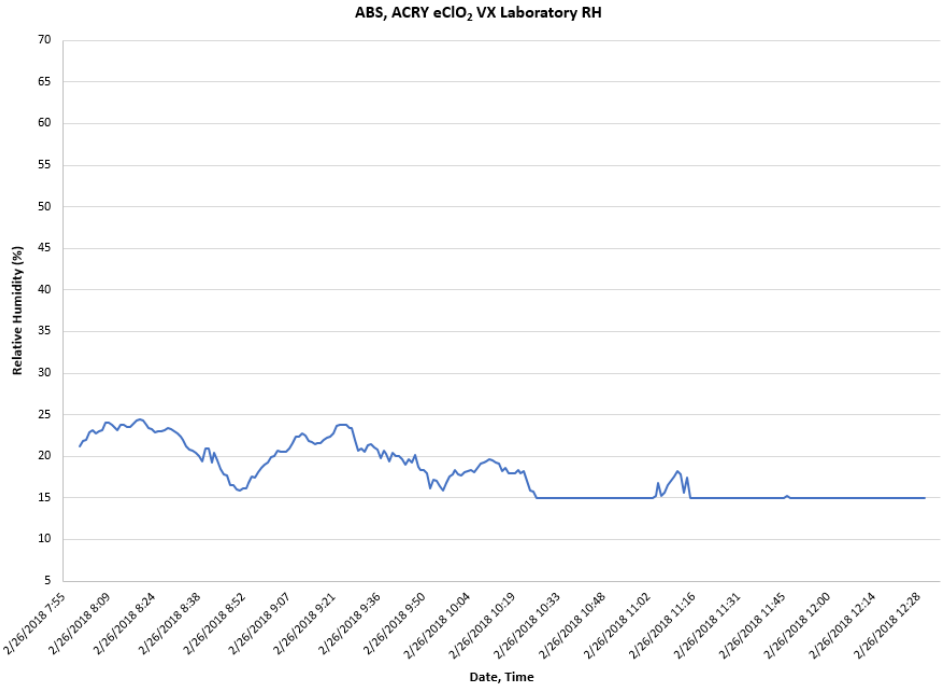

ALUM DF200, ABS DD VX Laboratory Temperature
Temperature (°C)
25.0
24.5
24.0
23.5
23.0
22.5
22.0
21.5
21.0
20.5
20.0
19.5
19.0
18.5
18.0
17.5
17.0
16.5
16.0
15.5
15.0
2/22/2018 7:56
2/22/2018 8:11
2/22/2018 8:25
2/22/2018 8:39
2/22/2018 8:54
2/22/2018 9:08
2/22/2018 9:23
2/22/2018 9:37
2/22/2018 9:51
2/22/2018 10:06
2/22/2018 10:20
2/22/2018 10:35
2/22/2018 10:49
2/22/2018 11:03
2/22/2018 11:18
2/22/2018 11:32
2/22/2018 11:47
2/22/2018 12:01
Date, Time

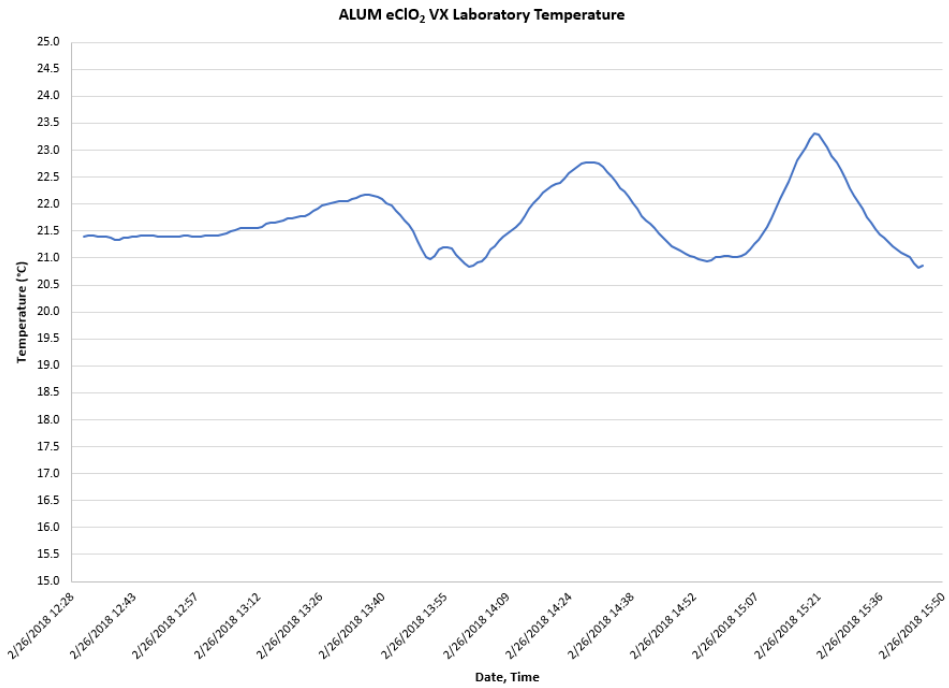

ALUM DF200, ABS DD VX Laboratory RH
Relative Humidity (%)
70
65
60
55
50
45
40
35
30
25
20
15
10
5
2/22/2018 7:56
2/22/2018 8:11
2/22/2018 8:25
2/22/2018 8:39
2/22/2018 8:54
2/22/2018 9:08
2/22/2018 9:23
2/22/2018 9:37
2/22/2018 9:51
2/22/2018 10:06
2/22/2018 10:20
2/22/2018 10:35
2/22/2018 10:49
2/22/2018 11:03
2/22/2018 11:18
2/22/2018 11:32
2/22/2018 11:47
2/22/2018 12:01
Date, Time

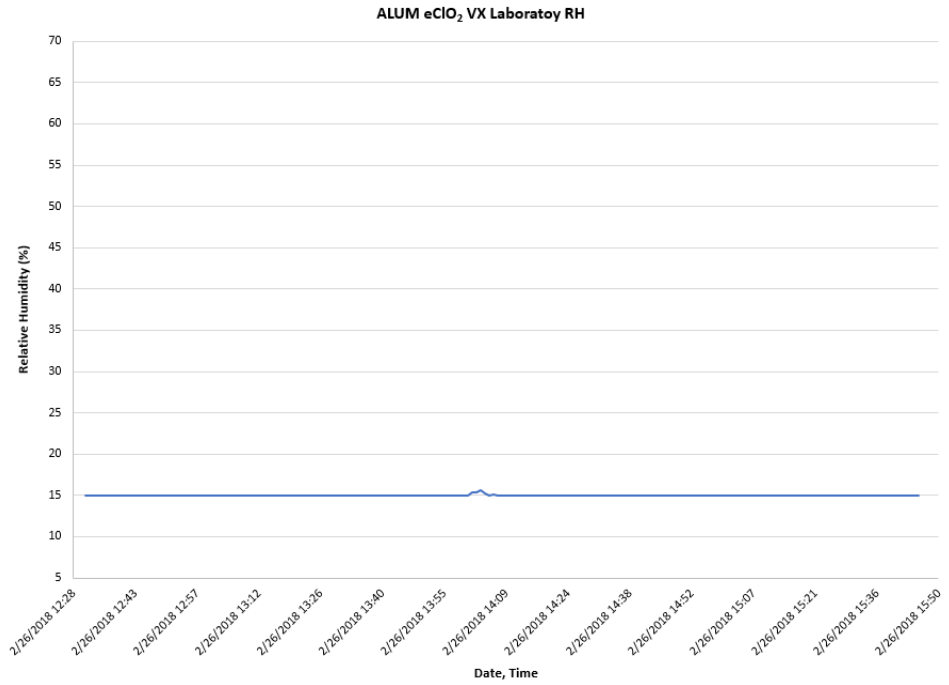

ACRY, ALUM DD VX Laboratory Temperature
Temperature (°C)
25.0
24.5
24.0
23.5
23.0
22.5
22.0
21.5
21.0
20.5
20.0
19.5
19.0
18.5
18.0
17.5
17.0
16.5
16.0
15.5
15.0
2/28/2018 7:55
2/28/2018 8:09
2/28/2018 8:24
2/28/2018 8:38
2/28/2018 8:52
2/28/2018 9:07
2/28/2018 9:21
2/28/2018 9:36
2/28/2018 9:50
2/28/2018 10:04
2/28/2018 10:19
2/28/2018 10:33
2/28/2018 10:48
2/28/2018 11:02
2/28/2018 11:16
2/28/2018 11:31
Date, Time

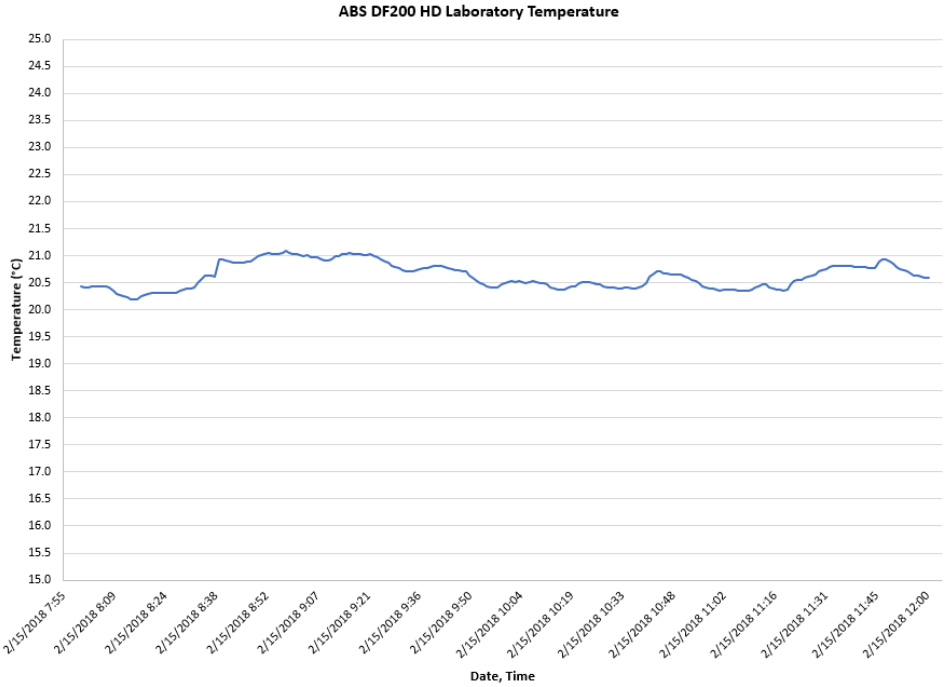

ACRY, ALUM DD VX Laboratory RH
Relative Humidity (%)
70
65
60
55
50
45
40
35
30
25
20
15
10
5
2/28/2018 7:55
2/28/2018 8:09
2/28/2018 8:24
2/28/2018 8:38
2/28/2018 8:52
2/28/2018 9:07
2/28/2018 9:21
2/28/2018 9:36
2/28/2018 9:50
2/28/2018 10:04
2/28/2018 10:19
2/28/2018 10:33
2/28/2018 10:48
2/28/2018 11:02
2/28/2018 11:16
2/28/2018 11:31
Date, Time

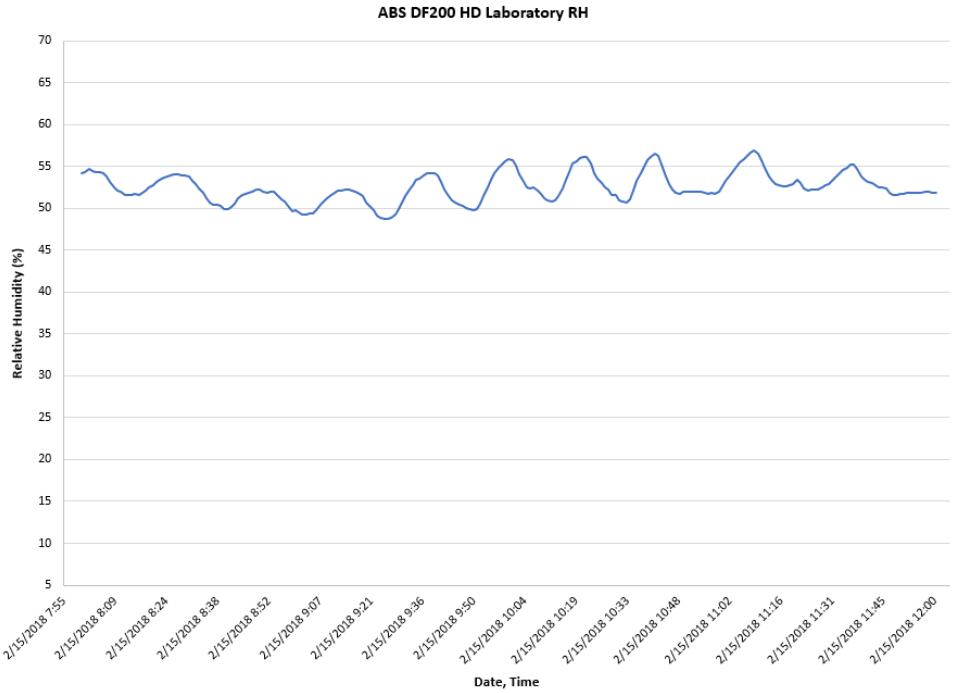

ABS, ACRY eClO2 VX Laboratory Temperature
Temperature (°C)
25.0
24.5
24.0
23.5
23.0
22.5
22.0
21.5
21.0
20.5
20.0
19.5
19.0
18.5
18.0
17.5
17.0
16.5
16.0
15.5
15.0
2/26/2018 7:55
2/26/2018 8:09
2/26/2018 8:24
2/26/2018 8:38
2/26/2018 8:52
2/26/2018 9:07
2/26/2018 9:21
2/26/2018 9:36
2/26/2018 9:50
2/26/2018 10:04
2/26/2018 10:19
2/26/2018 10:33
2/26/2018 10:48
2/26/2018 11:02
2/26/2018 11:16
2/26/2018 11:31
2/26/2018 11:45
2/26/2018 12:00
2/26/2018 12:14
2/26/2018 12:28
Date, Time

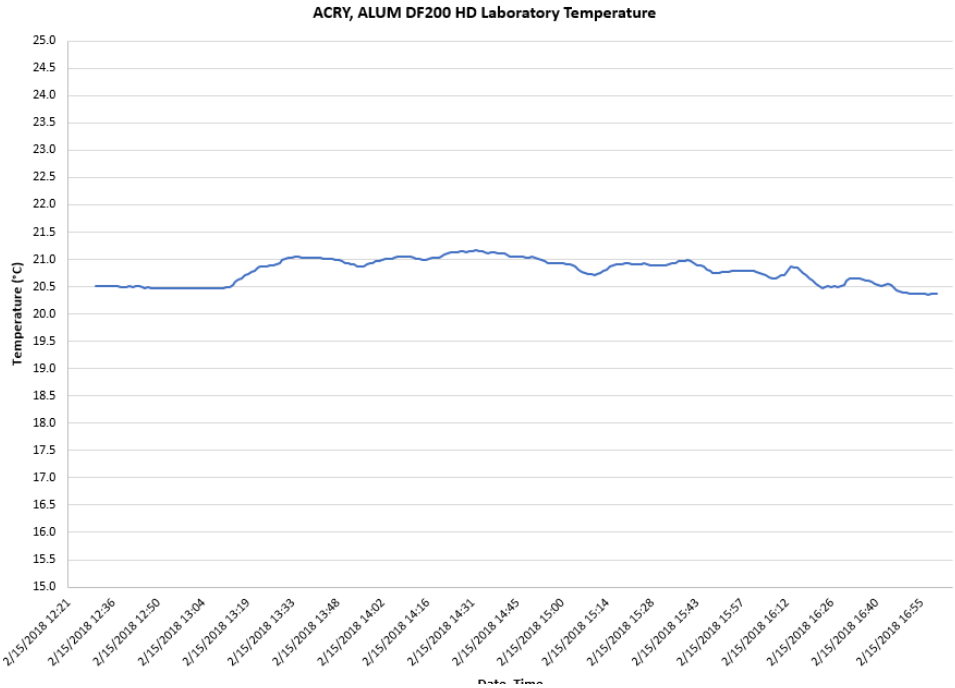

ABS, ACRY eClO2 VX Laboratory RH
Relative Humidity (%)
70
65
60
55
50
45
40
35
30
25
20
15
10
5
2/26/2018 7:55
2/26/2018 8:09
2/26/2018 8:24
2/26/2018 8:38
2/26/2018 8:52
2/26/2018 9:07
2/26/2018 9:21
2/26/2018 9:36
2/26/2018 9:50
2/26/2018 10:04
2/26/2018 10:19
2/26/2018 10:33
2/26/2018 10:48
2/26/2018 11:02
2/26/2018 11:16
2/26/2018 11:31
2/26/2018 11:45
2/26/2018 12:00
2/26/2018 12:14
2/26/2018 12:28
Date, Time

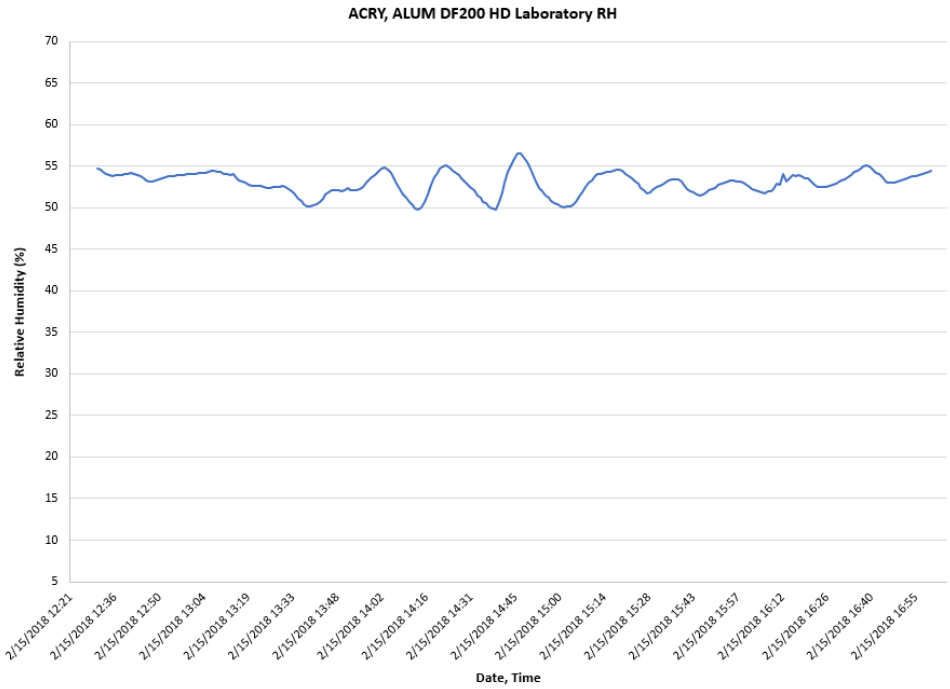

ALUM eClO2 VX Laboratory Temperature
Temperature (°C)
25.0
24.5
24.0
23.5
23.0
22.5
22.0
21.5
21.0
20.5
20.0
19.5
19.0
18.5
18.0
17.5
17.0
16.5
16.0
15.5
15.0
2/26/2018 12:28
2/26/2018 12:43
2/26/2018 12:57
2/26/2018 13:12
2/26/2018 13:26
2/26/2018 13:40
2/26/2018 13:55
2/26/2018 14:09
2/26/2018 14:24
2/26/2018 14:38
2/26/2018 14:52
2/26/2018 15:07
2/26/2018 15:21
2/26/2018 15:36
2/26/2018 15:50
Date, Time

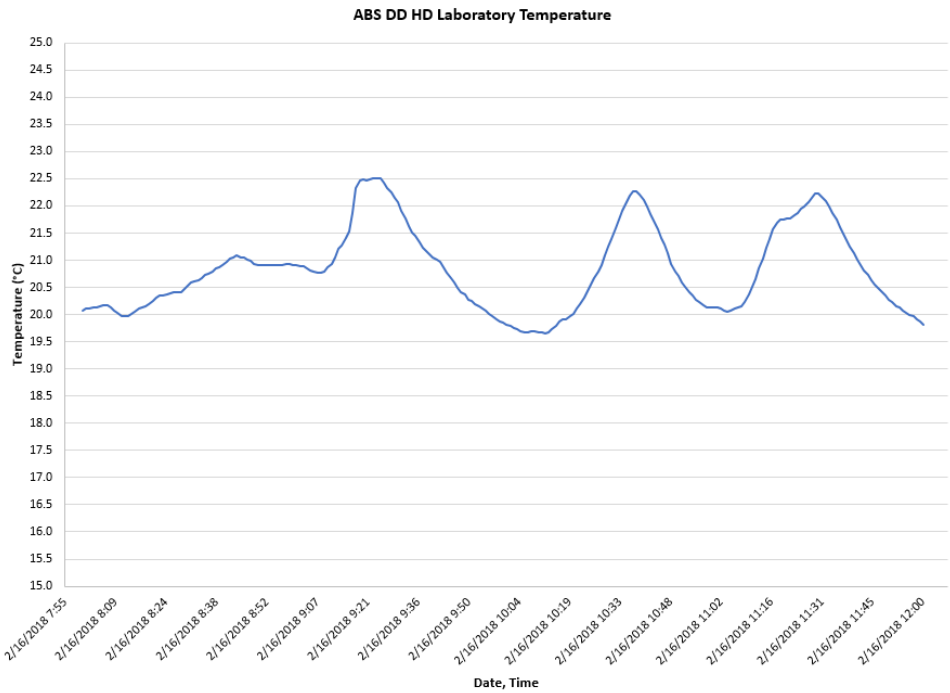

ALUM eClO2 VX Laboratoy RH
Relative Humidity (%)
70
65
60
55
50
45
40
35
30
25
20
15
10
5
2/26/2018 12:28
2/26/2018 12:43
2/26/2018 12:57
2/26/2018 13:12
2/26/2018 13:26
2/26/2018 13:40
2/26/2018 13:55
2/26/2018 14:09
2/26/2018 14:24
2/26/2018 14:38
2/26/2018 14:52
2/26/2018 15:07
2/26/2018 15:21
2/26/2018 15:36
2/26/2018 15:50
Date, Time

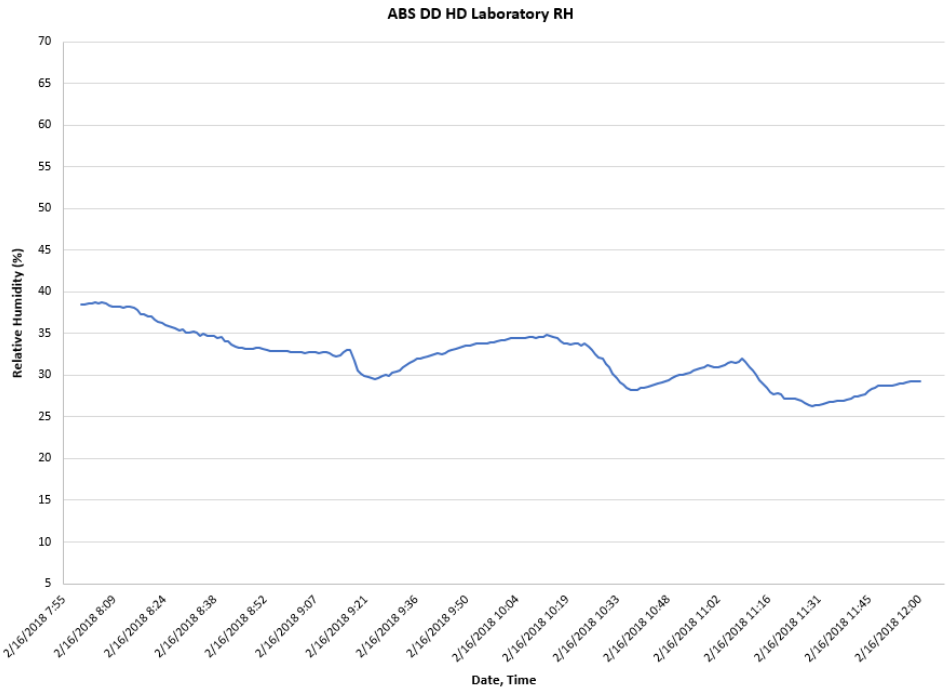

ABS DF200 HD Laboratory Temperature
Temperature (°C)
25.0
24.5
24.0
23.5
23.0
22.5
22.0
21.5
21.0
20.5
20.0
19.5
19.0
18.5
18.0
17.5
17.0
16.5
16.0
15.5
15.0
2/15/2018 7:55
2/15/2018 8:09
2/15/2018 8:24
2/15/2018 8:38
2/15/2018 8:52
2/15/2018 9:07
2/15/2018 9:21
2/15/2018 9:36
2/15/2018 9:50
2/15/2018 10:04
2/15/2018 10:19
2/15/2018 10:33
2/15/2018 10:48
2/15/2018 11:02
2/15/2018 11:16
2/15/2018 11:31
2/15/2018 11:45
2/15/2018 12:00
Date, Time

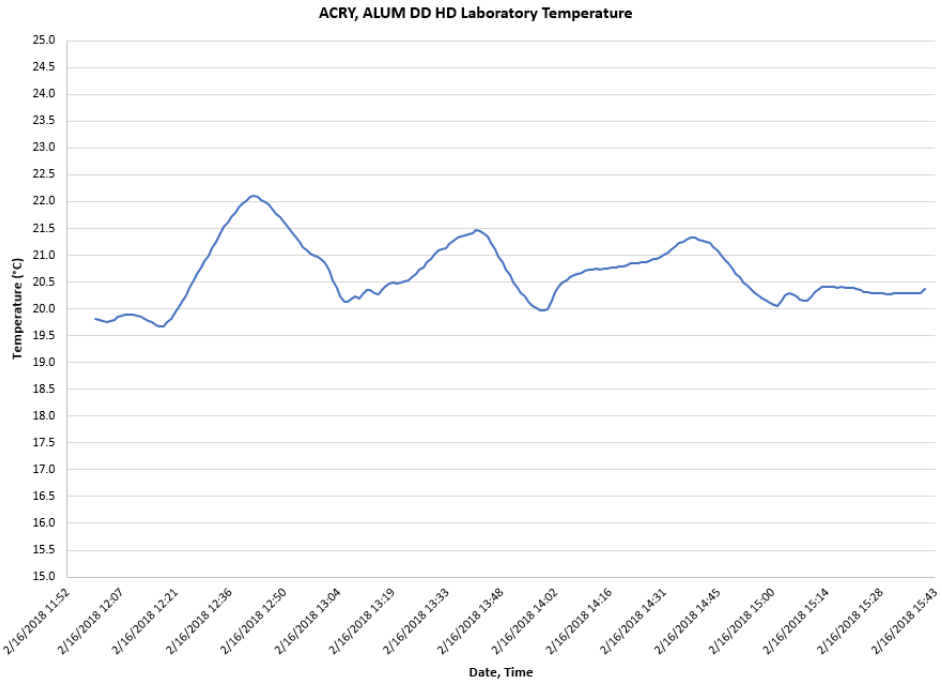

ABS DF200 HD Laboratory RH
Relative Humidity (%)
70
65
60
55
50
45
40
35
30
25
20
15
10
5
2/15/2018 7:55
2/15/2018 8:09
2/15/2018 8:24
2/15/2018 8:38
2/15/2018 8:52
2/15/2018 9:07
2/15/2018 9:21
2/15/2018 9:36
2/15/2018 9:50
2/15/2018 10:04
2/15/2018 10:19
2/15/2018 10:33
2/15/2018 10:48
2/15/2018 11:02
2/15/2018 11:16
2/15/2018 11:31
2/15/2018 11:45
2/15/2018 12:00
Date, Time

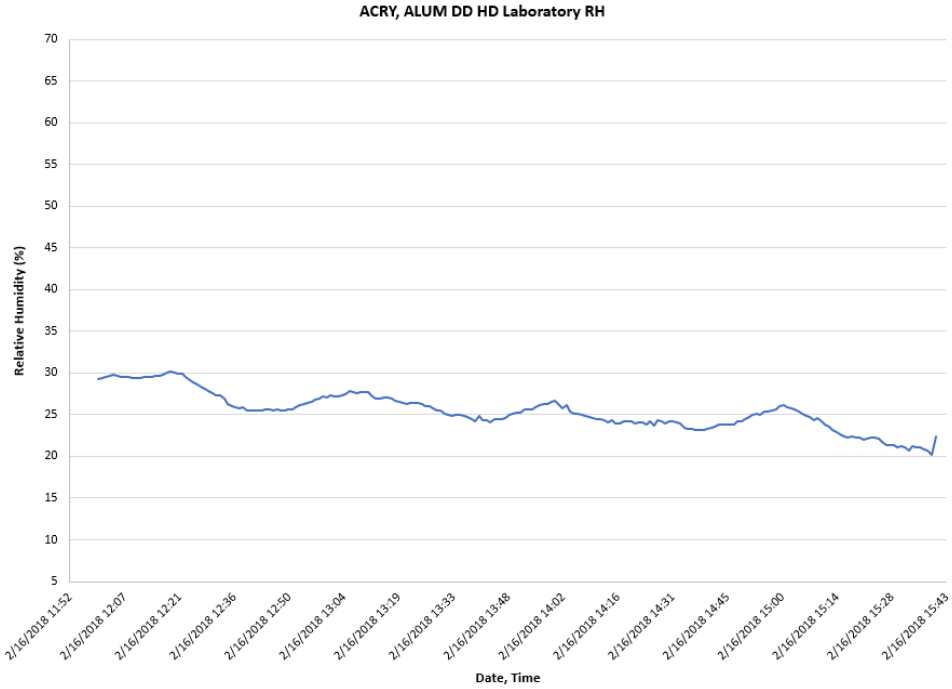

ACRY, ALUM DF200 HD Laboratory Temperature
Temperature (°C)
25.0
24.5
24.0
23.5
23.0
22.5
22.0
21.5
21.0
20.5
20.0
19.5
19.0
18.5
18.0
17.5
17.0
16.5
16.0
15.5
15.0
2/15/2018 12:21
2/15/2018 12:36
2/15/2018 12:50
2/15/2018 13:04
2/15/2018 13:19
2/15/2018 13:33
2/15/2018 13:48
2/15/2018 14:02
2/15/2018 14:16
2/15/2018 14:31
2/15/2018 14:45
2/15/2018 15:00
2/15/2018 15:14
2/15/2018 15:28
2/15/2018 15:43
2/15/2018 15:57
2/15/2018 16:12
2/15/2018 16:26
2/15/2018 16:40
2/15/2018 16:55

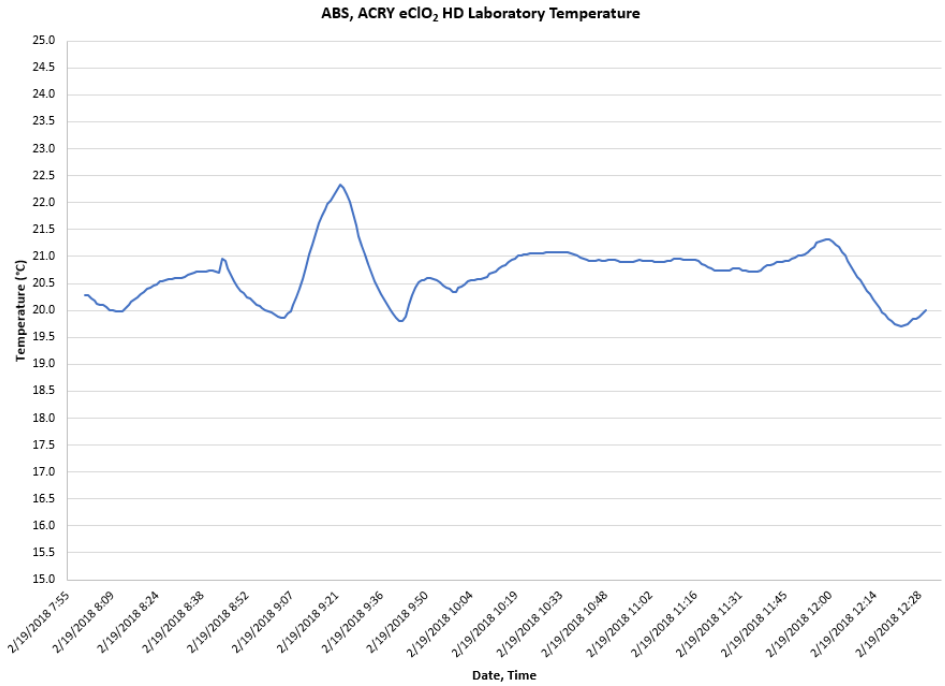

ACRY, ALUM DF200 HD Laboratory RH
Relative Humidity (%)
70
65
60
55
50
45
40
35
30
25
20
15
10
5
2/15/2018 12:21
2/15/2018 12:36
2/15/2018 12:50
2/15/2018 13:04
2/15/2018 13:19
2/15/2018 13:33
2/15/2018 13:48
2/15/2018 14:02
2/15/2018 14:16
2/15/2018 14:31
2/15/2018 14:45
2/15/2018 15:00
2/15/2018 15:14
2/15/2018 15:28
2/15/2018 15:43
2/15/2018 15:57
2/15/2018 16:12
2/15/2018 16:26
2/15/2018 16:40
2/15/2018 16:55
Date, Time

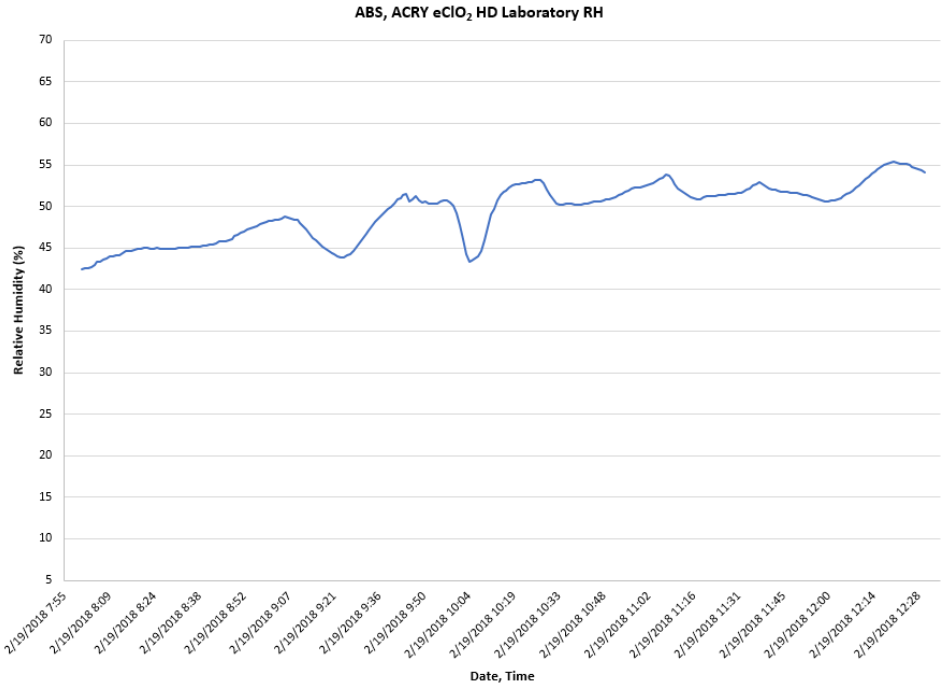

ABS DD HD Laboratory Temperature
Temperature (°C)
25.0
24.5
24.0
23.5
23.0
22.5
22.0
21.5
21.0
20.5
20.0
19.5
19.0
18.5
18.0
17.5
17.0
16.5
16.0
15.5
15.0
2/16/2018 7:55
2/16/2018 8:09
2/16/2018 8:24
2/16/2018 8:38
2/16/2018 8:52
2/16/2018 9:07
2/16/2018 9:21
2/16/2018 9:36
2/16/2018 9:50
2/16/2018 10:04
2/16/2018 10:19
2/16/2018 10:33
2/16/2018 10:48
2/16/2018 11:02
2/16/2018 11:16
2/16/2018 11:31
2/16/2018 11:45
2/16/2018 12:00
Date, Time

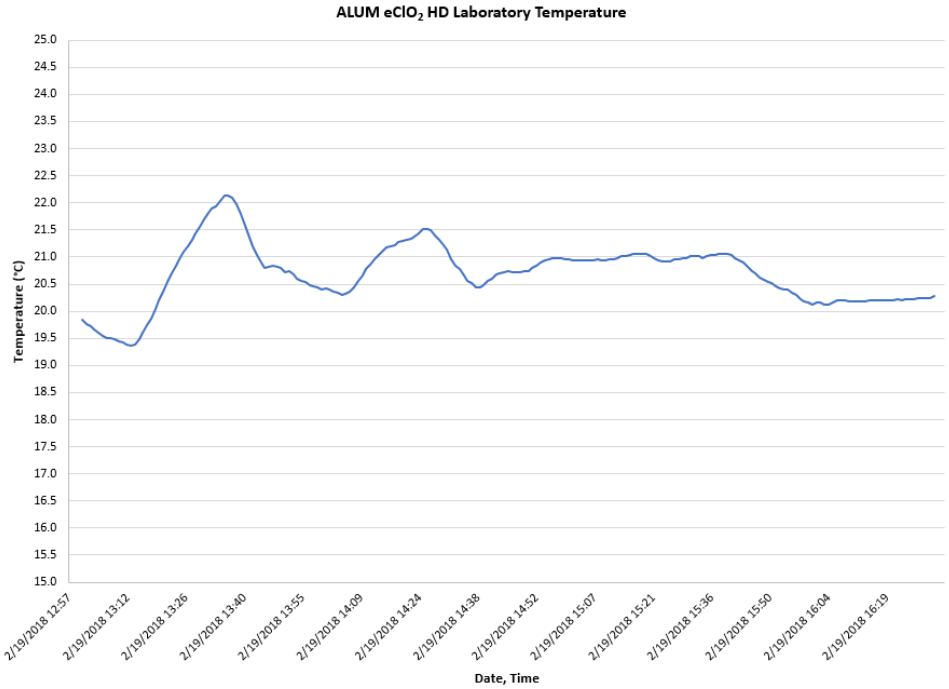

ABS DD HD Laboratory RH
Relative Humidity (%)
70
65
60
55
50
45
40
35
30
25
20
15
10
5
2/16/2018 7:55
2/16/2018 8:09
2/16/2018 8:24
2/16/2018 8:38
2/16/2018 8:52
2/16/2018 9:07
2/16/2018 9:21
2/16/2018 9:36
2/16/2018 9:50
2/16/2018 10:04
2/16/2018 10:19
2/16/2018 10:33
2/16/2018 10:48
2/16/2018 11:02
2/16/2018 11:16
2/16/2018 11:31
2/16/2018 11:45
2/16/2018 12:00
Date, Time

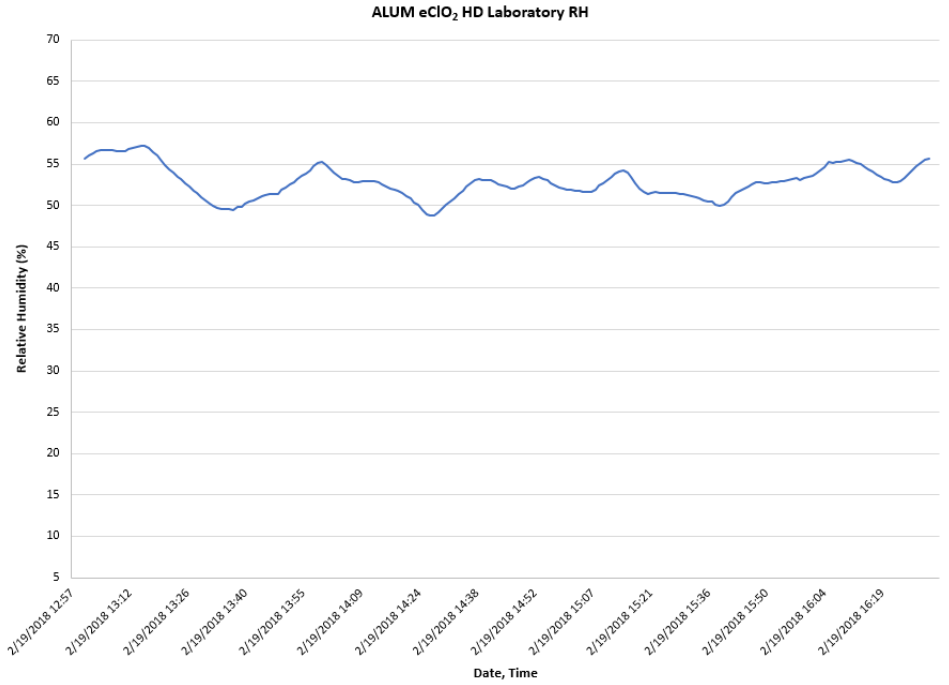

ACRY, ALUM DD HD Laboratory Temperature
Temperature (°C)
25.0
24.5
24.0
23.5
23.0
22.5
22.0
21.5
21.0
20.5
20.0
19.5
19.0
18.5
18.0
17.5
17.0
16.5
16.0
15.5
15.0
2/16/2018 11:52
2/16/2018 12:07
2/16/2018 12:21
2/16/2018 12:36
2/16/2018 12:50
2/16/2018 13:04
2/16/2018 13:19
2/16/2018 13:33
2/16/2018 13:48
2/16/2018 14:02
2/16/2018 14:16
2/16/2018 14:31
2/16/2018 14:45
2/16/2018 15:00
2/16/2018 15:14
2/16/2018 15:28
2/16/2018 15:43
Date, Time


ACRY, ALUM DD HD Laboratory RH
Relative Humidity (%)
70
65
60
55
50
45
40
35
30
25
20
15
10
5
2/16/2018 11:52
2/16/2018 12:07
2/16/2018 12:21
2/16/2018 12:36
2/16/2018 12:50
2/16/2018 13:04
2/16/2018 13:19
2/16/2018 13:33
2/16/2018 13:48
2/16/2018 14:02
2/16/2018 14:16
2/16/2018 14:31
2/16/2018 14:45
2/16/2018 15:00
2/16/2018 15:14
2/16/2018 15:28
2/16/2018 15:43
Date, Time


ABS, ACRY eClO2 HD Laboratory Temperature
Temperature (°C)
25.0
24.5
24.0
23.5
23.0
22.5
22.0
21.5
21.0
20.5
20.0
19.5
19.0
18.5
18.0
17.5
17.0
16.5
16.0
15.5
15.0
2/19/2018 7:55
2/19/2018 8:09
2/19/2018 8:24
2/19/2018 8:38
2/19/2018 8:52
2/19/2018 9:07
2/19/2018 9:21
2/19/2018 9:36
2/19/2018 9:50
2/19/2018 10:04
2/19/2018 10:19
2/19/2018 10:33
2/19/2018 10:48
2/19/2018 11:02
2/19/2018 11:16
2/19/2018 11:31
2/19/2018 11:45
2/19/2018 12:00
2/19/2018 12:14
2/19/2018 12:28
Date, Time


ABS, ACRY eClO2 HD Laboratory RH
Relative Humidity (%)
70
65
60
55
50
45
40
35
30
25
20
15
10
5
2/19/2018 7:55
2/19/2018 8:09
2/19/2018 8:24
2/19/2018 8:38
2/19/2018 8:52
2/19/2018 9:07
2/19/2018 9:21
2/19/2018 9:36
2/19/2018 9:50
2/19/2018 10:04
2/19/2018 10:19
2/19/2018 10:33
2/19/2018 10:48
2/19/2018 11:02
2/19/2018 11:16
2/19/2018 11:31
2/19/2018 11:45
2/19/2018 12:00
2/19/2018 12:14
2/19/2018 12:28
Date, Time


ALUM eClO2 HD Laboratory Temperature
Temperature (°C)
25.0
24.5
24.0
23.5
23.0
22.5
22.0
21.5
21.0
20.5
20.0
19.5
19.0
18.5
18.0
17.5
17.0
16.5
16.0
15.5
15.0
2/19/2018 12:57
2/19/2018 13:12
2/19/2018 13:26
2/19/2018 13:40
2/19/2018 13:55
2/19/2018 14:09
2/19/2018 14:24
2/19/2018 14:38
2/19/2018 14:52
2/19/2018 15:07
2/19/2018 15:21
2/19/2018 15:36
2/19/2018 15:50
2/19/2018 16:04
2/19/2018 16:19
Date, Time


ALUM eClO2 HD Laboratory RH
Relative Humidity (%)
70
65
60
55
50
45
40
35
30
25
20
15
10
5
2/19/2018 12:57
2/19/2018 13:12
2/19/2018 13:26
2/19/2018 13:40
2/19/2018 13:55
2/19/2018 14:09
2/19/2018 14:24
2/19/2018 14:38
2/19/2018 14:52
2/19/2018 15:07
2/19/2018 15:21
2/19/2018 15:36
2/19/2018 15:50
2/19/2018 16:04
2/19/2018 16:19
Date, Time

United States
Environmental Protection
Agency

Office of Research and Development (8101R)
Washington, DC 20460

Official Business
Penalty for Private Use
$300

PRESORTED STANDARD
POSTAGE & FEES PAID
EPA
PERMIT NO. G-35