U.S. ARMY

U.S. ARMY COMBAT CAPABILITIES DEVELOPMENT COMMAND
CHEMICAL BIOLOGICAL CENTER
ABERDEEN PROVING GROUND, MD 21010-5424

DEVCOM CBC-TR-1873

Methodology Assessment for Chemical Hazard Mitigation Technology Evaluations Using Multiple Panel Types

Brent A. Mantooth
Amanda M. Schenning
Doug S. Nichols
Thomas P. Pearl
Neil A. Hawbaker
John P. Davies
Michelle L. Sheahy
Janlyn H. Eikenberg
Joseph P. Myers
David G. Gehring

RESEARCH AND OPERATIONS DIRECTORATE

James R. Burns

ENGINEERING DIRECTORATE

Michael J. Chesebrough

DCS CORPORATION
Belcamp, MD 21017-1561

Jill L. Ruth
Craig S. Schenning

LEIDOS, INC.
Reston, VA 20190-5617

June 2024

Distribution Statement A. Approved for public release: distribution is unlimited.

Disclaimer

The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorizing documents.

REPORT DOCUMENTATION PAGE

1. REPORT DATE	2. REPORT TYPE	3. DATES COVERED	
		START DATE	END DATE
XX-06-2023	Final	Oct 2021	Dec 2023

4. TITLE AND SUBTITLE
Methodology Assessment for Chemical Hazard Mitigation Technology Evaluations Using Multiple Panel Types

5a. CONTRACT NUMBER	5b. GRANT NUMBER	5c. PROGRAM ELEMENT NUMBER

5d. PROJECT NUMBER	5e. TASK NUMBER	5f. WORK UNIT NUMBER
CB10409		

6. AUTHOR(S)
Mantooth, Brent A.; Schenning, Amanda; Nichols, Doug S.; Pearl, Thomas P.; Hawbaker, Neil A.; Sheahy, Michelle L.; Eikenberg, Janlyn H.; Myers, Joseph P.; Gehring, David G.; Davies, John P.; Burns, James R. (DEVCOM CBC); Chesebrough, Michael J.(DCS); Schenning, Craig S.; Ruth, Jill L. (Leidos)

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Director, DEVCOM CBC, ATTN: FCDF-CBR-PD, APG, MD 21010-5424
DCS Corporation; 4696 Millennium Drive, Suite 450, Belcamp MD 21017-1561
Leidos Inc.; 1750 Presidents Street, Reston, VA 20190-5617

8. PERFORMING ORGANIZATION REPORT NUMBER
DEVCOM CBC-TR-1873

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)	10. SPONSOR/MONITOR'S ACRONYM(S)	11. SPONSOR/MONITOR'S REPORT NUMBER(S)
Defense Threat Reduction Agency, 8725 John J. Kingman Road, MSC 6201, Fort Belvoir, VA 22060-6201	DTRA	

12. DISTRIBUTION/AVAILABILITY STATEMENT
Distribution Statement A. Approved for public release: distribution is unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT (LESS THAN 200 WORDS)
This report presents a methodology assessment for chemical hazard mitigation technology testing using multiple panel types. The methodology is used to analyze decontaminant performance and to translate the laboratory results to enable operationally relevant health-based assessments for vapor and contact exposure. A new shim panel (two materials separated by a variable thickness shim spacer) is introduced to quantify the effects of materials configurations that limit fluid access on agent retention and decontaminant performance. This effort employs the retained agent (RA) analysis method, which determines the total quantity of agent associated with a panel (or similar) after a treatment procedure. The methodology assessment includes aspects of verification and validation to establish the reproducibility of the output. A new scale-up calculation is established that utilizes per drop-normalized retained agent data from panels to predict the retained agent response on full-scale assets. Contamination and decontamination evaluations of small assets were performed in this assessment to demonstrate prediction within a factor of 2–10. Additionally, a calculation for maximum potential dose is introduced that assumes that all agent mass associated with the asset produces vapor or contact exposures, which enables the use of retained agent data to evaluate technologies to health-based decontamination requirements.

15. SUBJECT TERMS

Decontamination	Hazard Mitigation	Retained Agent
Chemical warfare agent (CWA)	Materials	Maximum Potential Dose
Shim Panel	Scale up	Methodology Assessment

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES
a. REPORT	b. ABSTRACT	C. THIS PAGE		
U	U	U	UU	270

19a. NAME OF RESPONSIBLE PERSON	19b. PHONE NUMBER *(Include area code)*
Renu B. Rastogi	(410) 436-7545

STANDARD FORM 298 (REV. 5/2020)
Prescribed by ANSI Std. Z39.18

Blank

PREFACE

The work described in this report was authorized under project no. CB10409. The work was started in October 2021 and completed in December 2023.

The use of either trade or manufacturers' names in this report does not constitute an official endorsement of any commercial products. This report may not be cited for purposes of advertisement.

U.S. Army Combat Capabilities Development Command Chemical Biological Center (DEVCOM CBC) was previously known as U.S. Army Edgewood Chemical Biological Center (ECBC).

The text of this report is published as received and was not edited by the Technical Releases Office, U.S. Army Combat Capabilities Development Command Chemical Biological Center.

This report has been approved for public release.

Acknowledgments

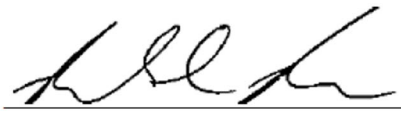
The authors acknowledge the following individuals for their hard work and assistance with the execution of this technical program:

- Dr. Charles Bass, Dr. Glenn Lawson (Defense Threat Reduction Agency, Joint Science and Technology Office [DTRA JSTO]; Fort Belvoir, VA), and Dr. James Noah (contractor for DTRA JSTO; Man-Machine Systems Assessment; Arlington, VA) for their financial and programmatic support of this program;
- Ms. Melissa Smith and Katrina Jewell (US Marine Corp, JPEO-JPM P, Stafford VA) for their financial support of this program;
- Dr. Terrence D'Onofrio, Dr. Mark Varady (U.S. Army Combat Capabilities Development Command, Chemical Biological Center [DEVCOM CBC]; Aberdeen Proving Ground, MD), Dr. Alden Adrion (U.S. Army Test and Evaluation Command; Aberdeen Proving Ground, MD), Mr. Joe Novick (Naval Surface Warfare Center; Stafford, VA), and Mr. Jim Nock (Evaltek Inc., contractor for OPTEVFOR Navy, Norfolk, VA) for their technical peer review and helpful discussions;
- DEVCOM CBC Decontamination Sciences Branch members for their efforts in performing this study.

Blank

Chemical Hazard Mitigation Technology Evaluations Using Multiple Panel Types

The Contamination Mitigation (CM) Capability Area Process Action Team (CAPAT) concurs with the methodology and best practice calculation guidance contained here. T&E WIPTs will determine how and where this methodology and best practice calculation guidance are implemented into their program for use. Formal implementation of the methodologies described here will follow the Test Operations Procedure process. For any non-concurrences, a dissenting paper will be attached.

Organization	Signature	Date
Army Test and Evaluation Office (SAAL-TE)	HOLSTE.MEGAN.JEANETTE.1284239390 Digitally signed by HOLSTE.MEGAN.JEANETTE.1284239390 Date: 2024.05.19 13:58:42 -04'00' Megan Holste	
Joint Program Executive Office of Chemical Biological Defense (JPEO-CBD) Test & Evaluation	NIBLICK.CHRISTOPHER.JAMES.1256873113 Digitally signed by NIBLICK.CHRISTOPHER.JAMES.1256873113 Date: 2024.05.15 15:44:00 -04'00' Chris Niblick	20240515
Joint Requirements Office for Chemical, Biological, Radiological and Nuclear Defense (JRO-CBRND)	 MAJ Michael C. Moore	09 Apr 2024
Joint Science and Technology Office (JSTO)	ODELL.BRETT.KYLE.1274940553 Digitally signed by ODELL.BRETT.KYLE.1274940553 Date: 2024.05.03 08:44:25 -04'00' Brett Odell	3 May 2024
US Army Evaluation Center (AEC)	HUGHES.JULIANE.OLSEN.1285055837 Digitally signed by HUGHES.JULIANE.OLSEN.1285055837 Date: 2024.04.05 11:36:05 -04'00' Juliane Hughes	
Operational Test and Evaluation Force (OPTEVFOR)	THIERING.JOSEPH.LEE.1257775557 Digitally signed by THIERING.JOSEPH.LEE.1257775557 Date: 2024.05.30 10:54:54 -04'00' Joseph Thiering	

Air Force Operational Test and Evaluation Center (AFOTEC)	KLEIN.TERRY.L.1288435061 Digitally signed by KLEIN.TERRY.L.1288435061 Date: 2024.05.03 11:05:55 -05'00' Maj Terry Klein	
Marine Corps Operational Test & Evaluation Activity (MCOTEA)	WADLEY.MICHAEL.CRAIG.1130810841 Digitally signed by WADLEY.MICHAEL.CRAIG.1130810841 Date: 2024.04.16 09:49:38 -04'00' Michael Wadley	
Contamination Mitigation (CM) CAPAT Co-Chair	ADRION.ALDEN.CHARLES.1543451777 Digitally signed by ADRION.ALDEN.CHARLES.1543451777 Date: 2024.04.22 13:59:34 -04'00' Alden Adrion	
CM CAPAT Co-Chair	BURNS.JAMES.R.1276086134 Digitally signed by BURNS.JAMES.R.1276086134 Date: 2024.05.02 16:03:06 -04'00' Jim Burns	

EXECUTIVE SUMMARY

This report presents a methodology assessment for chemical hazard mitigation technology testing using multiple panel types. The methodology is used to generate retained agent data elements that are used in subsequent calculations to translate the laboratory results to enable operationally relevant vapor and contact exposure health-based assessments regarding personnel interactions with decontaminated assets. This report covers the full process from laboratory measurement of the data elements through data normalization, scale-up, and a worst-case exposure assessment estimate, a maximum potential dose, that can be compared to health-based vapor and contact requirements to assess hazard mitigation technologies.

Asset-level responses were estimated using new calculations that greatly enhance panel data use and analysis capability. Specifically, this work demonstrates a new paradigm of analysis capabilities to represent full-scale assets in operational context using laboratory-derived results. This effort employs the retained agent (RA) analysis method, which determines the total quantity of agent associated with a panel (or similar) after a treatment procedure. This new calculation approach requires that multiple *source term* inputs describing the RA on a per drop basis from each agent-material-feature are acquired in testing. An emphasis is placed on identifying the critical laboratory data to acquire that facilitates acceptable accuracy in asset-level outputs. A core philosophy applied in the development of this methodology was to *identify the simplest approach to answer the relevant questions with acceptable accuracy using the least resources necessary*.

A new shim panel (two materials separated by a variable thickness shim spacer) is introduced to quantify the effects of materials configurations that limit fluid access on agent retention and decontaminant performance. The agent entrained in the shim panel gap is much harder to decontaminate, data collected here showed cases where efficacy was almost 2000 times less than observed on traditional flat panel testing. As a result, these features may be responsible for a disproportionate contribution of potential exposure to personnel, that was not captured in previous test methodologies. Incorporating shim panels into decontamination evaluations is critical to provide accurate descriptions of asset-level post-decontamination hazards. The shim panels were designed to be testable using current test infrastructure.

This methodology assessment spans the introduction of shim panels and the implementation of new calculation approaches including per drop normalization and the scale-up calculations using RA data. The methodology assessment includes a characterization of the reproducibility of the RA data elements (i.e., source term outputs). This methodology assessment considered liquid and vapor phase hazard mitigation treatment procedures for flat and shim panels that have been contaminated with liquid chemical warfare agent.

A significant new capability includes scale-up calculations that use laboratory results to represent full-scale assets. It has been demonstrated that starting challenge alone insufficiently specifies a contamination condition for an accurate and conclusive measurement of performance for hazard mitigation technologies.[1] Efficacy results can strongly depend on the drop volumes used to generate a starting challenge on materials. This implies that if the laboratory tested agent drop volume does not match the operational drop volumes, the scale-up calculations may not be accurate. A new data analysis approach that accounts for variable liquid drop volume and count was developed and implemented. This per drop normalization approach captures the effect of drop volume and variable contamination density and is the foundation for

the scale-up of laboratory test conditions to operational context. One impact of this approach is that *data analyzed on a per drop basis can be used to calculate the asset level response for any contamination density*.

The new scale-up calculation accounts for how each agent drop contributes to the total retained agent mass of the asset; the whole is equal to the sum of the RA per drop. The laboratory generated RA per drop provides a *source term* that is aggregated to represent an asset. This requires that laboratory testing produces the RA source terms for each material-feature combination found on an asset with which agent interacts. This data use provides a shift in perspective in the objectives of laboratory RA data from strictly characterizing *efficacy* as defined by log reduction to also include efficacy in terms of mitigation of health effects at the asset level. To assess the accuracy of the scale-up calculation small assets were tested in the laboratory using highly controlled contamination. The results of the small asset test were compared to the laboratory data scale-up calculation and demonstrated prediction within the established acceptance criterion, within a factor of 10 (over or under prediction).

Once the scale-up calculation is performed, new analysis capabilities are possible. The asset-level RA represents the total mass of agent that could result in an exposure to personnel. A calculation for maximum potential dose (MPD) is introduced that assumes that all agent mass associated with the asset produces vapor or contact exposures, which enables the use of RA data to evaluate technologies to health-based decontamination requirements. This MPD output provides an upper bound (worst case) exposure estimate associated with the asset of interest. The use of RA data to produce an MPD output is estimated to be *20 times less resource intensive* (i.e., 20 times less resources and time) to characterize compared to acquiring the vapor emission and contact transfer data used to perform explicit exposure assessments. It is an ideal first step to build an understanding of how operational conditions and mitigation technology factors integrate to enable the mitigation of potential health effects in personnel using the decontaminated assets.

Overall, the methodology acceptance criteria were met, with a few documented exceptions. The control samples demonstrated reproducible and accurate contaminant delivery and recovery. Multiple treatment processes were characterized on a wide range of samples. This report shows that per drop normalization and asset scale-up approaches can accurately represent the agent retained by small assets. From these demonstrations, the methods are shown to provide the reproducibility and accuracy to enable the test and evaluation of hazard mitigation technologies. This methodology assessment provides the verification for the shim panel to transition to a useable test instrument and validation of the associated methodology to use laboratory data to represent full-scale assets. A recommended procedural method for inclusion into a future TOP and best practice calculation guidance is provided as a separate report.

1. Mantooth, B.A.; Pearl, T.P.; Stevenson, S.M.; Smallwood, S.Q.; Eikenberg, J.H.; Sheahy, M.L.; Gehring, D.G.; Chesebrough, M.J.; Ruth, J.L. *Normalization of Contamination Conditions for Measurement of Agent Retention and Decontamination Performance on Materials*; CCDC CBC-TR-1767; DEVCOM Chemical Biological Center: Aberdeen Proving Ground, MD, 2022. UNCLASSIFIED Report, Public Release (AD1170213).

CONTENTS

PREFACE iii

EXECUTIVE SUMMARY v

1. INTRODUCTION 1

1.1 Methodology Development 3
1.1.1 Overview of New Methods 3
1.1.2 Shim Panels 4
1.2 Methodology Assessment Criteria 5
1.2.1 Verification Overview 5
1.2.2 Validation Overview 7
1.3 Laboratory Scale-Up Capability and Health Effect Outputs 9
1.3.1 Mapping Laboratory Data to Operational Context 9
1.3.2 Maximum Potential Dose 10
1.3.3 Asset Testing Limitations versus Panel Testing Capabilities 11
1.3.4 Inputs and Data Elements to Produce Health-Based Outputs 12
1.4 Capabilities, Assumptions, and Limitations 14

2. MATERIALS AND PROCESSES FOR PANEL TESTING 16

2.1 Materials 16
2.2 Contamination 17
2.3 Decontamination Treatments 19
2.4 Panel Extraction and Chromatography 21
2.5 Dose Confirmation Samples 22
2.6 Laboratory Temperature and Humidity 23

3. EXTRACTION EFFICIENCY 24

3.1 Test Design 24
3.2 Results 25
3.2.1 HD Extraction Efficiency 25
3.2.2 VX Extraction Efficiency 28
3.3 Summary 29

4. REPRODUCIBLE AGENT DELIVERY TO PANELS 29

4.1 Test Design 29
4.2 Results 31
4.2.1 HD Positive Controls 33
4.2.2 VX Positive Controls 40
4.3 Summary 46

5. SENSITIVITY ANALYSIS FOR MATERIAL SELECTION 48

5.1 Test Design 49
5.2 Results 50
5.2.1 HD Data for Steel 51
5.2.2 HD Data for Alkyd Coating 52
5.2.3 HD Data for Polyurethane Coating 52
5.2.4 VX Data for Steel 54
5.2.5 VX Data for Alkyd Coating 55
5.2.6 VX Data for PU Coating 55
5.3 Comparison Between Horizontal and Vertical Orientations 56
5.4 Summary 58

6. SOURCE TERM AND SCALE-UP CALCULATIONS 59

6.1 Scale-up Calculations 60
6.2 Random Sampling Monte Carlo Techniques 61
6.3 Data Normalization and Variable Agent Drop Volume Regression 62
6.4 Source Term Inputs for Monte Carlo Calculations 64
6.4.1 Direct Source Terms 64
6.4.2 Multivolume Fit Source Terms 64
6.4.3 Indirect Drop Volume Source Terms 65
6.5 Quantifying Efficacy with Log Difference 66
6.6 Confidence, Prediction, and Tolerance Intervals 66
6.7 Relative Contributions 69
6.8 Assessment of Scale-up Accuracy 70
6.9 Limit of Quantitation Considerations 71

7. DECONTAMINATION TREATMENT SOURCE TERM DATA 71

7.1 Test Description 72
7.1.1 Material Selection 73
7.1.2 Treatment Processes 73
7.1.3 Criteria for Validation 74
7.2 Soapy Water Treatment 74
7.2.1 DCS Results 74
7.2.2 Steel 76
7.2.3 Alkyd Coating 81
7.2.4 Polyurethane Coating 84
7.2.5 Silicone Elastomer 87
7.3 CHAD Treatment Results 90
7.3.1 DCS Results 90
7.3.2 Steel 90
7.3.3 Alkyd Coating 95
7.3.4 Polyurethane Coating 98
7.3.5 Silicone Elastomer 101
7.4 Trends in RA Variability 104
7.5 Significance of Entrained Agent 106
7.6 Source Term Fitting Accuracy - RMSE 107

7.7 Summary 109

8. REPRODUCIBILITY ESTIMATION VIA INTERMEDIATE PRECISION STANDARD DEVIATION 110

8.1 Definitions for IPSD Calculations 110
8.2 Calculations 112
8.3 Statistical Outliers and IPSD Results 113
8.4 IPSD Testing Over Multiple Days 113
8.5 IPSD Results 117

9. MIXED MATERIAL SHIM PANELS 120

9.1 Test Design 120
9.2 Results 121

10. ASSET TESTING RESULTS 125

10.1 Asset Contamination and Source Term Selection 125
10.2 Treatment Process 127
10.3 Results 128

11. SCALE-UP CALCULATION ACCURACY ASSESSMENT 130

11.1 Source Term Inputs 130
11.2 Asset Level Results 132
11.2.1 Multi-tool Asset with 0.2 µL Drops and Soapy Water Spray Treatment 132
11.2.2 Multi-tool Asset with 1 µL Drops and Soapy Water Spray Treatment 138
11.3 Accuracy Summary for M_{Asset} Results 141
11.4 Accuracy of M_{Asset} for Other Conditions 145
11.5 Considerations for Future Test Designs 146

12. MAXIMUM POTENTIAL DOSE DEMONSTRATION 149

12.1 Percutaneous Exposure 149
12.2 Vapor Exposure Dose 151
12.3 Health Effects Outputs Are Systems Level Interactions 154
12.4 Summary 155

13. CONCLUSIONS 156

13.1 New Capabilities 156
13.2 Verification 157
13.3 RA Data Element Variability 157
13.4 Sensitivity Analysis 158
13.5 Per Drop Normalization 159
13.6 Scale-up Calculation 160

13.7 Maximum Potential Dose Health Effect Output 160
13.8 Methodology Implementation 161

ACRONYMS AND ABBREVIATIONS 166

Appendix A : Methodology Development Background 168
Appendix B : Data Transforms and Calculations 189
Appendix C : Starting Challenge - Relationship of Environmental Contamination Density to Asset Contamination 196
Appendix D : Temperature and Humidity Summary Table 202
Appendix E : Effects of Panel Orientation 203
Appendix F : Monte Carlo Techniques 209
Appendix G : Interpretation of Regression Slope and Accuracy of Indirect Calculation Assumptions 213
Appendix H : Asset Scale-up Calculations 222

FIGURES

1. Shim panel assembly diagram (left) and image of a steel shim panel with 1 µL of liquid contaminant (right), drops may or may not bridge the top and bottom materials. 5
2. Conceptual contaminated vehicle asset, pink indicates variable volume agent drops contaminating various parts of the vehicle (left). Controlled contamination of a small asset used to demonstrate the methodology (right), numbered red dots indicate the location of contamination. 9
3. High-level flow chart illustrating relationship of vignette inputs, data elements, and outputs. 13
4. Treatment process for all panels, shim panel used in illustration. 16
5. Painted shim panel parts (left) and assembled (right) for coated materials. 17
6. Photographs of the Gilson pipette (left) and Masterflex syringe pump (right) contaminant delivery tools. 19
7. Image of the Thermotron chamber used for the CHAD treatment. 20
8. Image of a Preval sprayer with 16 oz reservoir that generates a liquid aerosol spray. 21
9. Extraction efficiency of HD with chloroform solvent measured with GC-MSD. Raw RA results are presented in the top graph, calculated extraction efficiency and CV (%) are provided in bottom graphs. Error bars for EE represent one standard deviation. 26
10. Extraction efficiency of HD with IPA solvent measured with GC-QQQ. Raw RA results are presented in the top graph, calculated extraction efficiency and CV (%) are provided in bottom graphs. Error bars for EE represent one standard deviation. 27
11. Extraction efficiency of VX with IPA solvent. Raw RA results are presented in the top graph, calculated extraction efficiency and CV (%) are provided in bottom graphs. Error bars for EE represent one standard deviation. 28
12. Images of dosing PU coated shim panels with a syringe and steel shim panels with a pipette using methyl salicylate with a red dye, for 0.02 inch and 0.002 inch shims. The sequence of images from left to right spans no more than 30 s. 30
13. Positive control samples for 0.2 µL drops of HD using a syringe. Individual RA data points are provided in the top graph, calculated RA/DCS ratio in the bottom graph. The green shaded region indicates the acceptance criterion of 1 ± 0.2. 34
14. Positive control samples for 0.2 µL drops of HD using a P2 pipette. 35
15. Positive control samples for 1 µL drops of HD using a syringe. Data points indicated with an × were excluded from the statistical analysis 36
16. Positive control samples for 1 µL drops of HD using a M10 pipette. 37
17. Positive control samples for 4 µL drops of HD using a syringe. 38
18. Positive control samples for 4 µL drops of HD using a M10 pipette. 39
19. Positive control samples for 0.2 µL drops of VX using a syringe. 40
20. Positive control samples for 1 µL drops of VX using a syringe. Data points indicated with an × were excluded from the statistical analysis 42
21. Positive control samples for 1 µL drops of VX using a M10 pipette. 43
22. Positive control samples for 4 µL drops of VX using a syringe. Data points indicated with an × were excluded from the statistical analysis 44
23. Positive control samples for 4 µL drops of VX using a M10 pipette. 45

24. Summary of RA/DCS responses and CV for all positive control samples as a box and whisker plots comparing pipette and syringe delivery. 47
25. Sensitivity data for HD on steel with soapy water spray. 51
26. Sensitivity data for HD on Alkyd coating with soapy water spray. 52
27. Sensitivity data for HD on PU coating with soapy water spray. 53
28. Sensitivity data for VX on steel with soapy water spray. 54
29. Sensitivity data for VX on Alkyd coating for soapy water spray. 55
30. Sensitivity data for VX on PU shim panels. LD is relative to the applied mass. 56
31. LD comparing RA for the horizontal to vertical orientations for Alkyd, PU, and steel materials with HD and VX. Error bars represent the 95% CI for the LD. Grey shaded region indicates the difference threshold of ± 0.4 LD. 57
32. Statistical moments and intervals for a log-normal distribution, pink circles demonstrate an example dataset of five replicates with the indicated μ and σ. 69
33. Variable agent drop volume results for HD on Steel with Soapy Water Spray treatment. The shaded region represents antilog($\mu_{fit}(V)$ ± RMSE). Data values are scattered in the x axis to facilitate visualization. 76
34. Inverse transformed indirect fits for HD on steel with soapy water spray for 0.2 μL drops (left) and 1 μL drops (right). Asterisks (*) indicate the specific data values used to produce each indirect fit. The RMSE of the indirect fit is indicated in the legend, the σ indicates the log transformed standard deviation of the specific drop volume data used (Table 39). The shaded region corresponds to antilog($\mu_{Indirect}(V) \pm \sigma$) 79
35. LD as a function of agent drop volume compared to DCS for HD on steel with soapy water spray. Graphs for the fit (left), indirect with 0.2 μL data (middle), and indirect with 1 μL data (right). Data points with error bars are discretely calculated LD values with 95% CI for 1 and 0.2 μL drops. Data points are slightly offset in x to facilitate visualization. 80
36. Variable agent drop volume results for HD on Alkyd coating with Soapy Water Spray. The shaded region represents antilog($\mu_{fit}(V)$ ± RMSE). Datapoints have been slightly offset in the x-axis to facilitate visualization. 81
37. Inverse transformed indirect fits for HD on Alkyd coating with soapy water spray for 0.2 μL drops (left) and 1 μL drops (right). Asterisks (*) indicate the specific data values used to produce each indirect fit. The shaded region corresponds to antilog($\mu_{Indirect}(V) \pm \sigma$). 82
38. LD as a function of agent drop volume compared to DCS for HD on Alkyd coating with soapy water spray. Data points with error bars are specifically calculated LD values with 95% CI for 1 and 0.2 μL drops, data are slightly offset in x to facilitate visualization. 83
39. Variable agent drop volume results for HD on PU coating with Soapy Water Spray. The shaded region represents antilog($\mu_{fit}(V)$ ± RMSE). Datapoints have been slightly offset in the x-axis to facilitate visualization. 84
40. Inverse transformed indirect fits for HD on PU coating with soapy water spray for 0.2 μL drops (left) and 1 μL drops (right). Asterisks (*) indicate the specific data values used to produce each indirect fit. The shaded region corresponds to antilog($\mu_{Indirect}(V) \pm \sigma$). 85

41. LD as a function of agent drop volume compared to DCS for HD on PU with soapy water spray. Data points with error bars are specifically calculated LD values with 95% CI for 1 and 0.2 µL drops, data are slightly offset in x to facilitate visualization. 86
42. Variable agent drop volume results for HD on Silicone with Soapy Water Spray treatment. The shaded region represents antilog($\mu_{fit}(V)$ ± RMSE). Datapoints have been slightly offset in the x-axis to facilitate visualization. 87
43. Inverse transformed indirect fits for HD on silicone with soapy water spray for 0.2 µL drops (left) and 1 µL drops (right). Asterisks (*) indicate the specific data values used to produce each indirect fit. The shaded region corresponds to antilog($\mu_{Indirect}(V) \pm \sigma$). 88
44. LD as a function of agent drop volume compared to DCS for HD on silicone with soapy water spray. Data points with error bars are specifically calculated LD values with 95% CI for 1 and 0.2 µL drops, data are slightly offset in x to facilitate visualization. 89
45. Variable agent drop volume results for HD on Steel with CHAD treatment. The shaded region represents antilog($\mu_{fit}(V)$ ± RMSE). Datapoints have been slightly offset in the x-axis to facilitate visualization. 91
46. Inverse transformed indirect fits for HD on steel with CHAD for 0.2 µL drops (left) and 1 µL drops (right). Asterisks (*) indicate the specific data values used to produce each indirect fit. The shaded region corresponds to antilog($\mu_{Indirect}(V) \pm \sigma$). 93
47. LD as a function of agent drop volume compared to DCS for HD on steel with CHAD. Data points with error bars are specifically calculated LD values with 95% CI for 1 and 0.2 µL drops, data are slightly offset in x to facilitate visualization. 94
48. Variable agent drop volume results for HD on Alkyd coating with CHAD. The shaded region represents antilog($\mu_{fit}(V)$ ± RMSE). Datapoints have been slightly offset in the x-axis to facilitate visualization. 95
49. Inverse transformed indirect fits for HD on alkyd coating with CHAD for 0.2 µL drops (left) and 1 µL drops (right). Asterisks (*) indicate the specific data values used to produce each indirect fit. The shaded region corresponds to antilog($\mu_{Indirect}(V) \pm \sigma$). 96
50. LD as a function of agent drop volume compared to DCS for HD on Alkyd with CHAD. Data points with error bars are specifically calculated LD values with 95% CI for 1 and 0.2 µL drops, data are slightly offset in x to facilitate visualization. 97
51. Variable agent drop volume results for HD on PU coating with CHAD. The shaded region represents antilog($\mu_{fit}(V)$ ± RMSE). Datapoints have been slightly offset in the x-axis to facilitate visualization. 98
52. Inverse transformed indirect fits for HD on PU coating with CHAD for 0.2 µL drops (left) and 1 µL drops (right). Asterisks (*) indicate the specific data values used to produce each indirect fit. The shaded region corresponds to antilog($\mu_{Indirect}(V) \pm \sigma$). 99
53. LD as a function of agent drop volume compared to DCS for HD on PU coating with CHAD. Data points with error bars are specifically calculated LD values with 95% CI for 1 and 0.2 µL drops, data are slightly offset in x to facilitate visualization. 100
54. Variable agent drop volume results for HD on Silicone with CHAD treatment. The shaded region represents antilog($\mu_{fit}(V)$ ± RMSE). Datapoints have been slightly offset in the x-axis to facilitate visualization. 101
55. Inverse transformed indirect fits for HD on silicone with CHAD for 0.2 µL drops (left) and 1 µL drops (right). Asterisks (*) indicate the specific data values used to produce each indirect fit. The shaded region corresponds to antilog($\mu_{Indirect}(V) \pm \sigma$). 102

56. LD as a function of agent drop volume compared to DCS for HD on Silicone with CHAD. Data points with error bars are specifically calculated LD values with 95% CI for 1 and 0.2 µL drops, data are slightly offset in x to facilitate visualization. 103
57. Standard deviation of the log transformed RA data (σ), for 0.2 and 1 µL drops across materials, features, and treatment procedures. The dashed line represents the average σ for DCS of 0.06 $\log_{10}(\mu g)$. 104
58. Magnitude and variability of the RA data shown as µ ± σ, text values provide the CV_{log}. 105
59. Source term summary for 1 µL drops across materials and feature sizes for CHAD (top) and Soapy Water Spray (bottom). Values in parenthesis are factor differences (i.e., how many times greater) for the shim panels from the equivalent flat panel condition. 107
60. RMSE trends for each analysis technique across materials and features (lower RMSE indicates a better fit). 108
61. RA across test days for CHAD with 1 µL HD drop volume. Each color represents results collected on a different test session. Individual sample RA results are provided in the top graph, the bottom portion of each graph illustrates the arithmetic mean of the log-transformed RA. Error bars represent one standard deviation of the log-transformed RA. 114
62. RA across test days for Soapy Water Spray with 1 µL HD drop volume. Each data grouping/color represents results collected on a different test session. Error bars represent one standard deviation of the log-transformed RA. 114
63. $IPSD_T$ outputs for each material-feature-treatment. 119
64. Influence of material interfaces on 2,5 lutidine simulant vapor emission, no mitigation treatment applied (from ref[3]). 120
65. Mixed material shim panel results for 1 µL drops of HD with soapy water spray. Top row provides the individual RA data points. The bottom row provides the mean and standard deviation of the log-transformed RA data. Star denotes the condition for similar materials. 122
66. LD comparison of RA for common material (e.g., PU bottom-PU top) to mixed material (e.g., PU bottom-steel top) shim panels. The silicone shim panels always used steel for the top material which is used as the common material. The grey region illustrates the ± 0.4 LD difference threshold. 122
67. Mixed material shim panel results for 1 µL drops of HD with CHAD. Top row provides the individual RA data points, bottom row provides the mean and standard deviation of the log-transformed RA data. 123
68. LD comparison of RA for common material (e.g., PU bottom-PU top) to different material (e.g., PU bottom-steel top) shim panels. The silicone shim panels always used steel for the top material which is used as the common material. The grey region illustrates the ± 0.4 LD difference threshold. 124
69. Illustration of locations for drop placement on the multi-tool. 126
70. Illustration of locations for drop placement on the Picatinny rail accessory. 127
71. Asset RA results for CHAD and soapy water spray treatments. The top of each graph shows the individual data points, points are jittered in the x axis for better visualization. The lower graph shows the arithmetic mean and standard deviation of the $\log_{10}$ transformed RA. 128

72. Comparison CHAD treatment $\mu^* \pm \sigma^*$ values for each fit approach for 0.2 µL drops (left) and 1 µL drops (right). 131
73. Comparison soapy water spray treatment $\mu^* \pm \sigma^*$ values for each fit approach for 0.2 µL drops (left) and 1 µL drops (right). 132
74. MC output visualization for the multi-tool asset with 0.2 µL HD drops treated with soapy water spray. The circle with error bars to the right of each swarm plot represent the geometric mean and 95% PI for the data. 134
75. Histogram of the M_{Asset} output fit to a log-normal distribution (red line), for the multi-tool asset with 0.2 µL HD drops treated with soapy water spray 136
76. Relative contribution and mass contribution graphs for each feature for the multi-tool asset with 0.2 µL HD drops treated with soapy water spray, values in parenthesis are the median response for the material-feature indicated. 137
77. MC output visualization for the multi-tool asset with 1 µL HD drops treated with soapy water spray. The circle with error bars to the right of each swarm plot represent the geometric mean and 95% PI for the data. 138
78. Histogram of the M_{Asset} output fit to a log-normal distribution (red line), for the multi-tool asset with 0.2 µL HD drops treated with soapy water spray. 140
79. Relative contribution and mass contribution graphs for each feature for the multi-tool asset with 1 µL HD drops treated with soapy water spray, values in parenthesis are the median response for the material-feature indicated. 141
80. Summary output of M_{Asset} calculations for CHAD and soapy water spray treatments, circle shows the geometric mean, error bars correspond to the 95% PI, the numerical value is R (predicted / measured). 142
81. Trends for the ratio R for geometric mean (predicted / measured) with each source term calculation technique. The text values indicate the $R_{Absolute}$ magnitude. Green shading indicates the validation criterion of 0.1–10. 143
82. Comparison of a decaying vapor emission rate with a steady-state emission approximation. 152

TABLES

1. Summary of methodology changes relative to TOP 08-2-061B and TOP-08-2-060A. 3
2. Method verification components 6
3. Sensitivity data demonstration 6
4. Method validation components. 8
5. Factors influencing health-based outputs. 14
6. Panel material information. 17
7. Liquid density at room temperature and typical purity for the different contaminants used. 18
8. Liquid dispensing tools used to deliver agent. 18
9. Decontamination treatment information. 19
10. Performance specifications for the CHAD chamber. 20
11. Chromatography overview for the analytes of interest. 21
12. Instrument parameters for liquid samples on the GC-QQQ 22
13. Test parameters for verification of shim panel extraction. 24
14. Statistical Results for HD EE using IPA 27
15. Statistical Results for VX EE using IPA 29
16. Test parameters for verification of contaminant delivery and recovery. 31
17. Nominal mass delivered per drop volume 32
18. DCS accuracy compared to $M_{Nominal}$ 33
19. Statistics for positive control samples for 0.2 µL drops of HD using a syringe 35
20. Statistics for positive control samples for 0.2 µL drops of HD using a P2 pipette 36
21. Statistics for positive control samples for 1 µL drops of HD using a syringe 37
22. Statistics for positive control samples for 1 µL drops of HD using a M10 pipette 38
23. Statistics for positive control samples for 4 µL drops of HD using a syringe 39
24. Statistics for positive control samples for 4 µL drops of HD using a M10 pipette 40
25. Statistics for positive control samples for 0.2 µL drops of VX using a syringe 41
26. Statistics for positive control samples for 1 µL drops of VX using a syringe 43
27. Statistics for positive control samples for 1 µL drops of VX using a pipette 44
28. Statistics for positive control samples for 4 µL drops of VX using a syringe 45
29. Statistics for positive control samples for 4 µL drops of VX using a pipette 46
30. Test parameters to characterize effects of decontaminant application orientation and panel orientation 50
31. DCS accuracy compared to nominal mass 51
32. Example assignment of drops to specific material-features and drop volumes, based on the Picatinny rail accessory illustrated in Figure 2 60
33. Z values for one-sided tolerance intervals, Z_p is used for probability levels the Z_α value is used for confidence levels 67
34. Tolerance interval $k_1$values for a range of sample sizes (n) for 95% percent confidence (α) for 95% of the population (p) 67
35. 95% confidence *t* values used for calculating PI 68
36. Test parameters to characterize the reproducibility of the RA method on different panel types for two different decontamination treatments. 73
37. Contamination conditions for validation testing per agent-material-treatment. 73

38. DCS statistics for each test for soapy water treatment 75
39. Variable agent drop volume regression results for HD on Steel with Soapy Water Spray treatment. 77
40. Statistical summary for HD on Steel with Soapy Water Spray treatment for 0.2 and 1 μL agent drops 78
41. Comparison of RMSE (in $\log_{10}(\mu g)$ units) for each of the source term fitting techniques for HD on steel with soapy water spray.....79
42. Variable agent drop volume regression results for HD on Alkyd coating with Soapy Water Spray treatment. 81
43. Statistical summary for HD on Alkyd coating with Soapy Water Spray treatment for 0.2 and 1 μL agent drops. 82
44. Comparison of RMSE (in $\log_{10}(\mu g)$ units) for each of the source term fitting techniques for HD on Alkyd coating with soapy water spray 83
45. Variable agent drop volume regression results for HD on PU coating with Soapy Water Spray treatment. 84
46. Statistical summary for HD on PU coating with Soapy Water Spray treatment for 0.2 and 1 μL agent drops. 85
47. Comparison of RMSE (in $\log_{10}(\mu g)$ units) for each of the source term fitting techniques for HD on PU coating with soapy water spray 86
48. Variable agent drop volume regression results for HD on Silicone with Soapy Water Spray treatment. 87
49. Statistical summary for HD on silicone-steel with Soapy Water Spray treatment for 0.2 and 1 μL agent drops. 88
50. Comparison of RMSE (in $\log_{10}(\mu g)$ units) for each of the source term fitting techniques for HD on silicone with soapy water spray.....89
51. DCS statistics for each test for soapy water treatment 90
52. Variable agent drop volume regression results for HD on Steel with CHAD treatment.....91
53. Statistical summary for HD on Steel with CHAD treatment for 0.2 and 1 μL agent drops 92
54. Comparison of RMSE (in $\log_{10}(\mu g)$ units) for each of the source term fitting techniques for HD on steel with CHAD 93
55. Variable agent drop volume regression results for HD on Alkyd coating with CHAD treatment. 95
56. Statistical summary for HD on Alkyd coating with CHAD treatment for 0.2 and 1 μL agent drops. 96
57. Comparison of RMSE (in $\log_{10}(\mu g)$ units) for each of the source term fitting techniques for HD on Alkyd coating with CHAD.....97
58. Variable agent drop volume regression results for HD on PU coating with CHAD treatment. 98
59. Statistical summary for HD on PU coating with CHAD treatment for 0.2 and 1 μL agent drops. 99
60. Comparison of RMSE (in $\log_{10}(\mu g)$ units) for each of the source term fitting techniques for HD on Alkyd coating with CHAD.....100
61. Variable agent drop volume regression results for HD on Silicone with CHAD treatment. 101

62. Statistical summary for HD on silicone-steel with CHAD treatment for 0.2 and 1 µL agent drops. 102
63. Comparison of RMSE (in $\log_{10}(\mu g)$ units) for each of the source term fitting techniques for HD on silicone with CHAD 103
64. Summary of mean RMSE values ($\log_{10}(\mu g)$) across the materials for each treatment-feature combination for the different analysis techniques 109
65. Test conditions for IPSD determination across multiple test sessions 113
66. Statistical summary of multi-day data for CHAD treatment 115
67. Statistical summary for multi-day data for Soapy Water Spray treatment 116
68. REML method output for $IPSD_T$ on log-transformed RA data 117
69. Test parameters to characterize effects of mixed material shim panels....121
70. Test conditions for asset validation testing. 125
71. Assignment of asset drops to panel source data for the multi-tool. 126
72. Assignment of asset drops to panel source data for the Picatinny rail accessory. 127
73. Asset RA Summary Statistics 129
74. Source term input values for µ and σ for each fit type for HD on Steel with CHAD 131
75. Source term input values for µ and σ for each fit type for HD on Steel with soapy water spray 132
76. Description of drop count and drop volumes used to represent the contamination of the multi-tool asset with 0.2 µL drops 133
77. M_{Asset} output for the tested asset and calculation variations for the multi-tool asset with 0.2 µL HD drops treated with soapy water spray. 134
78. Accuracy ratio of the calculation variations for the multi-tool asset with 0.2 µL HD drops treated with soapy water spray. 135
79. Quantile analysis of the M_{Asset} output for the multi-tool asset with 0.2 µL HD drops treated with CHAD. 136
80. Description of drop count and drop volumes used to represent the contamination of the multi-tool asset with 1 µL drops 138
81. M_{Asset} output for the tested asset and calculation variations for the multi-tool asset with 1 µL HD drops treated with soapy water spray 139
82. Accuracy ratio of the calculation variations for the multi-tool asset with 1 µL HD drops treated with soapy water spray. 139
83. Quantile analysis of the M_{Asset} output for the multi-tool asset with 0.2 µL HD drops treated with CHAD. 140
84. Summary statistics for R values for M_{Asset} across the analysis techniques 144
85. Summary statistics for $R_{Absolute}$ values for upper bound intervals for each calculation technique. 144
86. Contamination Test Designs 147
87. Sampling plan options for future testing for each agent-material-feature-treatment combination....147
88. Percutaneous skin concentration output for HD contamination and treatment of Multi-tool assets. 150
89. Percutaneous skin concentration output for *emulated* VX results. 151
90. MPD vapor exposure assessment for two multi-tool assets in an enclosed vehicle for 8 h. 154

METHODOLOGY ASSESSMENT FOR CHEMICAL HAZARD MITIGATION TECHNOLOGY EVALUATIONS USING MULTIPLE PANEL TYPES

1. INTRODUCTION

The purpose of hazard mitigation technologies* for chemical warfare agents is to enable unprotected personnel† to use decontaminated assets‡ without inducing negative health effects. Test and evaluation (T&E) of technology performance (efficacy) needs to account for contaminant distribution on an asset, the ability of the decontaminant to remove the agent, and how much of the agent may result in an exposure to unprotected personnel. A previously identified methodology gap is the ability to measure decontamination efficacy for complex surfaces (i.e., not flat and horizontal) and as a function of small and variable size drop applications. The test methodology assessed in this report directly addresses these gaps.

A methodology assessment plan (MAP) was developed to evaluate the reproducibility and repeatability§ of the retained agent (RA) analysis method on a range of materials in different orientations and configured as flat and shim panels.** RA analysis determines the total quantity of agent associated with a contaminated sample, by solvent extraction, after a treatment procedure. The RA mass is the primary data element produced in laboratory testing as part of this methodology assessment.

Current hazard mitigation test methods are documented in Test Operating Procedure (TOP) documents 08-2-060A and 08-2-061B. This effort supports the consolidation of these TOPs by attempting to *identify the simplest approach to answer the relevant questions with acceptable accuracy using the least resources necessary*. Overall, the methodology assessment includes verification for the shim panel to transition to a useable test instrument and validation of the associated methodology to use laboratory data to represent full-scale assets. This report covers the full process from laboratory measurement of the data elements through data normalization, scale-up, and a worst-case exposure assessment estimate, a maximum potential dose, that can be compared to health-based vapor and contact requirements to assess hazard mitigation technologies. The methodology assessment will:

---

* The term 'hazard mitigation technology' is used interchangeably with 'decontaminant' throughout the document.

† Personnel may be warfighters, first responders, or anyone that may interact with a contaminated object.

‡ An asset is any object that has been decontaminated that personnel interact with after decontamination, example items could include a pocket multi-tool, a radio, a vehicle, a ship, or any commercial or Government material. Its longest dimension may be a few cm, a few m, or larger.

§ Reproducibility and repeatability are evaluated here as *a single laboratory effort*. Repeatability is defined to include multiple laboratories, here this value is approximated using intermediate precision standard deviation (IPSD) for one laboratory across multiple test days.

** The term *panel* is synonymous with *coupon* and refers to any test article, with simplified geometry, meant to represent part of an asset. Panels can be flat, shim, horizontal, or vertical, and can range in size provided they conform to available test infrastructure. This method focuses on the testing of panels, as opposed to assets (which can be full-scale or scaled down equipment items).

1. Demonstrate the ability to measure RA data elements for flat and shim panels with and without treatment procedures:
    1.1. Demonstrate the ability to extract agent from shim panels using control samples
    1.2. Demonstrate shim panel testing on multiple agent-material combinations
    1.3. Reproducibly apply different treatment procedures (e.g., liquid versus vaporous treatments) that remove[††] agent from these materials
    1.4. Quantify repeatability and intermediate-precision-standard deviation (i.e., single laboratory reproducibility)
2. Demonstrate per drop normalization calculations
3. Demonstrate the accuracy of scale-up calculations based on laboratory RA data for a small asset

This methodology assessment follows the format of method verification and validation, although this is not a Verification and Validation as conducted for Test Infrastructure or Models. Here, verification is the assessment that various aspects of the methodology are being conducted correctly, e.g., the correct mass is applied to the panels and the extraction of agent from the panels is sufficient. Validation is associated with the characterization of accuracy and precision (i.e., reproducibility) of the method. Here, validation includes the demonstration of the scale-up calculation to accurately represent the total quantity of agent retained by an asset using laboratory panel data. Throughout the document, data types for verification and validation demonstrations will be associated with each of the goals as numbered above.

This methodology assessment effort focuses on using RA analysis to represent asset-level responses and to translate this response for comparison against health effect-based requirements. Vapor emission (VE) and contact transfer (CT) analysis methods, and associated exposure assessment outputs, are not addressed in this effort (see Appendix A). The shim panel, RA analysis method, and updated calculations, together, comprise a significantly improved capability over previous methods. Future efforts will need to address the VE and CT analysis methods and calculations.

This effort produced 2,464 chromatography results collected in 28 test sessions, where each chromatography measurement corresponds to one panel. Asset test responses were estimated using new calculations and techniques that greatly enhance panel data use and analysis capability. Specifically, this work demonstrates a new paradigm of analysis capabilities to represent full scale assets using laboratory-derived results. This new calculation approach requires that multiple *source term* inputs that describe the RA on a per drop basis from each agent-material-feature are acquired in testing.

Overall, this report demonstrates that the methodology acceptance criteria were met, with a few documented exceptions. The control samples demonstrated reproducible and accurate contaminant delivery and recovery. Multiple treatment processes were characterized on

†† The term remove agent is used to include any reduction in the quantity of agent caused by any process including but not limited to evaporation, physical removal, or chemical reaction.

a wide range of samples. Lastly, as a first demonstration of the laboratory scale-up calculation, this report shows that per drop normalization can be used to accurately represent the agent retained by small assets. Furthermore, an approach to translate these results to health-based outputs is demonstrated. From these demonstrations, the methods are shown to provide the reproducibility and accuracy to enable the test and evaluation of hazard mitigation technologies. A recommended procedural method for inclusion into a future TOP is appended as a supplement to the report.

1.1 Methodology Development

1.1.1 Overview of New Methods

The updated methodology has been titled "Hazard Mitigation Performance Test Methods for Chemical Warfare Agents," and incorporates context from both TOP 08-2-061B (Residual and Contact Mass) and TOP 08-2-060A (Vapor Flux). The method is used to characterize the efficacy of hazard mitigation processes and technologies. While this method assessment report focuses on the shim panel method, the vision for the new hazard mitigation methodology as a whole is summarized in Table 1 for context.

Table 1. Summary of methodology changes relative to TOP 08-2-061B and TOP-08-2-060A.

Topic	Definitions from Previous TOPs	New
Shim Panel	N/A	Addition of panel type
Vertical Surface Panel (Flat)	N/A	Addition of panel type
Contamination Specification	Based on contamination density (g/m^2)	Per drop normalization to enable performance analysis as a function of drop size and density and enables calculations for variable contamination of asset. Contamination density is used in calculations, but not as a test parameter.
Decontamination Treatment Procedures	Invoked template for test execution, limited guidance for addressing objectives as a function of technology type	Mostly guidance-based test designs with a focus on technology-specific considerations using an overarching test guide or strategy
Pre-Study Activities	Low detail	Higher detail
Output Types	N/A	Describe the types of output data and analysis options
Maximum Potential Dose	N/A	Best practice calculation guidance provides the ability to use RA data to produce an upper bound estimate for vapor and contact exposures

The updates to the methodology include changes to treatment options and sampling, however, there are fundamental (revolutionary rather than evolutionary) changes in data interpretation and calculations (Section 6). While the primary focus of the methodology update is the addition of a shim panel to enable the characterization of entrained agent, contamination specifications for testing have been updated to use a new per drop normalization approach rather than a surface-averaged starting challenge (i.e., contamination density). This change in definition of contamination provides significant simplification to calculations and enables an easier translation of laboratory results to operational contexts, including variable operational contamination conditions. Contamination density is now accounted for in the scale-

up calculations, rather than a test condition applied to a panel. The updates to data interpretation and calculations are included as components of this report.

1.1.2 Shim Panels

The ability of a decontaminant to remove agent is highly dependent on the agent distribution and the associated accessibility of the decontaminant treatment to the agent. The fundamental processes associated with hazard mitigation include fluid flow (of agent and decontaminant), mass transport (e.g., sorption, evaporation), and chemical reactions (e.g., between agent and material as well as agent and decontaminant). Additionally, how liquid agents interact with different surfaces may influence RA, and these interactions can be strongly driven by agent physical properties such as viscosity (e.g., thickener[1]) and surface tension (e.g., chemical composition of the agent liquid),[2] intermolecular forces at liquid-solid interfaces (e.g. surface adhesion), and decontaminant flow characteristics (e.g., vapors, liquids, slurries). All of these interactions occur on flat panels. Introducing complex features with gaps or capillary features can have significant influence on the agent distribution and the ability of a decontaminant to access and remove the agent entrained in these features.

A study investigating the effect of liquid interactions with gaps and interfaces between materials on hazard mitigation technology performance identified that hazard mitigation techniques may perform very differently (i.e., orders of magnitude lower) compared to flat panels.[3] Essentially, the entrained agent may be very difficult to decontaminate due to flow restrictions associated with confined geometries (e.g., seams, threads, gaps, holes).[3-5] When flat and shim panel data is used to represent the agent sources from a full-scale asset, it was identified that post-decontamination, entrained agent may disproportionally contribute to the potential exposure to unprotected personnel relative to agent released from flat horizontal surfaces. For example, if only 1% of the drops contaminating an asset interact with these constrained geometry features, they may be responsible for 74% of the vapor emitted from the decontaminated asset (based on volatile simulant data).[3] This entrained contaminant may be a significant agent source that has not being characterized or accounted for in evaluation of hazard mitigation technologies using only flat panels. While multiple feature geometries were investigated in previous work, it was determined that a single panel type, i.e., the shim panel (Figure 1), could be used to generally characterize decontamination efficacy for agent entrained in confined geometries. The shim spacer establishes an air gap between two materials, here shim thicknesses of 2 and 20 mil[‡‡] are used. Shim panel testing utilizes *the same test infrastructure as flat panels,* the introduction of shim panels is similar to introducing a new material into testing. An overview of shim panel concepts implemented in the methodology is provided in Appendix A.

‡‡ 1 mil is equivalent to 0.001 inch (25.4 µm), 2 mil is 0.002 inch (50.8 µm), 20 mil is 0.020 inch (508 µm).

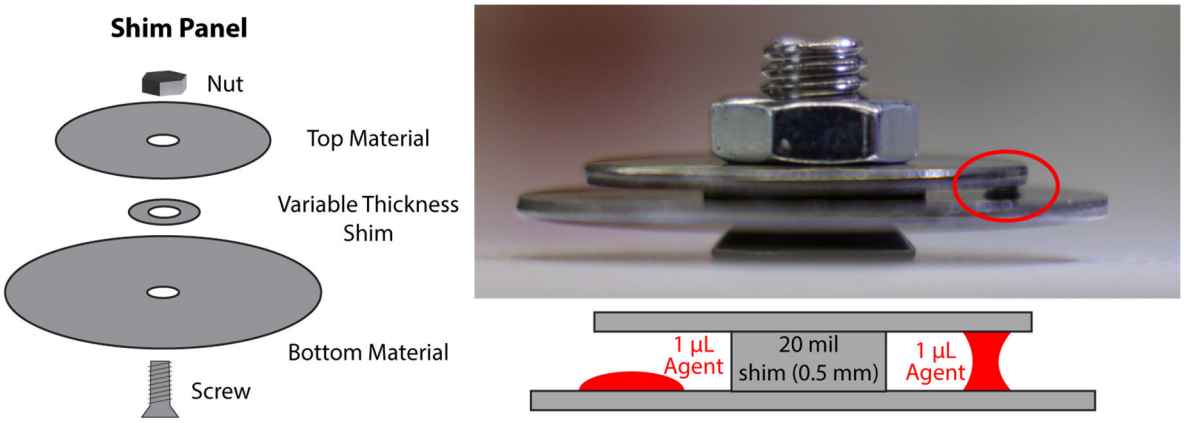


Figure 1. Shim panel assembly diagram (left) and image of a steel shim panel with 1 µL of liquid contaminant (right), drops may or may not bridge the top and bottom materials.

1.2 Methodology Assessment Criteria

The laboratory methodology generates RA analysis outputs as the extracted mass from a panel after a treatment process. Multiple steps are involved with generating an agent distribution (i.e., contaminating the panel), performing the treatment process, and extracting and analyzing the RA mass. These components are isolated, as part of verification and validation, to demonstrate that each step is being conducted reproducibly. For example, it is demonstrated that the correct mass of agent is delivered with accuracy and precision, it is demonstrated that agent is sufficiently extracted from panels, and that the chromatography reproducibly quantifies the RA mass. These demonstrations establish that the materials are reproducibly contaminated and analyzed to enable the characterization of the affect (i.e., the RA mean and standard deviation) of the treatment process. Because these steps include all processing except for the hazard mitigation technology treatment process, it is inferred that variability in RA data greater than observed in the isolated control tests can be attributed to variability of the treatment process for a given agent-material-feature combination.

1.2.1 Verification Overview

The scope of the method verification and validation covers multiple topics as outlined in Table 2–3. The verification demonstrates the ability to apply and recover contamination from these panel types without a decontamination treatment process. Next, sensitivity and demonstration data sets are generated to characterize factors that may influence efficacy, e.g., panel orientation. Results are then analyzed to determine if variable orientation should be included in technology evaluations.

Table 2. Method verification components

Component	Purpose	Conditions	Agents (drop volume)	Acceptance Criteria	Report Section
Positive Control Extraction Recovery of Shim Panel Assembly (Goal 1.1)	Verify agent extraction efficiency	**Materials**: Steel, PU coating, Alkyd coating, steel-silicone (shim) **Features**: flat panel, 0.002 inch shim panel **Contamination Duration**: 15 min	HD, VX 0.2 µL	≥ 80% extraction efficiency	3
Application of Contaminant to Shim Panels (Goal 1)	Verify reproducibility and accuracy for agent application and recovery	**Contamination Tools:** Syringe, M10, Repeater **Materials**: Steel, PU coating **Features**: flat panel, 0.002 inch shim panel, 0.020 inch shim panel **Orientation**: horizontal, vertical **Contamination Duration**: 1 min	HD, VX 0.2, 1, 4 µL	Mean value is within ± 20% of the DCS. CV for each sample will be reported	4

DCS is a dose confirmation sample, CV = coefficient of variation (std dev / mean)

Table 3. Sensitivity data demonstration

Component	Purpose	Conditions	Agents (drop volume)	Acceptance Criteria	Report Section
Shim Panel Decontaminant Application Process and Panel Orientation (Goal 1.2, 1.3)	Sensitivity analysis to determine if horizontal panel data can provide 'worst case' estimates for vertical panels and how much liquid application technique may influence results	**Materials**: Steel, PU Coating, Alkyd coating **Features**: Flat Panel, 0.002 inch, 0.020 inch **Orientation**: horizontal, vertical	HD, VX 1 µL	These data are provided for future test planning guidance on use of different orientations. CV for each condition will be reported.	5

Not all agents are included for each test design because previous related method validation efforts have characterized the response of many agent-material combinations.[6,7] Each agent may have specific agent-material interactions (e.g., sorption, spreading) and specific chemical reactions with the decontaminant, however, the methodology enables the characterization of these factors regardless of the agent. The observed reproducibility may vary with agent-material combination. As a result, most testing in this effort focuses on one agent, HD, a moderate volatility agent, to represent how other liquid agents behave. However, since VX has demonstrated different adhesion to certain material surfaces relative to HD,[5] it has been included in select tests.

Each test design uses either constant (e.g., only 1 µL drops tested) or variable drop volumes (e.g., 0.2–4 µL drops tested). For each panel, the response is specific to the drop volume applied; if multiple drops are applied to the same panel, they are all of the same volume. For most sensitivity tests, only one drop volume is investigated to understand how different variables or factors influence the measured result. Variable drop volumes were tested when the data is intended to be used for asset-level calculations or if the ability to deliver specific drop volumes was being verified.

1.2.2 Validation Overview

The validation of the methodology has two primary aspects. First, is the demonstration of the reproducibility of the methodology to generate the RA source terms (i.e., data elements), documented in Sections 7 and 8. Second, is the assessment of the scale-up calculation accuracy using laboratory data to represent the total mass retained by an asset (Section 11). Two types of small assets were contaminated, treated, and extracted to provide an M_{Asset} result. In this case, the *contamination was specifically and carefully controlled* to enable the assessment of calculation accuracy between the tested assets and the laboratory scale-up result. These validation aspects were evaluated using several conditions outlined in Table 4, including liquid (soapy water) and vaporous (hot-air) decontamination treatments. It was hypothesized that the vaporous technology would be more reproducible and less sensitive to the flow restrictions created by the constrained geometry of the shim panels. Conversely, efficacy and reproducibility for a liquid treatment may be significantly influenced by the flow restrictions of the shim panels.

The validation plan was developed to evaluate variable contamination configurations (i.e., application of different agent drop volumes). The scale-up calculation requires RA as a function of agent drop volume as an input. Three approaches to process the RA data were evaluated for their accuracy to predict asset level responses. Each approach requires different quantities of test data. The methodology assessment evaluated all configurations to characterize the accuracy of the asset level output in each case. Details, rationale, and context on these evaluations and criteria are provided in Sections 6, 7, and 11.

Treatment processes may be performed reproducibly (i.e., the same procedure is used the same way), however the affect of the treatment process on RA mass may range from reproducible to highly variable. The purpose of the methodology is to *characterize the magnitude and variability (i.e., mean and standard deviation) of the RA mass after a treatment process is conducted as prescribed by the technology developer (or test specification)*. The variability observed in the RA analysis output is a function of the full treatment process and is significantly influenced by agent-material interactions, the agent distribution when the treatment process is performed (e.g., agent liquid on the surface, agent absorbed into the material, or in the shim gap), and the characteristics of the treatment process (e.g., technology application and mechanisms of agent removal). Some agent-material-features may produce minimal variability while others may be highly variable for a given treatment procedure. In the context of method validation (i.e., establishing method reproducibility), consider that the variability of the RA data after a treatment process is a *characteristic of the treatment process* for a given agent-material-feature panel. The variability of the RA data is an output that could be optimized (i.e., reduced) by altering technology formulations or application processes.

Several of the assessment criteria utilize CV as a measure of variability. Through this effort it was realized that the standard deviation of the log-transformed results provides a better description of relative variability for data generated by this methodology, as described in Appendix B. The reproducibility of RA data after a treatment process demonstrated in this report are specific to the technologies used and their application processes.

Table 4. Method validation components.

Component	Purpose	Materials/Treatments	Agents (drop volume)	Acceptance Criteria	Report Section
Reproducibility Testing (Goal 1.3, 1.4, 2, and 3)	Quantify the data variations resulting from the same condition repeated within the same test day with all precision factors held constant (i.e., within test day variability assessment for a single laboratory).	**Materials**: Steel, PU coating, Alkyd coating, Silicone **Features**: Flat Panel, 0.002 inch, 0.020 inch **Treatments**: 1) Soapy water spray followed by water rinse (30 min residence) 2) CHAD treatment (2 h at 76 °C, 95% RH)	HD 1 ×0.2, 0.6, 1, 2, 4 µL	Data supports accuracy assessment, report CV per condition. **Reproducibility goal**: relative within day variation < 60% CV	7
RA IPSD Testing (Goal 1.4)	Evaluate the IPSD (i.e., similarity of values over multiple days using multiple calibration curves) of the treatment process on flat panels and shim panels.	**Materials**: Steel, Alkyd coating **Features**: Flat Panel, 0.002, 0.020 inch **Treatments**: 1) soapy water spray followed by water rinse (30 min residence) 2) CHAD treatment (2 h at 76 °C, 95% RH)	HD 1 ×1 µL	IPSD reported, goal of < 60% CV.	8
Mixed-material Shim Panels (Goal 1.2)	Verify effect of mixed materials on decontamination effectiveness vs. single material shim panels.	**Materials**: Steel-silicone, Steel-PU coating, PU Coating-silicone, Alkyd coating-silicone **Features**: 0.002, 0.020 inch	HD 1 ×1 µL	Data is used for sensitivity analysis: If the $\log_{10}$ difference* (LD) for similar material to mixed-material condition is statistically significant and the mean LD is < 0.4, then the single material case can be used to represent mixed-material interfaces.	9
Asset Testing (Goal 3)	Generate asset level RA data.	**Assets**: Multi-tool, Picatinny rail accessory **Treatments**: 1) soapy water spray followed by water rinse (30 min residence) 2) CHAD treatment (2 h at 76 °C, 95% RH)	HD 4 × 1 µL, 5 × 0.2 µL	Data supports accuracy assessment, report CV per condition.	10
Asset-level output calculation accuracy (Goal 2, 3)	Perform asset level calculation and assess its accuracy using laboratory panel data to represent an asset with the RA-based asset level outputs.	Asset level output will be conducted using the three contamination configurations	N/A	**Calculation Accuracy** Ratio of predicted / measured: Acceptance within factor of 10–0.1 Confidence intervals will be provided for predicted and measured conditions.	11

* $\log_{10}$ difference is defined in Appendix B

1.3 Laboratory Scale-Up Capability and Health Effect Outputs

The context of the end data use influences the concepts used in an analysis which shapes the types of data required to conduct the analysis. Starting with this end point in mind influenced the approach to how the RA data elements are measured and the subsequent calculations.

1.3.1 Mapping Laboratory Data to Operational Context

The data produced by this test methodology is intended to represent what would occur on full-scale assets in an operational environment. Contamination in the operational environment will be highly variable. Agent may randomly splash parts of an asset resulting in different materials being contaminated with a range of agent drop volumes (Figure 2). The initial shim panel development demonstrated that some material features retain 1–10,000× more agent than other material features. Therefore, it is important to correctly identify which features are contaminated. Next, the agent drop volumes vary significantly based on multiple operational factors, which also influence the quantity and distribution of agent associated with asset materials that may present potential exposure sources to personnel. No contamination event will occur exactly the same way (e.g., the same 'splash' pattern on an asset). Asset level results produced by the methodology need to facilitate handling randomized contamination to produce outputs that can be used to describe the range of conditions personnel may encounter in the operational environment. The approach to handle these considerations is presented in Section 6.

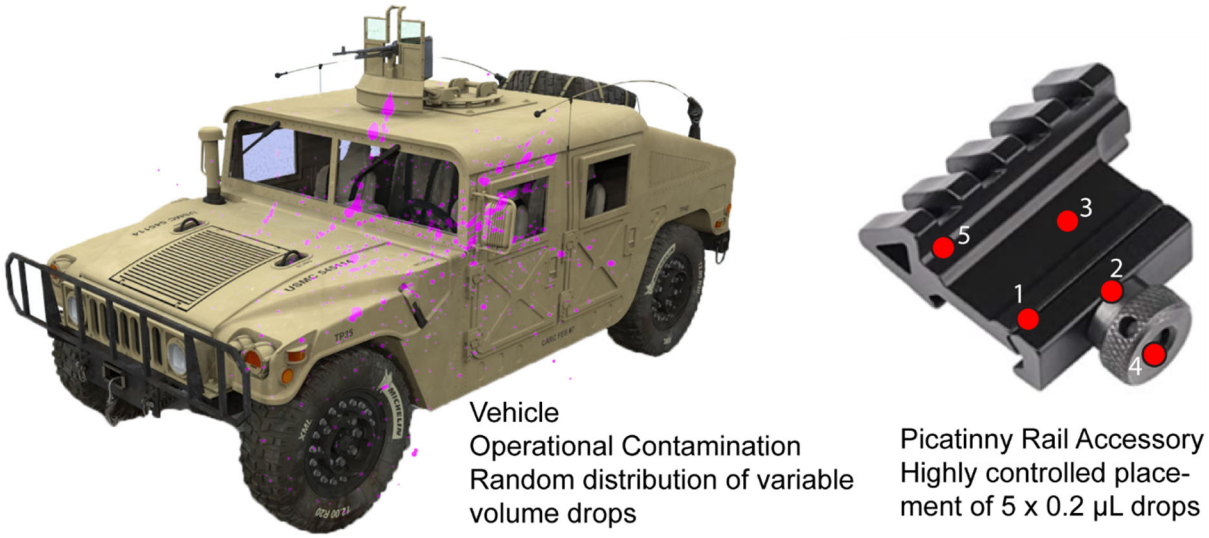


Figure 2. Conceptual contaminated vehicle asset, pink indicates variable volume agent drops contaminating various parts of the vehicle (left). Controlled contamination of a small asset used to demonstrate the methodology (right), numbered red dots indicate the location of contamination.

This methodology assessment demonstrates that a per drop aggregation approach enables the scale-up of laboratory data to represent an asset. This is shown for a highly controlled contamination of two assets (including the Picatinny rail accessory illustrated in Figure 2). It is assumed that if asset RA resulting from a highly controlled contamination condition can be predicted with accuracy, the same process can be used for the random contamination of other assets. The methodology focuses only on the scale-up calculation where

the drops and drop volume and contaminated material-feature *are provided*. The details and procedures of how to produce random contamination of an asset (i.e., how to identify the drop volumes and contaminated material-features for random contamination) are independent from the scale-up calculation. For accurate outputs, the materials of construction and the geometry of the asset are needed. Additionally, there is a need to understand how the asset was contaminated. A concept of how randomized contamination could be performed is provided in Appendix C. The methodology can produce an output when the contamination details of the asset are provided.

To enable the scale-up calculation, the primary data element is the RA mass per drop (source term). The objective of the RA analysis method is to generate an appropriate agent distribution on a material (flat or shim panel), perform a treatment process, and measure the RA mass per agent drop (demonstrated in Section 7). This RA mass indicates the total agent mass associated with one agent drop interacting with a material that could produce an exposure to unprotected personnel. This source term is used in a scale-up calculation to represent the total RA mass associated with the asset (M_{Asset}). The M_{Asset} calculation output (Output B_0 of the methodology, Appendix A Figure A-1) can be used to produce an upper bound estimate for health-based outputs.

1.3.2 Maximum Potential Dose

A new output capability in the methodology involves the use of the total agent associated with an asset, M_{Asset}, to approximate a maximum potential dose (MPD) for vapor and contact exposures. In this case the total RA associated with an asset is assumed to produce a vapor or contact exposure. This is an upper bound estimate for the maximum dose that could be observed for personnel interacting with the asset. Multiple factors such as chemical volatility and chemical distribution contribute to how much the MPD may overestimate the actual vapor or contact exposure. Collecting the VE and CT data to enable full exposure assessments is significantly more complicated and costly compared to RA data and MPD dose estimates. If this MPD output is below the relevant health effect requirement, then any detailed exposure assessment associated with this asset would be unnecessary. If the MPD is above the health-based requirement, there is a chance that a full VE or CT evaluation could also exceed the health-based requirement.

RA outputs have typically been used for laboratory-based analysis only. It was previously not clear how a total mass measurement could be translated for use in vapor or contact exposures. As part of other decontamination process studies, it was shown that RA reflects the total agent mass that is available for personnel to experience a dose from a contaminated material, regardless of the exposure route, including vapor emission or contact transfer.[8] In this methodology, we use this principle of RA as an upper bound (i.e., worst case) estimate of available agent mass in combination with a revised scale-up calculation of RA response from panels to assets. It is then assumed that all mass associated with the asset is emitted as a vapor over a specified duration (i.e., steady state vapor emission) or that that all mass produces contact transfer to support evaluations of hazard mitigation technologies to health-based requirements. For most cases, only a fraction of the total mass will result in an exposure dose, but in the case that all mass is transferred via inhalation or contact, the MPD provides the estimate for this worst-case situation.

The volatility of the agent may influence the relationship of an upper bound MPD estimate compared to an actual VE or CT exposure. For example, high volatility agents like GD may actually emit all retained agent as a vapor, MPD would provide a more accurate vapor exposure assessment. Whereas for a low volatility agent only a small fraction of the retained agent may be emitted as a vapor, making the MPD a potentially significant overestimate for vapor exposure. For contact exposure, high volatility agents may result in a significant quantity of vapor emission which would decrease the mass available for CT. In this case, CT exposure assessments for high volatility agents using MPD may significantly overestimate the actual CT exposure. For low volatility agents this evaporative loss would be small and the MPD may be closer to the actual CT exposure dose. In all cases the MPD will provide an upper bound estimate, but various factors influence the difference between the upper bound potential and actual exposure.

It is estimated that the RA and MPD dose approach requires about *20 times less resources* (cost and time) compared to VE and CT testing and analysis. The use of MPD analysis enables programs to investigate many more conditions of interest (e.g., different contamination times, pre-treatments, etc.) to optimize technology implementation or identify and prioritize conditions for the more costly VE and CT testing. A demonstration of MPD calculations for vapor and contact exposure is conducted and compared to toxicity values to indicate upper bound estimates of negative health effects (Section 12).

1.3.3 Asset Testing Limitations versus Panel Testing Capabilities

Directly testing a hazard mitigation technology on a full-scale asset is nontrivial for logistical (e.g., requires access to full-scale assets for testing and ability to dispose of them afterwards) and technical reasons (e.g., requires means of physically contaminating, treating, and sampling an asset and appropriately interpreting the collected data). The results of hazard mitigation testing are a function of agent distribution when a treatment process is applied. Therefore, when a full-scale asset is tested, the results specifically apply only to the *exact* conditions tested (e.g., exact agent drop volumes, drop count, and placement, for the exact treatment timing and process of the specific asset tested). In the context of assets, the scale of vehicles, contamination to 1 g/m^2 starting challenge could require on the order of 20,000 agent drops. This specificity of the conditions makes it challenging to utilize the asset test results to indicate what would occur in different conditions (e.g., different contamination distribution or density, different timing, different asset). As a result, passing or failing a health-based requirement for the tested condition does *not* indicate that the technology would also pass or fail under different conditions. The logistical challenges also limit the number of tests that can be conducted to provide statistical confidence in the measured data. However, there is significant utility in testing full-scale assets to verify the accuracy of laboratory scale-up calculations used for extrapolation to many different conditions (i.e., the laboratory data is more versatile and extensible).

Panel testing and scaling of the resulting data enables the calculation of health-based outputs for many different conditions (see Appendix C.5) and is the most practical approach to evaluate hazard mitigation technologies with operational context. Representative materials are identified and tested in the form of panels to characterize how the hazard mitigation technology alters the quantity of agent associated with the relevant materials of the asset(s), now on a per drop basis. Using the premise that "the whole equals the sum of the parts," assets can be represented from panel data by aggregating the per drop contamination that would occur on an

asset. The total agent retained by an asset can be determined in this fashion, or exposure assessments for vapor or contact transfer can also be generated similarly by collecting and summing material-level source terms. Panel data can be acquired with high throughput and robust statistical replication to enable the approximation of what could occur for a range of assets comprised of the tested materials under various test conditions. This methodology assessment demonstrates the accuracy associated with representing RA response and MPD for an asset with panel RA data.

1.3.4 Inputs and Data Elements to Produce Health-Based Outputs

The health-based output is derived from a combination of inputs regarding the operational vignette and the data elements generated from the test methodology. Figure 3 captures the high-level data and processes required to translate laboratory data to a health-based assessment. The bulk of this report focuses on the data elements and source terms associated with the RA test methodology. However, asset-level calculations (detailed in Section 6) require the specification of the asset of interest and a contamination vignette. Health-based outputs require the specification of contact and vapor exposure vignettes that describe how personnel interact with the assets. A demonstration of asset description and contamination is provided in Section 12, and MPD for vapor and contact exposure is provided in Section 13. The number of details associated with the contamination vignettes and exposure vignettes can become overwhelming, the approach of MPD was to simplify the calculation and provide upper bound estimates. A summary of the parameters and their influence on the outputs are provided in Table 5.

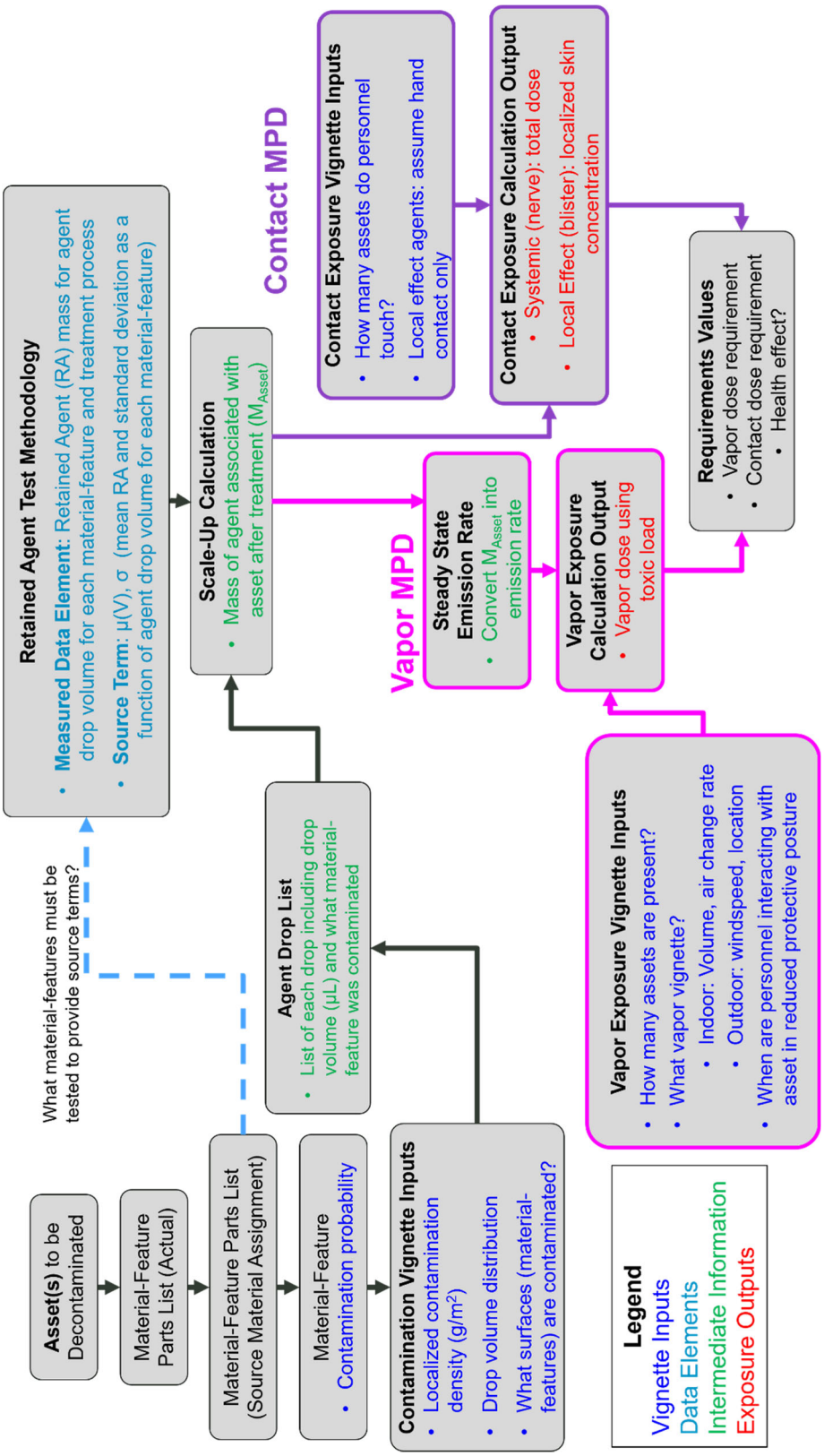


Figure 3. High-level flow chart illustrating relationship of vignette inputs, data elements, and outputs.

Table 5. Factors influencing health-based outputs.

Component	Description	Uncertainty Source(s)
Measured data element	Experimental variations	Experimental Uncertainty: Variability of material, variability of treatment process, accuracy and precision of contamination and chromatography measurement
Agent Distribution	Any factor that alters the agent distribution may influence decontaminant efficacy and post-treatment RA or source terms	Process variations such as differences in timing, differences in decontaminant application techniques, or the timing and sequence of treatments if multiple technologies or treatments are used could alter RA or source terms
Source Term	Relationship of data element to source term function, $\mu(V)$	Fitting error
Material Representation	Ability of panels to represent actual asset materials	Ability of tested material to represent actual materials of construction • Ability of shim panel to represent all constrained geometry features (e.g., gaps, seams, weather stripping, corners, threads) • Differences in orientations (from purely horizontal or purely vertical) • Material substitutions (one material is used to represent a class of similar material responses) • Differences across material manufacturers or production lots
Asset: contamination probability	Ability to accurately determine the probability a given material-feature may be contaminated	Resolution of asset parts, materials of construction and features. Higher spatial resolution may not produce actual improvement in calculation of contamination probability accuracy.
Asset: surface contamination	Ability to accurately determine what regions of an asset may be contaminated for a given contamination vignette	Vignette details are non-trivial, distance from agent source could influence drop volume distributions and what asset surfaces are actually contaminated
Vapor exposure vignette	Ability to describe the environment to enable calculation of environmental vapor concentration produced by asset vapor emission	The environment into which an asset emits determines the resulting vapor concentration, the accurate description of room volume, air flow, wind direction, and proximity to the source will influence results
Contact exposure vignette	Ability to accurately describe how personnel touch the asset	Variations in the handling of one or more assets could influence calculated versus 'actual' results

1.4 Capabilities, Assumptions, and Limitations

The capabilities of the methodology in this report address conducting hazard mitigation treatment processes for flat and shim panels that have been contaminated with liquid chemical warfare agent. The methodology assessment focused on the use of RA analysis method total mass characterizations of panels after a treatment process. Procedures to use the RA data as source terms in a scale-up calculation are provided. A maximum potential dose concept is used to quantify worst case potential vapor and contact transfer exposures dosages to enable health-based assessments using the asset-level outputs. The majority of the test infrastructure for flat panel and shim panel materials is the same, some additional hardware is used to hold shim panels during the treatment procedures (see Appendix A).

The primary assumptions used in the methodology and calculations are summarized here, with details and context provided in the appropriate report sections. It has become recognized that the agent distribution when the treatment process is applied is a significant factor towards efficacy. A fundamental assumption of this work is that the contamination process is generating representative and reproducible agent distributions on the materials of interest. The characterization of the *quantity* of agent delivered is quantified as part of the methodology, but characterization of the agent *distribution* is non-trivial and not a part of the methodology. Further, for shim panel configurations the agent distribution is obscured by opaque materials. The following enumerates additional assumptions in the measurement and calculation procedures.

For shim panels it is assumed that:

- Entrained agent effects are influenced by feature size, rather than specific feature geometry (e.g., thread, gap, material interface).
- Contaminant delivered to a shim panel is entrained in the shim panel feature and the contaminant distribution (e.g., as a function of liquid spreading) in the shim panel is sufficiently representative of what would be observed from operational contamination.
- Two shim thicknesses (suggested defaults of 0.002 inch and 0.020 inch) can be used to represent small and moderate size entrained geometries (see Appendix A).[3]
- The shim gap thickness is assumed to provide the primary flow restrictions that influence decontaminant performance. The shim panel feature depth (Appendix A, Figure A-6) is not varied (0.187 inch for the selected parts) and is an acceptable compromise between deeper or shallower features on assets.
- Shim panel results represent per drop agent interactions with any complex feature on an asset.

For asset level outputs it is assumed that:

- The response of an asset can be represented using the aggregate response of individual agent drops on representative materials and features (i.e., aggregation of per drop normalized data is appropriate for flat and complex features).
- Agent characteristics such as mass purity, viscosity, and liquid surface tension used in testing is representative of the agent that would be encountered in operational context.[1,2,9,10]
- Decontamination efficacy is not a function of contamination density, but rather agent drop volume.[11]
- Decontaminants are applied in an overwhelming quantity (i.e., there are no limiting reagent effects).[11]
- The RA mean and standard deviation (μ and σ) parameters used as inputs in the drop aggregation calculation are sufficiently accurate descriptions of

the data distributions (i.e., data sample sizes provide sufficiently accurate statistical descriptions).

Once asset level responses are determined, the MPD interpretation assumes:

- All asset RA will result in an exposure dose (either vapor or contact), which may be a significant overestimate.

2. MATERIALS AND PROCESSES FOR PANEL TESTING

The following section documents general materials and hardware associated with testing flat and shim panels that were used in developmental testing and in verification and validation testing. Figure 4 illustrates the general treatment process for the RA analysis method.

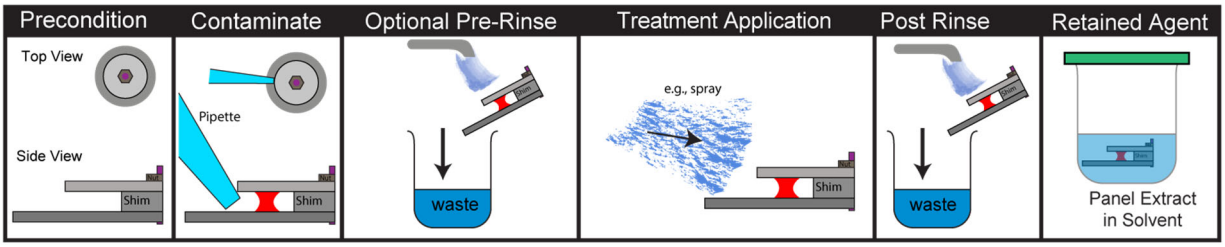


Figure 4. Treatment process for all panels, shim panel used in illustration.

2.1 Materials

Multiple materials were tested in the study to capture different types of behaviors resulting from deposition of liquid agent drops including whether liquid agents spread and absorb into the materials. Specific materials included steel, silicone elastomer, as well as two paints, a green polyurethane (PU) coating where liquid agents tend to spread and a grey Alkyd coating where liquid agents tend to not spread, i.e., drops remain sessile. These materials were chosen since they are commonly used in decontamination evaluations, previous work has shown how these materials retain agent, and the spreading behavior of the agents is known to be different across these materials.[12] The effects of agent flow into the shim panel gap are anticipated to be influenced by the wetting characteristics of the material, the selected samples span cases of significant spreading to non-spreading.[3-5] Table 6 provides information regarding the test materials used. The painted washer materials were painted on one side, shim panel assembly arranged the washers so that the painted sides face each other (Figure 5). Details of shim panel assembly are provided in Appendix A.

Table 6. Panel material information.

Material	Description	Geometry
Steel	316 Steel	0.75 inch top washer 0.047 inch thick 1.25 inch bottom washer 0.057 inch thick flat panels may use bottom washers or 2 inch flat disks
Alkyd coating[1]	Grey Alkyd coating on zinc plated steel	0.75 inch top washer 0.042–0.051 inch thick 1.25 inch bottom washer 0.042–0.051 inch thick flat panels may use bottom washers or 2 inch flat disks
Polyurethane (PU) Coating[1]	Green PU coating on zinc plated steel	0.75 inch top washer 0.042–0.051 inch thick 1.25 inch bottom washer 0.042–0.051 inch thick flat panels may use bottom washers or 2 inch flat disks
Silicone	Silicone Elastomer MQ/VMQ/PMQ/ PVMQ, part number SI303300, Goodfellow	2 inch disk with hole punched for shim panel screw. Only used as bottom washer supported by steel washer due to flexibility of material.
Note 1: After application to washers, coatings were cured at ambient conditions for 7 days. The PU coatings were subsequently cured for 7 days at 122 °F to provide an accelerated cure that resembles coatings with a 3 month ambient cure.[13] The Alkyd samples were subsequently cured for 30 days at 122 °F.		

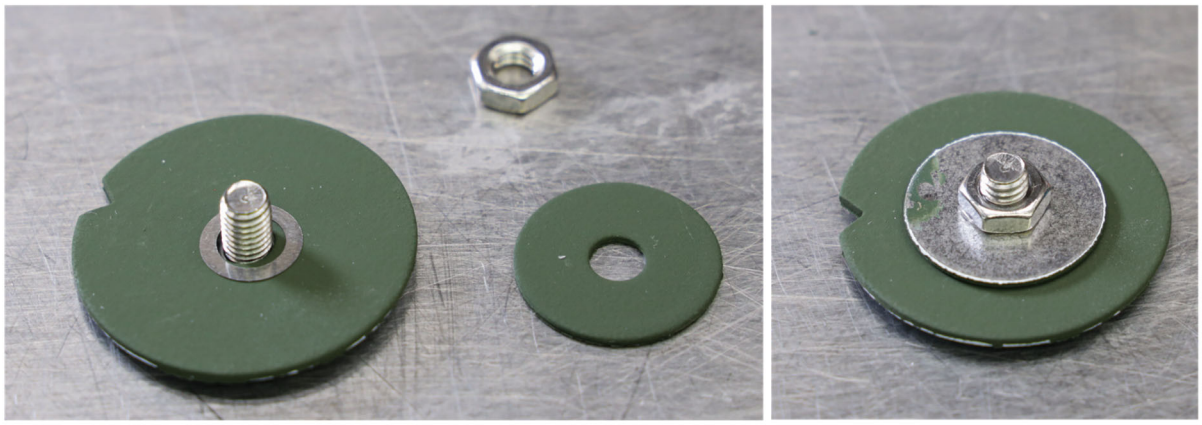


Figure 5. Painted shim panel parts (left) and assembled (right) for coated materials.

2.2 Contamination

Multiple contaminants were used as part of this study to evaluate moderate and low volatility agents as listed in Table 7. All contaminants used, including HD, VX, and methyl salicylate (MeS), were dispensed as liquids at room temperature (RT) to test materials and had >96% mass purity. The simulant MeS was used for pre-validation testing in select conditions. The effects of agent mass purity and other agent attributes on agent retention and decontaminant performance are documented elsewhere.[1,2,9,10]

Table 7. Liquid density at room temperature and typical purity for the different contaminants used.

Agent	RT Liquid Density (g/mL)	Agent Mass Purity (%)
HD	1.268	99.8
VX	1.008	96.9
MeS	1.17	99.8

Contaminant was delivered to the panels using multiple devices as indicated in Table 8. The methodology does not require a specific tool to be used, only that the correct mass is applied via the desired drop volume, as confirmed with dose confirmation samples. Each liquid dispensing tool has advantages and limitations associated with delivering the liquid agent. To facilitate accurate drop volumes using syringes a Masterflex Touch-Screen Syringe Pump (division of Cole Parmer, Vernon Hills, IL) with programable infusion/withdrawal was used. For optimal accuracy across the range of drop volumes, a smaller diameter 10 µL syringe was used for drops less than 1 µL, and a 50 µL syringe was used for drops 1 µL or larger. Note, after the positive control tests (presented in later sections), all testing used the syringe pump to deliver the contaminants.

Table 8. Liquid dispensing tools used to deliver agent.

Description	Drop Volume Range	Notes
Gilson Microman Positive Displacement Pipette (M10E)	1-10 µL, continuous adjustment	Positive displacement, drops are touched off from plastic tip, each drop application requires reloading the pipette from the agent container
Gilson Pipetman air piston pipette (P2)	0.2-2 µL, continuous adjustment	Air piston pipettes are designed to deliver aqueous solutions based on assumptions about viscosity and volatility, air piston pipettes are less ideal for agent delivery but the only way to deliver drop volumes < 1 µL with a pipette.
Syringe pump with Hamilton 10 µL gas-tight syringe (part 1701 N)	0.1-2 µL, continuous adjustment	Positive displacement, drops are touched off from steel tip, multiple drops can be delivered before reloading of agent from agent container. The 10 µL syringe used a cemented 26s gauge needle (0.47 mm outer diameter), the 50 µL syringe used a cemented 22s gauge needle (0.72 mm outer diameter).
Syringe pump with Hamilton 50 µL gas-tight syringe (part 1705 N)	1-50 µL, continuous adjustment	

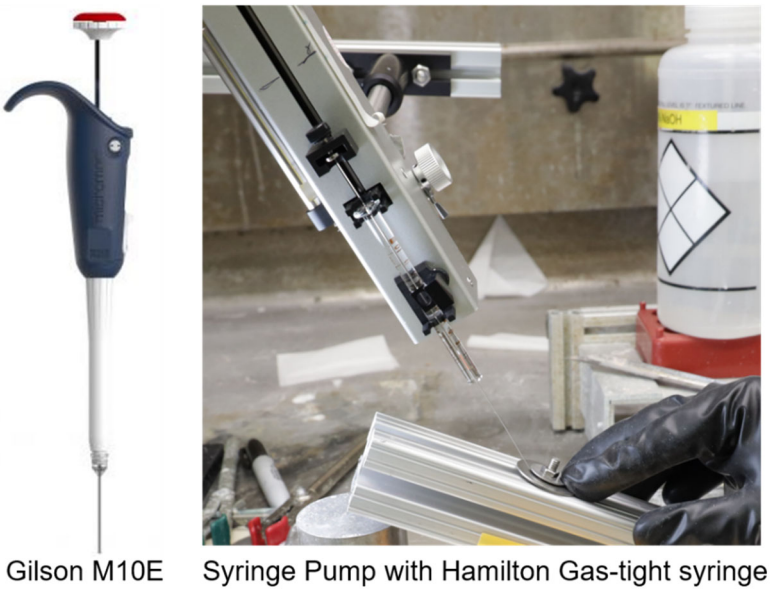


Figure 6. Photographs of the Gilson pipette (left) and Masterflex syringe pump (right) contaminant delivery tools.

The syringe pump uses a touch-sensitive liquid crystal display screen to trigger the syringe pump delivery. It was realized that pressing the button, especially with gloved fingers, did not always trigger properly, and sometimes resulted in a 'double dose'. It was identified that the use of a stylus pen with conductive tip such as those used on computer tablets, provided much more reliable interactions with the instrument.

2.3 Decontamination Treatments

The methodology can be used to evaluate any hazard mitigation technology. Two decontamination types are used in the verification and validation effort including liquid and vaporous technologies (Table 9). Each report section describes the details of the decontamination treatment.

Table 9. Decontamination treatment information.

Decontaminant	Applicator	Description
Soapy water	Preval sprayer	Example of a liquid decontaminant based on the CARM methodology[8] that uses General Purpose Detergent: Liquid Nonionic, Water Soluble, Type 1; NSN 7930-00-282-9699. The soapy water is prepared by mixing 552 µL of the soap solution into 1 L of DI water.
CHAD	Vapor chamber	Vaporous treatment with temperatures of 60-80 ºC and high relative humidity ~95% RH

The Chemical Hot Air Decontamination (CHAD) technique, which uses elevated temperature in combination with adjustable humidity over a specific time duration, serves as an example vaporous decontamination treatment. The DEVCOM CBC lab generates these conditions using a Thermotron (Chicago, IL) S-1.0-8200 Benchtop Environmental Chamber which is a 0.89 ft^3 (25 L) chamber (Figure 7) that can maintain set point temperature and humidity conditions; performance specifications are listed in Table 10. The chamber is supplied

with a fresh air flow rate of 1.6 L/min resulting in an air change rate of 2 hr^{-1}. The details of the conditions used in testing are provided with each test design.

Figure 7. Image of the Thermotron chamber used for the CHAD treatment.

Table 10. Performance specifications for the CHAD chamber.

Factor	Range
Temperature range	-40°F to +266°F (-40°C to +130°C)
Humidity range	20% RH to 95% RH Limited by a +59°F (+15°C) minimum dew point and a maximum dry bulb of +185°F (+85°C)
Heating performance	77°F to 150°F (25°C to 65°C) 6 minutes
Temperature control tolerance	±2°F (± 1.1°C) Dry bulb temperatures from control point after stabilization at the control sensor
Humidity control tolerance	± 2.5% RH at a dry bulb temperature above 68°F (20°C)

Soapy water is a common decontaminant, the surfactant selected here is the soap specified in the decontamination field manual FM 3-11.5.[14] The soapy water liquid decontaminant, is applied using a Preval sprayer (Preval, division of Nakoma Products, LLC). The sprayer uses a Venturi effect to draw the liquid into the sprayer for application and works with a large range of liquids and paints, making it an effective, laboratory-friendly applicator for most liquid decontaminants, shown in Figure 8. This sprayer has been approved for use in DEVCOM CBC laboratory agent testing. Details of the application process are documented for each test.

Figure 8. Image of a Preval sprayer with 16 oz reservoir that generates a liquid aerosol spray.

2.4 Panel Extraction and Chromatography

All extractions were performed for 24 h in chloroform or 2-propanol (also referred to as isopropyl alcohol or IPA) for HD, or 2-propanol for VX, or as otherwise indicated in the test conditions (Table 11). Extraction solution samples were diluted and the analytes quantified using gas chromatography (GC) with a mass spectrometer for HD, (Agilent 6890/7890 GC equipped with an Agilent 5975 mass selective detector (MSD) [Agilent Technologies; Santa Clara, CA] or an Agilent 7890 GC equipped with an Agilent Model 7000 MS Triple Quad [Agilent Technologies; Santa Clara, CA]) or liquid chromatography (LC) with a mass spectrometer for VX (Agilent 1200/1290 series LC and Applied Biosystems [Carlsbad, CA] API5000/5500 triple-quadrupole mass spectrometer equipped with a TurboV ion source). All chromatography samples include an agent specific internal standard, details on each chromatography method have been published elsewhere.[14]

Table 11. Chromatography overview for the analytes of interest.

Analyte	Concentration range (ng/mL)	Platform	Internal Standard	Solvent
HD	2–2000	GC-MSD	chloroethyl phenyl sulfide (CEPS)	chloroform
HD	2–2000	GC-QQQ*	$(^{13}C)_2$HF-labeled analog	2-propanol
MeS	25–2000	GC-MSD	D_4 labeled MeS	2-propanol
VX	1–748	LC-MS/MS	D_5 labeled VX	2-propanol

The HD methodology for the GC-MS Triple Quad (GC-QQQ) has not been previously published and is briefly described here. Sample introduction was performed through a Gertsel Multi-Purpose Sampler (MPS). The Gerstel system included a Cooled Injection System (CIS) with Peltier cooling. A CIS-4 baffled deactivated liner, with no packing, was utilized in the Gerstel CIS. Helium was used as a carrier gas, at an average linear velocity of 39 cm/sec, in solvent vent mode. All experiments utilized a capillary column with a bonded phase of 5% phenylmethyl polysiloxane (30 m × 0.25 mm) with a 0.25 µm film thickness. Instrumental and acquisition parameters are summarized in Table 12. ^{13}C - double labeled HD was added as an internal standard to all standards and samples for HD.

Table 12. Instrument parameters for liquid samples on the GC-QQQ

INJECTION PARAMETERS	
Mode: Solvent Vent	Injection Volume: 2.0 µL, splitless
Initial Temperature: 25 °C	Post Injection Dwell: 0.00 min
Ramp Rate: 12.00 °C/s	Vent Time: 0.30 min
Final Temperature: 260 °C	Vent Flow: 20 mL/min
Hold Time: 6.50 min	Vent Pressure: 19.5 psi
Inlet Pressure: 19.53 psi	Purge Flow: 50 mL/min at 1.00 min
Sample Pumps: 7	Viscosity Delay: 0 sec
Solvent A Washes: 3	Solvent B Washes: 3
Solvent A: 2-propanol	Solvent B: Methanol
OVEN PARAMETERS	
Initial Temperature: 70 °C	Initial Time: 0.75 min
Ramp: 35 °C/min to 120 °C, then 10 °C/min to 150 °C then 40°C/min to 280 °C for 0.00 min	
Equilibration Delay: 0.25 min	
DETECTOR PARAMETERS	
Solvent Delay: 5.3 min	Gain: 10
Scan Type: MRM	Dwell Time: 20 msec
MS Source: 230 °C	MS Quad: 150 °C
MS Transfer Line: 280 °C	Collision Energy: 15V for Quant MRM
He Quench Gas: 2.30mL/min	N2 Collision Gas: 1.5mL/min
MRMs for HD: 109>63*, 158>109	MRMs for IS: 110>64*, 160>110
*MRM used for quant	

Calibration standards were analyzed for each analytical queue. The calibration curve was established using the appropriate curve model and weighting. A calibration standard must yield the reported concentration for each level at ±20% compared to the expected concentration. If a calibration level does not meet this acceptance criterion, it was removed from the calibration curve and the curve was re-evaluated. No more than three calibration levels can be removed from within the analytical range. Every sample queue analyzed provides a Limit of Detection (LOD) and Limit of Quantitation (LOQ) as based on the queue regression.[15] Due to the large calibration range and statistical characterization of the chromatography response, weighted quadratic equations are used for the regression.[14] The LOD is defined as the critical level for which the signal is reliably attributed to the presence of analyte, whereas the LOQ is the level where the precision of the concentration satisfies an expected value. Here the LOQ used a level of 10 times the standard deviation at the lowest detectable signal (i.e., LOD).[15]

Chromatography queues include continuing calibration verification (CCV) samples, to ensure instrument control and continued confidence in the analytical results. CCV samples are run after nine test samples. If CCV-reported concentrations show a calculated difference greater than ±30% of the target value, corrective action is performed. Corrective action includes a repeat analysis of all samples in the CCV bank. For samples that are above the calibration range, the sample is diluted until the results are within the calibration range.

2.5 Dose Confirmation Samples

Previous test protocols have utilized dose confirmation samples (DCS) that quantify the amount of agent delivered to the panels on a given test session. For example, five

replicates of a 2 × 1 µL drop contamination condition would be collected with direct deposition into solvent as verification of agent purity and the ability to deliver agent for each test. While this is reasonable for a single contamination condition, collecting five DCS replicates for variable contamination conditions results in a high test burden. Therefore, as part of this study, only three replicates per drop volume were collected for variable agent drop volume tests. Previous testing has demonstrated the ability to reproducibly deliver agent using the indicated contamination tools, the DCS were used as a confirmation of accurate agent delivery and agent purity for a given test day. The lower replication provides the confirmation per day, and the results are not included in the statistical analysis of final output results.

2.6 Laboratory Temperature and Humidity

Temperature and relative humidity were monitored and recorded (Extech SD500 Humidity/Temperature Dataloggers) for the ambient laboratory and within an environmental chamber that contained the samples at all times except when they were being treated (i.e., contaminant deposition or decontamination application). While the laboratory and environmental temperatures are controlled, humidity is not. The methodology requires that 90 % of temperature measurements must be within ± 5 °C of the target temperature; there are no specifications for humidity. No temperature excursions outside of ± 5 °C were observed. The summary table describing the environmental conditions is provided in Appendix C. Based on Table C-1, all tests met the temperature criterion.

3. EXTRACTION EFFICIENCY

3.1 Test Design

For the verification of the extraction process (methodology assessment goal 1.1), a study was performed following the guidance provided in Appendix A of the methodology. A subset of configurations of materials identified in Table 6 was used with a single small drop volume to highlight cases of expected relative ease and difficulty in extraction efficiency. Panels were fully immersed in solvent during extraction, using appropriate glassware to accommodate different panel sizes. Specifically, 2 inch flat panels use an 8 oz glass jar, and the 1.25 inch shim panels can fit in a smaller 2 oz glass jar. The precleaned QC clear glass short wide mouth jars with solid PFTE lined caps were obtained from Scientific Specialties, part 370802 for 2 oz size, and part 270808 for the 8 oz size: The test conditions used for verification are provided in Table 13. The extraction method was verified by the quantification of extraction recovery. In general, the method allows for longer duration extraction or use of other solvents to be considered in the event of poor recovery. A sampling of 5 replicates is used here, 5 replicates has been the standard value used in previous testing.[16]

Table 13. Test parameters for verification of shim panel extraction.

Parameter	Conditions	Comments
Materials	Steel, PU coating, Alkyd coating, Steel-silicone (shim)	Shim panels were assembled with the same material for top and bottom washers. Shim panels used 1.25 inch bottom washers and 0.75 inch top washers. For flexible elastomer the top washer was steel.
Shim thickness	Flat panel, 0.002 in	Flat panel may be just the bottom washer. Only the more challenging 0.002 inch shim size is tested.
Agents	HD, VX	HD and VX present different chemistries and interactions with materials, both agents are selected here to demonstrate the ability to recover different contaminants.
Drop count ×volume (µL)	1 × 0.2	Applied using a syringe pump. The smaller 0.2 µL drop was found to be the more challenging to recover in previous testing (see Appendix C).
Age time	15 min	15 min was a duration long enough to enable some sorption by the material but not so long as to result in significant evaporative loss.
Extraction	Shim panels 2 oz jar; solvent volume (40 mL), duration 24 h flat panels 8 oz jar; solvent volume 20 mL, duration 1 h	Solvent: IPA for VX, chloroform for HD, shim panels extracted fully assembled.
Treatment	No treatment (positive control)	Contaminate and extract only, no treatment
Analysis Method	RA test	-
DCS	5 replicates	Used as a reference for actual mass delivered
Replication	5	-
Analysis	Quantify recovery (RA/DCS)	Verification criterion: extraction efficiency > 80%

The verification criterion for extraction efficiency, EE, is within 0.80 (i.e., 80%), or best achievable. The EE is calculated as the arithmetic mean of the RA samples divided by the arithmetic mean of the DCS.

$$EE = \frac{\text{mean}(RA)}{\text{mean}(DCS)} \quad \text{Eq. 1}$$

3.2 Results

EE testing was conducted in tests CAP016, CAP017, CAP019, and CAP034. Note that a TestID is assigned at the time a test is designed and does not necessarily indicate the sequence of test execution.

3.2.1 HD Extraction Efficiency

Extraction of 0.2 µL drops of HD was conducted after a 15 min age duration on all materials of interest. Arithmetic means are used for sample statistics for all positive control samples. In the following figures, results for flat panels correspond to cases where only a "Bottom Material" field is specified (i.e., "Top Material" is indicated as 'none') and the "Shim Thickness" is listed as 'flat'. Results for shim panels are sorted by the materials used for the bottom and top materials of the shim in addition to the thickness of the shim spacer in mil (i.e., thousandths of an inch; 2 mil is 0.002 in).

The individual RA results for HD extraction using chloroform measured with GC-MSD are presented in Figure 9. While most samples met the EE goal, the PU coating samples provided an EE < 0.7, which is below the desired recovery. Appendix A of the test methodology provides guidance on evaluating EE and approaches to improve extraction efficiency. As indicated in the test methodology, maximizing EE can be challenging and it is very difficult to differentiate between incomplete extraction, evaporation, chemical reaction with the material, or degradation. Note that Figure 9 shows that during the first EE test the syringe pump system was delivering about 350 µg HD, which is more than what is anticipated for a 0.2 µL drop (248.5 µg, based on Table 7). Since EE is normalized to the DCS, the overshot dose does not influence the EE evaluation. The syringe pump controller was configured to deliver the correct mass according to the chromatography output of DCS samples for all future use of the syringe pump by adjusting the delivered volume to a lower value (e.g., 3.7 µL) when specifically delivering 4 µL drops. Also, the DCS in Figure 9 included one case where the vial was double spiked, this sample was removed from the calculation of the mean DCS.

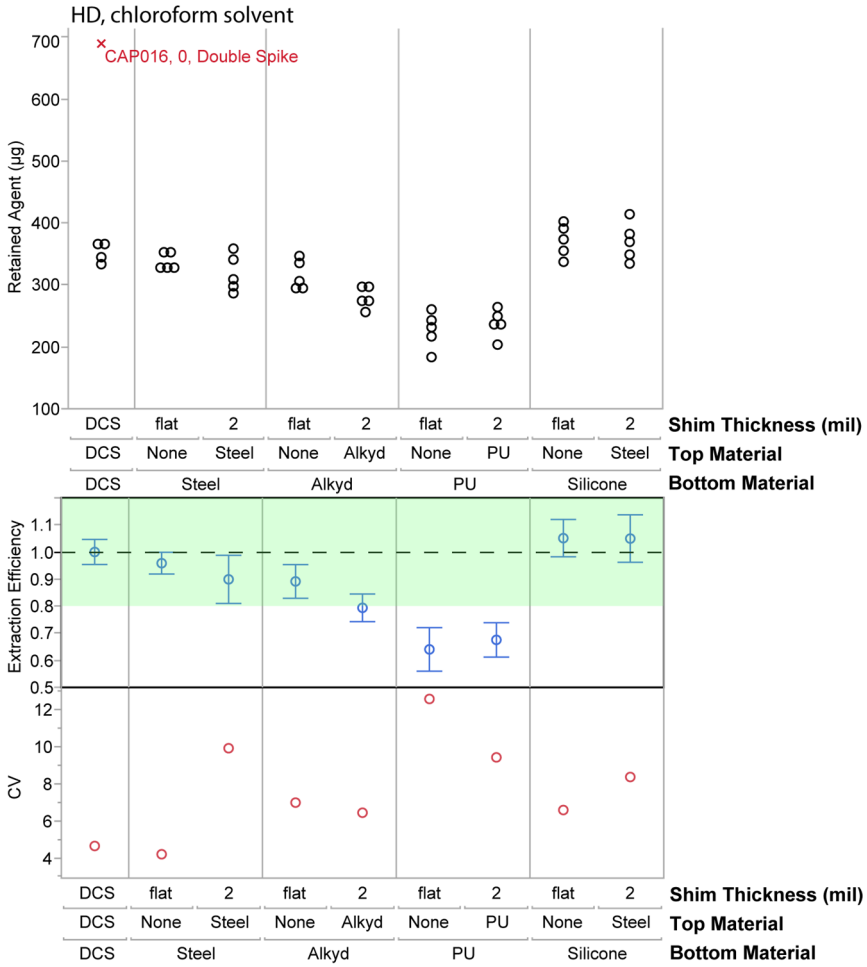


Figure 9. Extraction efficiency of HD with chloroform solvent measured with GC-MSD. Raw RA results are presented in the top graph, calculated extraction efficiency and CV (%) are provided in bottom graphs. Error bars for EE represent one standard deviation.

The use of different solvents was explored to identify if the EE for PU coatings could be improved. Multiple extraction configurations were considered, and the use of isopropanol (i.e., IPA or 2-propanol) was identified to provide superior extraction results. Additionally, the chromatography platform was changed from a GC-MSD to a triple quadrupole mass spectrometer (GC-QQQ) that provided better signal to noise and reproducibility. The EE results for HD using IPA as a solvent are provided in Figure 10. The full statistical results are presented in Table 14. Using IPA, all material and shim configurations exhibited EE > 0.8, except for PU coating with the 2 mil shim (0.798), meeting the acceptance criterion. The coefficient of variation (CV, standard deviation divided by mean) for all conditions was < 10% and as low as 4%, demonstrating that the dosing, extraction, and chromatography analysis processes are very reproducible.

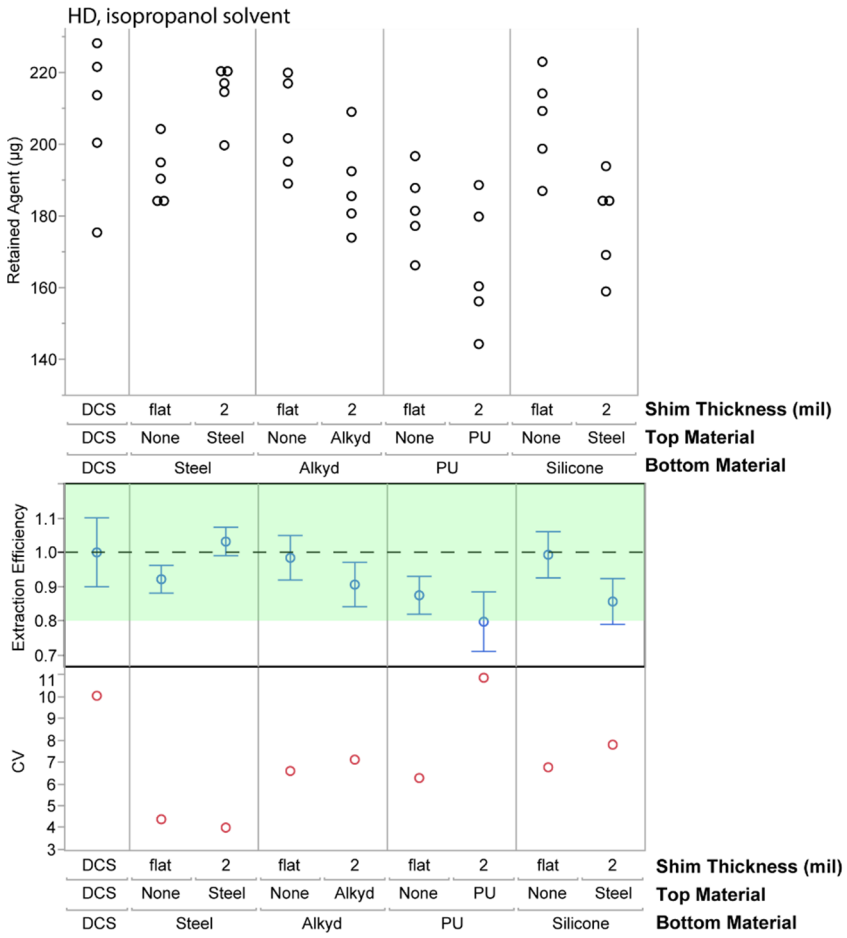


Figure 10. Extraction efficiency of HD with IPA solvent measured with GC-QQQ. Raw RA results are presented in the top graph, calculated extraction efficiency and CV (%) are provided in bottom graphs. Error bars for EE represent one standard deviation.

Table 14. Statistical Results for HD EE using IPA

Bottom Material	Top Material	Shim Thickness (mil)	Extraction Efficiency			
			N	Mean	Std Dev	CV (%)
DCS	DCS	DCS	5	1.0	0.10	10.0
Steel	None	flat	5	0.922	0.04	4.38
	Steel	2	5	1.032	0.041	4.0
Alkyd	None	flat	5	0.984	0.065	6.59
	Alkyd	2	5	0.906	0.064	7.11
PU	None	flat	5	0.875	0.055	6.27
	PU	2	5	0.798	0.087	10.9
Silicone	None	flat	5	0.993	0.067	6.76
	Steel	2	5	0.857	0.067	7.80

3.2.2 VX Extraction Efficiency

Extraction of 0.2 µL drops of VX was conducted after a 15 min age duration on all materials of interest. The individual RA results for VX extraction using IPA are presented in Figure 11. The full statistical results are presented in Table 15. In the case of flat panel PU, two samples were physically dropped during sample manipulation and not included in the analysis. All material and shim configurations provided EE > 0.8, meeting the acceptance criterion. The CV for all conditions was < 9% and as low as 3%, demonstrating that the dosing, extraction, and chromatography analysis processes are very reproducible.

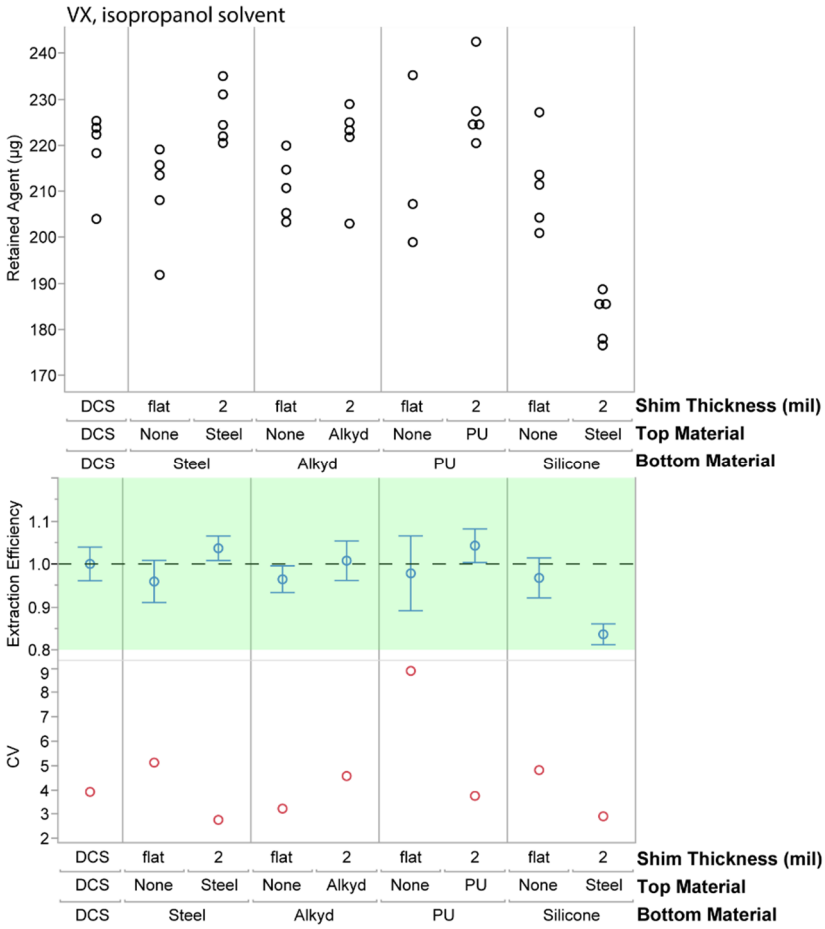


Figure 11. Extraction efficiency of VX with IPA solvent. Raw RA results are presented in the top graph, calculated extraction efficiency and CV (%) are provided in bottom graphs. Error bars for EE represent one standard deviation.

Table 15. Statistical Results for VX EE using IPA

Bottom Material	Top Material	Shim Thickness (mil)	Extraction Efficiency			
			N	Mean	Std Dev	CV (%)
DCS	DCS	DCS	5	1	0.039	3.91
Steel	None	flat	5	0.959	0.049	5.12
	Steel	2	5	1.036	0.029	2.76
Alkyd	None	flat	5	0.964	0.031	3.22
	Alkyd	2	5	1.008	0.046	4.57
PU	None	flat	3	0.978	0.087	8.9
	PU	2	5	1.042	0.039	3.75
Silicone	None	flat	5	0.967	0.047	4.81
	Steel	2	5	0.836	0.024	2.91

3.3 Summary

For all cases with HD and VX the EE was observed to be > 0.8, except for HD on PU coating with the 2 mil shim (EE = 0.798). The CV was < 10% in all cases demonstrating the reproducible dosing, extraction, and chromatography analysis processes. The chromatography platform to analyze HD samples was updated to use GC-QQQ for enhanced reproducibility and sensitivity.

4. REPRODUCIBLE AGENT DELIVERY TO PANELS

4.1 Test Design

Reproducibly contaminating the panel and generating the intended agent distribution (e.g., entrained in the shim) is a fundamental aspect of the test. Contamination for shim panels requires that the pipette or syringe tip is placed in contact with the feature, which is different than previous contamination approaches for flat panels that used ejected drops from devices such as an Eppendorf repeater pipette.

The EE study described in Section 3 confirmed that agent could be delivered and recovered for a single drop volume over a select set of materials configurations after a 15 min contamination duration. To specifically quantify the mass of agent delivered to a panel, a short contamination duration (1 min) is used to minimize evaporative loss of the agent. This effort focused on determining if mass delivered is influenced by panel orientation, feature type, the type of tool used to dispense the agent and the volume of agent delivered.

In regard to generating the intended agent distribution, as a precursor to agent delivery and recover measurements, visualization of liquid spreading using red dye infused MeS was performed (Figure 12). The sequence of images shows how the agent spreads on the material and/or flows into the shim gap with different materials. For the PU coating on the 2 mil shim, the MeS is seen to wet (darken the surface) along the top material interface, and spread on the bottom material. The drop applied to the 20 mil shim on PU seems to wet the bottom material and not interact with the top material. For both shim sizes on steel, the drop was immediately

drawn into the gap. During testing, the specific agent distribution generated within the shim panel gap is not visually accessible (see Appendix G for visualization of agent distributions using glass as a top material). It is assumed that the agent distribution generated by contamination is representative of the distribution generated in operational context.

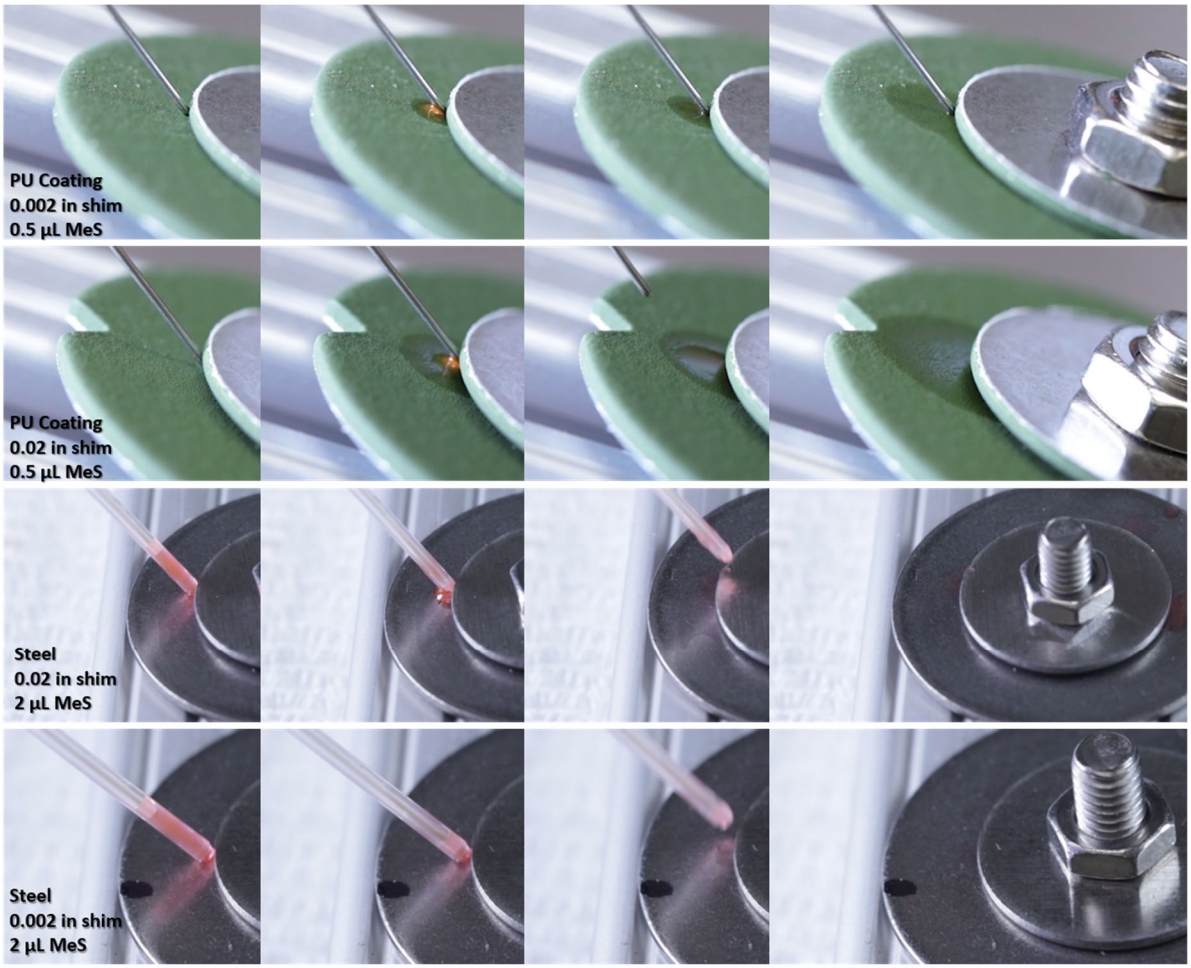


Figure 12. Images of dosing PU coated shim panels with a syringe and steel shim panels with a pipette using methyl salicylate with a red dye, for 0.02 inch and 0.002 inch shims. The sequence of images from left to right spans no more than 30 s.

A set of positive control samples was collected, using an RA test, to demonstrate the ability to reproducibly contaminate flat and shim panels. Because vertical surfaces are also considered, this positive control test included panel orientation as a variable. The drop volumes considered were limited to small (0.2 µL), moderate (1 µL), and large (4 µL) drop volumes. While the dose and recovery of the agent verifies that the desired quantity of agent was delivered, this result does not characterize the specific distribution of agent (e.g., how deeply the agent penetrates into the shim feature or if some quantity of agent flows out of the shim gap). Operators visually observed the panels to ensure drops were delivered within the shim feature and any cases where visible drops were not delivered into the feature were discarded (e.g., visible drops on the top exterior material).

The positive control test conditions are listed in Table 16. The acceptance criterion for panel RA positive control samples is a mean value within ± 20% of the DCS for each panel configuration, confirming that the expected agent mass has been delivered to and recovered from the panel. Part of the rationale for the ± 20% difference from the DCS is derived from the quality/reproducibility criterion for chromatography methods of ±15% variation allowed from continuous calibration verification samples (i.e., chromatography analysis has an ±15% variation). A sampling of 5 replicates per condition was used here, 5 replicates has been the standard value used in previous testing.[16]

Table 16. Test parameters for verification of contaminant delivery and recovery

Parameter	Conditions	Comments
Materials	Steel, PU coating	Shim panels will be assembled with the same material for top and bottom washers Steel is selected as an impermeable non-wetting material PU coating is a sorptive wetting material
Shim thickness and Panel Orientation	Flat panel, 2 mil, 20 mil Orientation: horizontal and vertical	Flat panel may be just the bottom washer
Agents	HD, VX	HD and VX present different chemistries and interactions with materials, both agents are selected here to demonstrate the ability to accurately dispense different viscosities.
Drop count ×volume (µL)	1 × 0.2 µL (syringe) 1 × 1 µL (pipette and syringe) 1 × 4 µL (pipette and syringe)	Applied using a syringe pump or pipette.
Age time	~1-2 min	Very short duration necessary; while extraction efficiency considers 15 min to enable sorption and interaction with the material, this test only uses ~2 min since the objective is to determine that the correct mass was *delivered to and recovered from* the panel.
Treatment	No treatment (positive control)	Contaminate and extract only, no treatment
Analysis Method	RA test	
DCS	5 replicates per drop volume used	Used as reference for actual mass delivered
Replication	5	--
Analysis	Quantify recovery (RA-DCS)/DCS	Verification criterion: difference ≤ 20% ratio of RA / DCS: 1 ± 0.2

4.2 Results

Positive control tests were conducted over 6 test sessions (CAP019, CAP021-024, and CAP026). The responses are presented as the individual RA data points divided by the mean of the respective DCS. For samples to be within the acceptance criterion this ratio should be 1 ± 0.2. Positive control samples have demonstrated high reproducibility (i.e., CV < 20%), and analysis by arithmetic mean or log transform of the results produce similar results (see Appendix B).[17] These results capture the process of contamination, extraction, sample dilution, and chromatography analysis, including the operator variability in each of these processes.

The nominal mass delivered per drop volume accounting for agent purity is calculated as

$$M_{Nominal} = V\rho\psi \quad \text{Eq. 2}$$

Where:

V is the volume of the drop (mL)
ρ is density of the liquid agent (g/mL)
ψ is mass purity of the agent (%)

The $M_{Nominal}$ values for the agent used here are provided in Table 17. The DCS is a measure of how a volumetric delivery tool such as a syringe or pipette delivers the specified volume to an extraction container and are quantified by the applicable chromatography platform. The accuracy of the DCS is evaluated by the ratio of DCS divided by the nominal mass. The summary of DCS results for the positive control samples are provided in Table 18. There were cases for VX at 1 µL where the sample vials were misspiked resulting in rejecting 2 samples from each of the syringe and pipette delivery replicates. Overall, the accuracy shows a slight negative bias with the syringe providing better accuracy than the equivalent pipettes. Deviations in accuracy may be caused by inaccurate purity reports for the agent (or chemical degradation since the accuracy was assessed), bias in delivered volume by the contamination tool, or a systematic bias in the extraction, dilution, and chromatographic analysis. The variations in the data tend to correlate with the contamination tool and drop volume indicating negative bias in the contamination tools may be present. The cases where the DCS are outside of the acceptance criterion correspond to the use of pipettes. For the assessment of positive controls, the evaluation is the similarity of panel results to the DCS, so the absolute accuracy of the DCS is not critical for this analysis.

Table 17. Nominal mass delivered per drop volume

Agent	Drop Volume (µL)	Nominal Mass (µg)
HD	0.2	248.5
	1	1242.6
	4	4970.6
VX	0.2	185.3
	1	926.4
	4	3705.4

*Note: HD and VX vials used for this study had purities of 98.0% and 91.9%, respectively.

Table 18. DCS accuracy compared to $M_{Nominal}$

Analyte	Contamination Tool	Agent Drop Vol. (µL)	DCS Mass (µg)				DCS Accuracy		
			N	Mean	Std Dev	CV	$M_{Nominal}$ (µg)	Mean DCS / $M_{Nominal}$	% Diff.
HD	10 µL Syringe	0.2	5	218.8	7.7	3.5	248.5	0.880	-12.0
	50 µL Syringe	1	5	1158.4	59.1	5.1	1242.6	0.932	-6.8
		4	5	4382.5	153.5	3.5	4970.6	0.882	-11.8
	P2 Pipette	0.2	5	400.4	29.5	7.4	248.5	1.611	61.1
	M10 Pipette	1	5	960.9	63.7	6.6	1242.6	0.773	-22.7
		4	5	4027.6	473.6	11.8	4970.6	0.810	-19.0
VX	10 µL Syringe	0.2	5	179.9	17.0	9.4	185.3	0.971	-2.9
	50 µL Syringe	1	3	854.6	5.2	0.6	926.4	0.923	-7.7
		4	5	2951.3	78.7	2.7	3705.4	0.796	-20.4
	M10 Pipette	1	3	707.2	81.3	11.5	926.4	0.763	-23.7
		4	5	2921.5	118.0	4.0	3705.4	0.788	-21.2

Red colored text indicates results out of acceptance criterion (1 ± 0.2)

During testing it was observed that some of the dispensed liquid agent would sometimes stick to the pipette tip and not transfer to the panel. Additionally, liquid would stick at times to the exterior pipette tip surface after drawing agent into the tip from the agent vial, even after contacting the tip to the agent vial to clean it off. Operators watched for this behavior and tried to ensure the agent was correctly delivered. This behavior was not observed for the steel needle of the syringe. This is one of several factors (discussed later) that led to the use of syringes for agent delivery.

4.2.1 HD Positive Controls

The results of the positive control data for HD contamination with both a pipette and a syringe are provided in Figure 13-Figure 18 and Table 19-Table 24. The results for 0.2 µL drops are provided in Figure 13 and Figure 14, and Table 19 and Table 20, for syringe and pipette, respectively. For 0.2 µL drops, the syringe is more accurate (i.e., RA/DCS is closer to 1.0) and reproducible (i.e., smaller CV). In the graphs used throughout the report orientation is denoted as H for horizontal and V for vertical. No significant differences in RA were observed between horizontal and vertical panels.

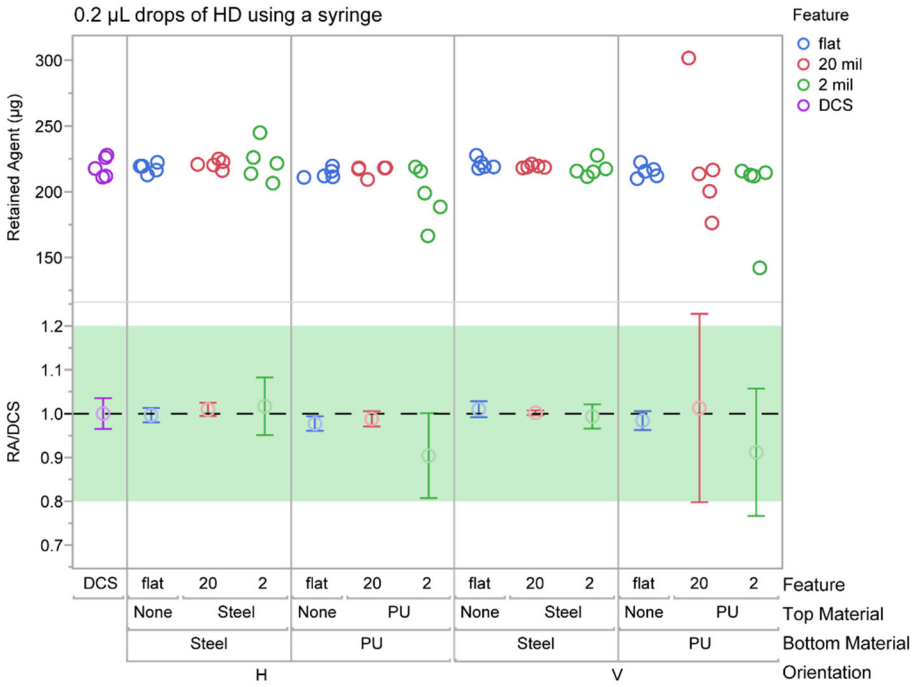


Figure 13. Positive control samples for 0.2 µL drops of HD using a syringe. Individual RA data points are provided in the top graph, calculated RA/DCS ratio in the bottom graph. The green shaded region indicates the acceptance criterion of 1 ± 0.2.

Table 19. Statistics for positive control samples for 0.2 µL drops of HD using a syringe

Orientation	Bottom Material	Top Material	Shim Thickness (mil)	N	Retained Agent (µg)		RA / DCS		
					Mean	Std. Dev.	Mean	Std. Dev.	CV
H	DCS	DCS	DCS	5	218.82	7.74	1.00	0.04	3.54
	Steel	None	flat	5	218.04	3.60	1.00	0.02	1.65
	Steel	Steel	20	5	220.95	3.30	1.01	0.02	1.49
	Steel	Steel	2	5	222.49	14.48	1.02	0.07	6.51
	PU	None	flat	5	213.86	3.64	0.98	0.02	1.70
	PU	PU	20	5	216.18	3.84	0.99	0.02	1.78
	PU	PU	2	5	197.72	21.27	0.90	0.10	10.76
V	Steel	None	flat	5	221.0	4.00	1.01	0.02	1.81
	Steel	Steel	20	5	219.19	1.15	1.00	0.01	0.53
	Steel	Steel	2	5	217.42	6.04	0.99	0.03	2.78
	PU	None	flat	5	215.34	4.77	0.98	0.02	2.22
	PU	PU	20	5	221.61	47.19	1.01	0.22	21.30
	PU	PU	2	5	199.43	31.89	0.91	0.15	15.99

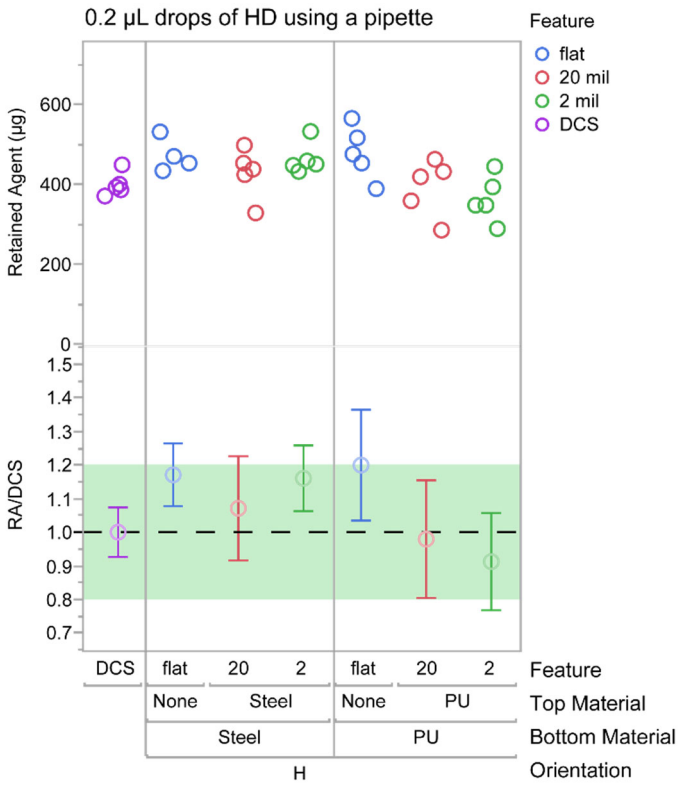


Figure 14. Positive control samples for 0.2 µL drops of HD using a P2 pipette.

Table 20. Statistics for positive control samples for 0.2 µL drops of HD using a P2 pipette

Orientation	Bottom Material	Top Material	Shim Thickness (mil)	N	Retained Agent (µg)		RA / DCS		
					Mean	Std. Dev.	Mean	Std. Dev.	CV (%)
H	DCS	DCS	DCS	5	400.4	29.51	1.00	0.07	7.37
	Steel	None	flat	5	468.76	37.41	1.17	0.09	7.98
	Steel	Steel	20	5	428.89	62.17	1.07	0.16	14.5
	Steel	Steel	2	5	464.68	39.22	1.16	0.10	8.44
	PU	None	flat	5	480.39	66.12	1.20	0.17	13.76
	PU	PU	20	5	392.13	70.20	0.98	0.18	17.9
	PU	PU	2	5	365.23	58.03	0.91	0.15	15.89

The results for 1 µL drops are provided in Figure 15 and Figure 16, and Table 21 and Table 22, for syringe and pipette, respectively. For 1 µL drops, the syringe is more accurate (i.e., RA/DCS is closer to 1.0) and reproducible (i.e., smaller CV). Pipette delivery in this case had significant positive bias, i.e., RA/DCS > 1, exceeding the criterion of 1 ± 0.2 (Figure 16). No significant differences in RA were observed between horizontal and vertical panels. For both horizontal and vertical orientations of the 2 mil shim for PU there was a low recovery of HD. This corresponds to the same panel configuration where low EE was observed. If low EE has been established for one drop volume, it would be expected to observe low recovery for other drop volumes as well, which is observed, though not to the same extent. In the vertical orientation for flat panels using the syringe, two samples may have been double dosed and were removed as outliers.

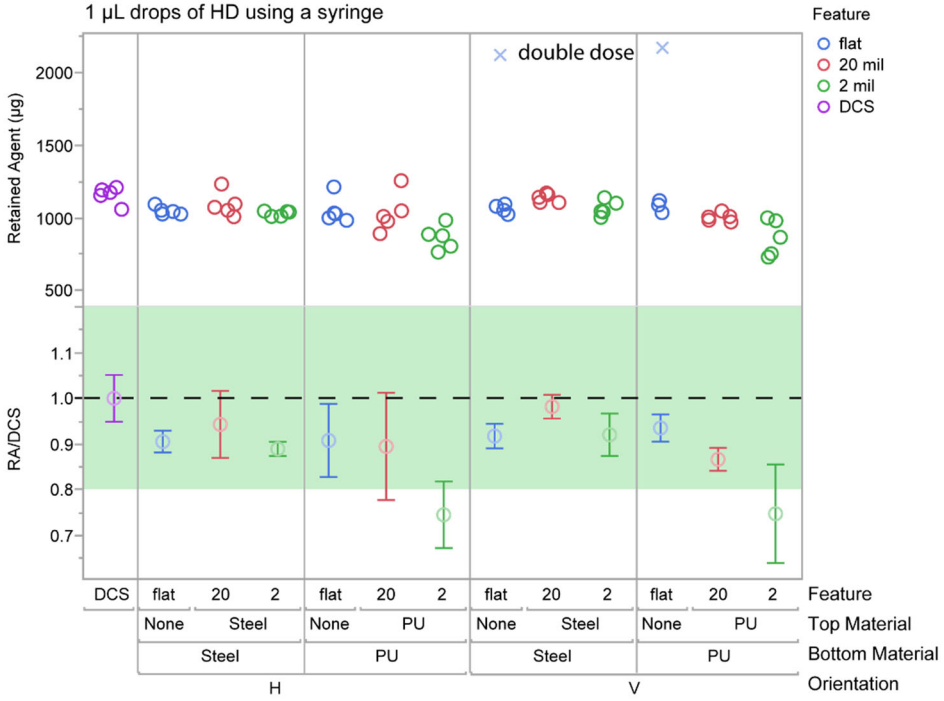


Figure 15. Positive control samples for 1 µL drops of HD using a syringe. Data points indicated with an × were excluded from the statistical analysis.

Table 21. Statistics for positive control samples for 1 µL drops of HD using a syringe

Orientation	Bottom Material	Top Material	Shim Thickness (mil)	N	Retained Agent (µg)		RA / DCS		
					Mean	Std. Dev.	Mean	Std. Dev.	CV (%)
H	DCS	DCS	DCS	5	1158.41	59.10	1.00	0.05	5.10
	Steel	None	flat	5	1048.79	27.67	0.91	0.02	2.64
	Steel	Steel	20	5	1092.33	84.88	0.94	0.07	7.77
	Steel	Steel	2	5	1030.39	18.01	0.89	0.02	1.75
	PU	None	flat	5	1051.65	92.63	0.91	0.08	8.81
	PU	PU	20	5	1036.51	135.88	0.89	0.12	13.11
	PU	PU	2	5	863.44	84.36	0.75	0.07	9.77
V	Steel	None	flat	4	1062.81	31.15	0.92	0.03	2.93
	Steel	Steel	20	5	1137.46	29.92	0.98	0.03	2.63
	Steel	Steel	2	5	1066.13	53.75	0.92	0.05	5.04
	PU	None	flat	4	1083.34	34.53	0.94	0.03	3.19
	PU	PU	20	5	1003.95	29.12	0.87	0.03	2.90
	PU	PU	2	5	866.16	124.73	0.75	0.11	14.40

Red colored text indicates results out of acceptance criterion (1 ± 0.2)

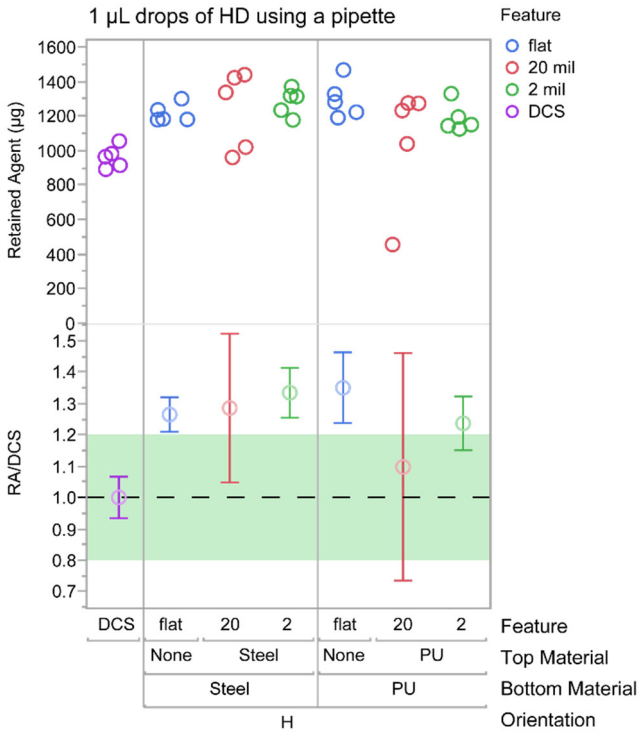


Figure 16. Positive control samples for 1 µL drops of HD using a M10 pipette.

Table 22. Statistics for positive control samples for 1 µL drops of HD using a M10 pipette

Orientation	Bottom Material	Top Material	Shim Thickness (mil)	N	Retained Agent (µg)		RA / DCS		
					Mean	Std. Dev.	Mean	Std. Dev.	CV (%)
H	DCS	DCS	DCS	5	960.87	63.65	1.00	0.07	6.62
	Steel	None	flat	5	1215.20	53.03	1.27	0.05	4.36
	Steel	Steel	20	5	1235.16	228.55	1.29	0.24	18.5
	Steel	Steel	2	5	1282.28	76.43	1.33	0.08	5.96
	PU	None	flat	5	1297.32	108.39	1.35	0.11	8.36
	PU	PU	20	5	1054.36	349.09	1.10	0.36	33.11
	PU	PU	2	5	1188.16	82.82	1.24	0.09	6.97

Red colored text indicates results out of acceptance criterion (1 ± 0.2)

The results for 4 µL drops are provided in Figure 17 and Figure 18, and Table 23 and Table 24, for syringe and pipette, respectively. All results are within the acceptance criterion.

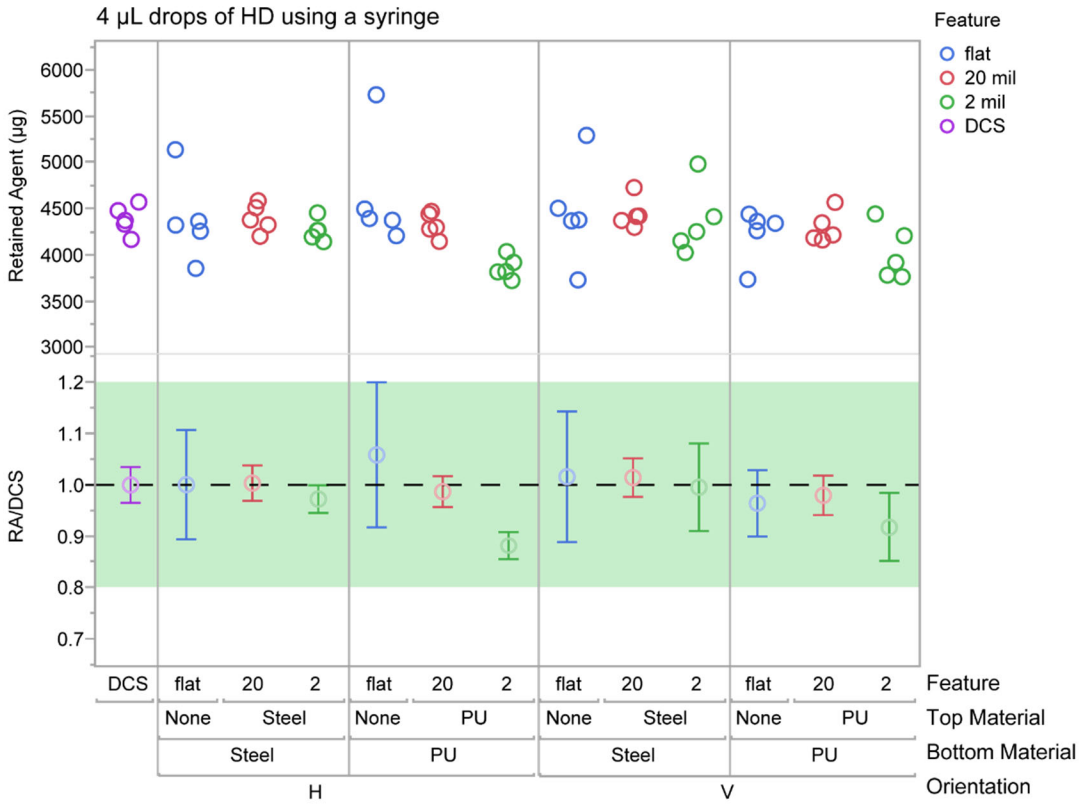


Figure 17. Positive control samples for 4 µL drops of HD using a syringe.

Table 23. Statistics for positive control samples for 4 µL drops of HD using a syringe

Orientation	Bottom Material	Top Material	Shim Thickness (mil)	N	Retained Agent (µg)		RA / DCS		
					Mean	Std. Dev.	Mean	Std. Dev.	CV (%)
H	DCS	DCS	DCS	5	4382.52	153.53	1.00	0.04	3.50
	Steel	None	flat	5	4384.81	466.78	1.00	0.11	10.65
	Steel	Steel	20	5	4398.03	151.58	1.00	0.04	3.45
	Steel	Steel	2	5	4261.75	118.01	0.97	0.03	2.77
	PU	None	flat	5	4639.26	619.56	1.06	0.14	13.35
	PU	PU	20	5	4325.26	132.03	0.99	0.03	3.05
	PU	PU	2	5	3859.46	119.31	0.88	0.03	3.09
V	Steel	None	flat	5	4452.43	558.45	1.02	0.13	12.54
	Steel	Steel	20	5	4445.24	164.73	1.01	0.04	3.71
	Steel	Steel	2	5	4362.69	374.23	1.00	0.09	8.58
	PU	None	flat	5	4225.75	283.51	0.96	0.07	6.71
	PU	PU	20	5	4293.05	168.81	0.98	0.04	3.93
	PU	PU	2	5	4020.00	294.91	0.92	0.07	7.34

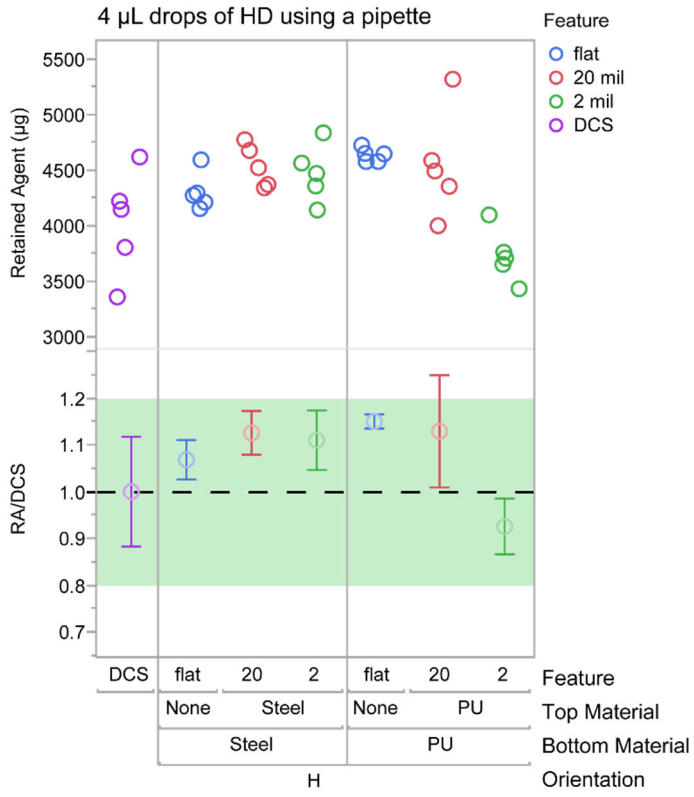


Figure 18. Positive control samples for 4 µL drops of HD using a M10 pipette.

Table 24. Statistics for positive control samples for 4 µL drops of HD using a M10 pipette

Orientation	Bottom Material	Top Material	Shim Thickness (mil)	N	Retained Agent (µg)		RA / DCS		
					Mean	Std. Dev.	Mean	Std. Dev.	CV (%)
H	DCS	DCS	DCS	5	4027.60	473.65	1.00	0.12	11.76
	Steel	None	flat	5	4302.89	170.11	1.07	0.04	3.95
	Steel	Steel	20	5	4534.67	188.46	1.13	0.05	4.16
	Steel	Steel	2	5	4471.64	256.70	1.11	0.06	5.74
	PU	None	flat	5	4633.14	61.17	1.15	0.02	1.32
	PU	PU	20	5	4548.22	484.01	1.13	0.12	10.64
	PU	PU	2	5	3727.58	240.47	0.93	0.06	6.45

4.2.2 VX Positive Controls

The results of the positive control data for VX contamination with both a pipette and a syringe are provided in Figure 19-Figure 23 and Table 25-Table 29. The results for 0.2 µL drops of VX delivered with a syringe are provided in Figure 19 and Table 25. In this case, pipette data was not acquired. No significant differences were observed for horizontal compared to vertical contamination.

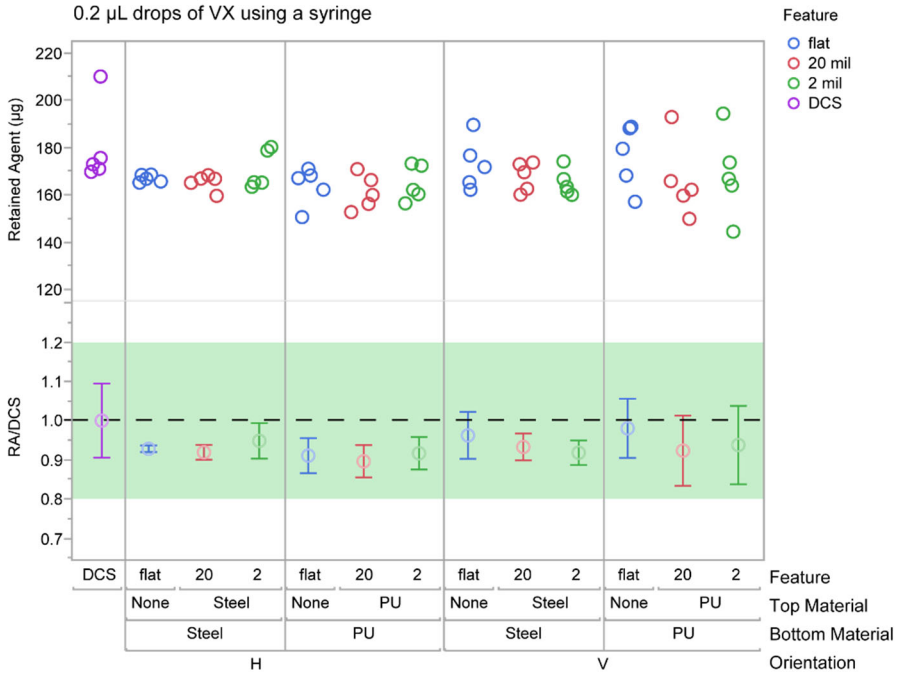


Figure 19. Positive control samples for 0.2 µL drops of VX using a syringe.

Table 25. Statistics for positive control samples for 0.2 µL drops of VX using a syringe

Orientation	Bottom Material	Top Material	Shim Thickness (mil)	N	Retained Agent (µg)		RA / DCS		
					Mean	Std. Dev.	Mean	Std. Dev.	CV (%)
H	DCS	DCS	DCS	5	179.9	16.99	1.00	0.09	9.45
	Steel	None	flat	5	166.9	1.54	0.93	0.01	0.92
	Steel	Steel	20	5	165.3	3.41	0.92	0.02	2.06
	Steel	Steel	2	5	170.6	8.18	0.95	0.05	4.80
	PU	None	flat	5	163.8	8.04	0.91	0.05	4.91
	PU	PU	20	5	161.2	7.37	0.90	0.04	4.57
	PU	PU	2	5	164.9	7.50	0.92	0.04	4.55
V	Steel	None	flat	5	173.1	10.79	0.96	0.06	6.23
	Steel	Steel	20	5	167.8	6.14	0.93	0.03	3.66
	Steel	Steel	2	5	165.1	5.62	0.92	0.03	3.40
	PU	None	flat	5	176.3	13.61	0.98	0.08	7.72
	PU	PU	20	5	166.1	16.10	0.92	0.09	9.69
	PU	PU	2	5	168.7	17.99	0.94	0.10	10.67

The results for 1 µL drops of VX are provided in Figure 20 and Figure 21, and Table 21 and Table 22, for syringe and pipette, respectively. The positive bias observed for the pipette with HD was not observed for VX. No significant differences in RA were observed between horizontal and vertical panels. For both horizontal and vertical orientation of the 2 mil shim for PU there was a low recovery of HD, but this trend was *not* observed for VX.

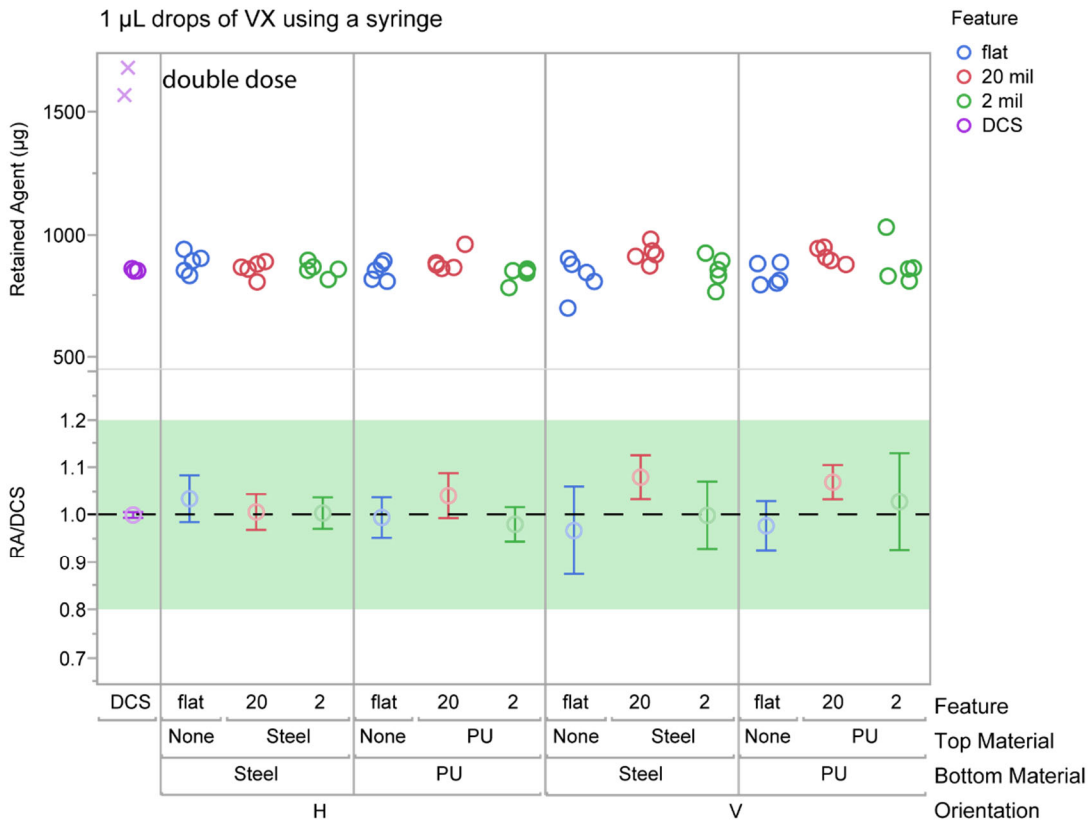


Figure 20. Positive control samples for 1 µL drops of VX using a syringe. Data points indicated with an × were excluded from the statistical analysis.

Table 26. Statistics for positive control samples for 1 µL drops of VX using a syringe

Orientation	Bottom Material	Top Material	Shim Thickness (mil)	N	Retained Agent (µg)		RA / DCS		
					Mean	Std. Dev.	Mean	Std. Dev.	CV (%)
H	DCS	DCS	DCS	3	854.61	5.19	1.00	0.01	0.61
	Steel	None	flat	5	883.70	41.91	1.03	0.05	4.74
	Steel	Steel	20	5	859.96	32.04	1.01	0.04	3.73
	Steel	Steel	2	5	858.06	28.11	1.00	0.03	3.28
	PU	None	flat	5	850.08	36.42	0.99	0.04	4.28
	PU	PU	20	5	889.17	40.32	1.04	0.05	4.53
	PU	PU	2	5	837.86	30.99	0.98	0.04	3.70
V	Steel	None	flat	5	826.56	78.91	0.97	0.09	9.55
	Steel	Steel	20	5	922.35	39.31	1.08	0.05	4.26
	Steel	Steel	2	5	853.91	60.31	1.00	0.07	7.06
	PU	None	flat	5	835.24	44.39	0.98	0.05	5.31
	PU	PU	20	5	913.33	30.59	1.07	0.04	3.35
	PU	PU	2	5	878.43	86.84	1.03	0.10	9.89

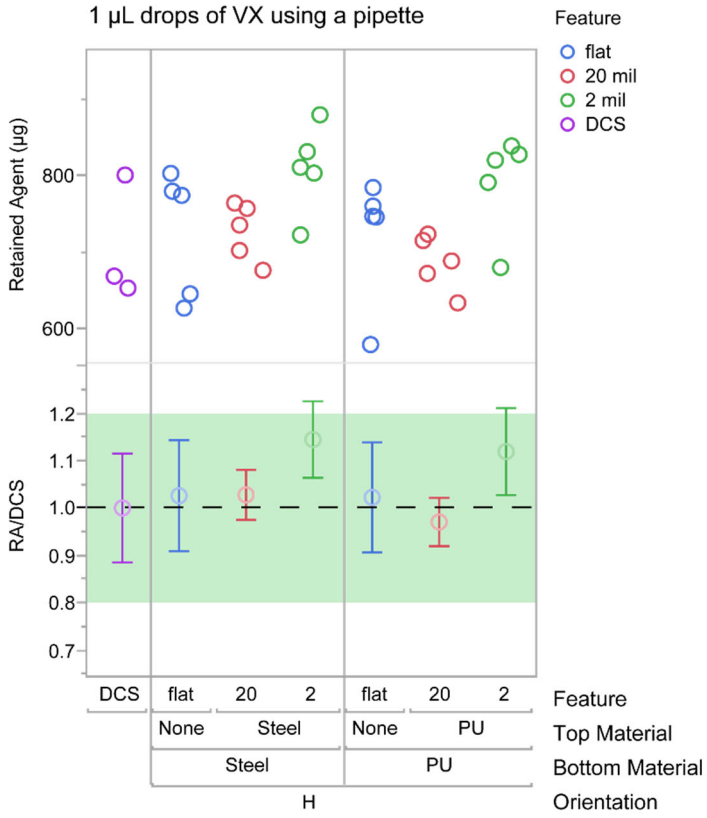


Figure 21. Positive control samples for 1 µL drops of VX using a M10 pipette.

Table 27. Statistics for positive control samples for 1 µL drops of VX using a pipette

Orientation	Bottom Material	Top Material	Shim Thickness (mil)	N	Retained Agent (µg)		RA / DCS		
					Mean	Std. Dev.	Mean	Std. Dev.	CV (%)
H	DCS	DCS	DCS	3	707.19	81.28	1.00	0.11	11.49
	Steel	None	flat	5	725.55	82.95	1.03	0.12	11.43
	Steel	Steel	20	5	726.78	37.32	1.03	0.05	5.13
	Steel	Steel	2	5	809.35	57.05	1.14	0.08	7.05
	PU	None	flat	5	723.00	82.16	1.02	0.12	11.36
	PU	PU	20	5	686.30	36.07	0.97	0.05	5.26
	PU	PU	2	5	791.38	64.92	1.12	0.09	8.20

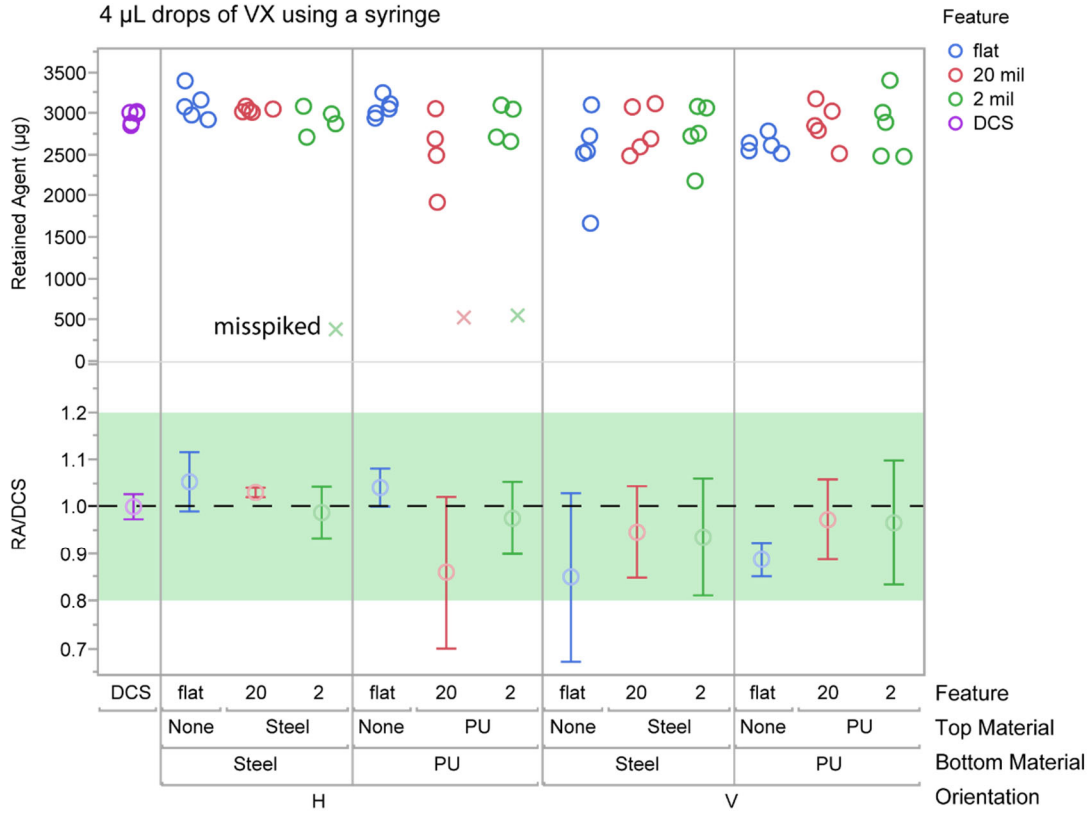


Figure 22. Positive control samples for 4 µL drops of VX using a syringe. Data points indicated with an × were excluded from the statistical analysis.

Table 28. Statistics for positive control samples for 4 µL drops of VX using a syringe

Orientation	Bottom Material	Top Material	Shim Thickness (mil)	N	Retained Agent (µg)		RA / DCS		
					Mean	Std. Dev.	Mean	Std. Dev.	CV (%)
H	DCS	DCS	DCS	5	2951.3	78.72	1.000	0.027	2.67
	Steel	None	flat	5	3106.8	184.57	1.053	0.063	5.94
	Steel	Steel	20	5	3040.2	29.58	1.030	0.01	0.97
	Steel	Steel	2	4	2914.7	161.69	0.988	0.055	5.55
	PU	None	flat	5	3071.1	117.78	1.041	0.04	3.84
	PU	PU	20	4	2538.3	473.99	0.860	0.161	18.67
	PU	PU	2	4	2879.1	226.92	0.976	0.077	7.88
V	Steel	None	flat	5	2509.1	526.93	0.850	0.179	21.00
	Steel	Steel	20	5	2791.8	287.84	0.946	0.098	10.31
	Steel	Steel	2	5	2760.5	366.05	0.935	0.124	13.26
	PU	None	flat	5	2618.2	105.35	0.887	0.036	4.02
	PU	PU	20	5	2870.3	251.27	0.973	0.085	8.75
	PU	PU	2	5	2850.8	388.74	0.966	0.132	13.64

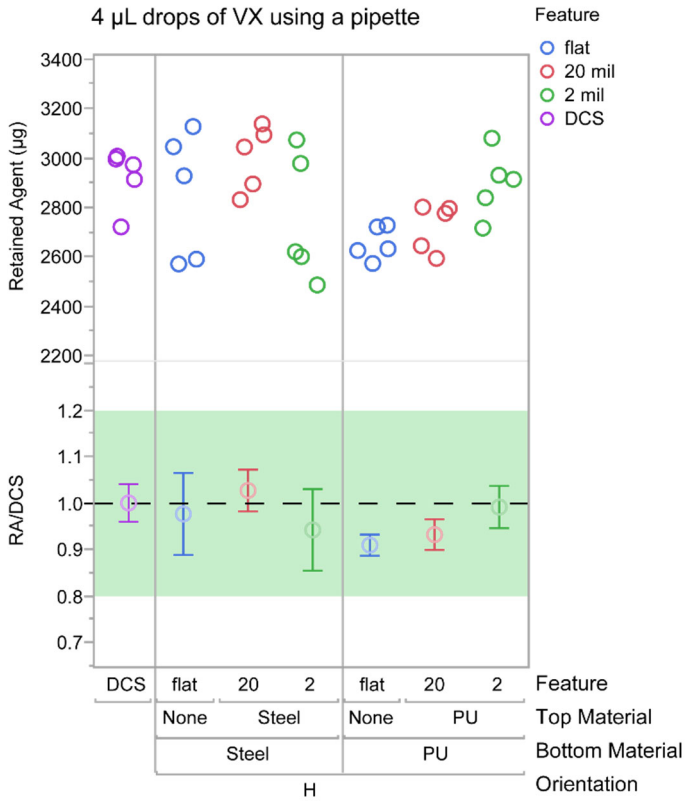


Figure 23. Positive control samples for 4 µL drops of VX using a M10 pipette.

Table 29. Statistics for positive control samples for 4 µL drops of VX using a pipette

Orientation	Bottom Material	Top Material	Shim Thickness (mil)	N	Retained Agent (µg)		RA / DCS		
					Mean	Std. Dev.	Mean	Std. Dev.	CV (%)
H	DCS	DCS	DCS	5	2921.5	117.96	1.00	0.04	4.04
	Steel	None	flat	5	2852.0	257.86	0.98	0.09	9.04
	Steel	Steel	20	5	2999.9	131.59	1.03	0.05	4.39
	Steel	Steel	2	5	2751.3	257.14	0.94	0.09	9.35
	PU	None	flat	5	2655.7	66.53	0.91	0.02	2.51
	PU	PU	20	5	2721.9	96.28	0.93	0.03	3.54
	PU	PU	2	5	2895.4	133.10	0.99	0.05	4.60

4.3 Summary

Overall, the positive control samples demonstrate that agent can be delivered to and recovered from panels of different materials, different orientations, and flat and shim configurations within the acceptance criterion of RA/DCS being within 1 ± 0.2 (i.e., ±20% of DCS). The average CV across all conditions was 7.7%, demonstrating the reproducibility of the ability to apply and recover the agent.

Select agent-material-feature configurations did have some occasional potential 'outlier' cases, but they seemed more random than associated with specific features such as shim thickness. For HD with PU and the 2 mil shim thickness, lower recovery was observed, and for 1 µL drops these recoveries were outside of the acceptance criterion. This coincides with cases where the EE was observed to be low.

Figure 24 provides a summary of all conditions tested per agent, drop volume, and delivery tool used. Figure 24 shows that in many cases the pipette and syringe delivery are similar and within acceptance levels, with syringe providing a slightly improved accuracy and precision. The positive bias for the 1 µL drops with HD may be associated with an observation that sometimes liquid agent adhered to the plastic pipette tip during the loading of the pipette. Operators were more careful to ensure removal of any liquid on the outer pipette tip after this test was performed.

The laboratory operators indicated a preference to use the syringe moving forward based on perceived safer operations, it was easier to place the syringe point at the desired location on the shim panel, and that a syringe could deliver multiple drops before reloading with agent. Whereas the pipette requires reloading for each aliquot. All other results presented here used the syringe pump for contamination.

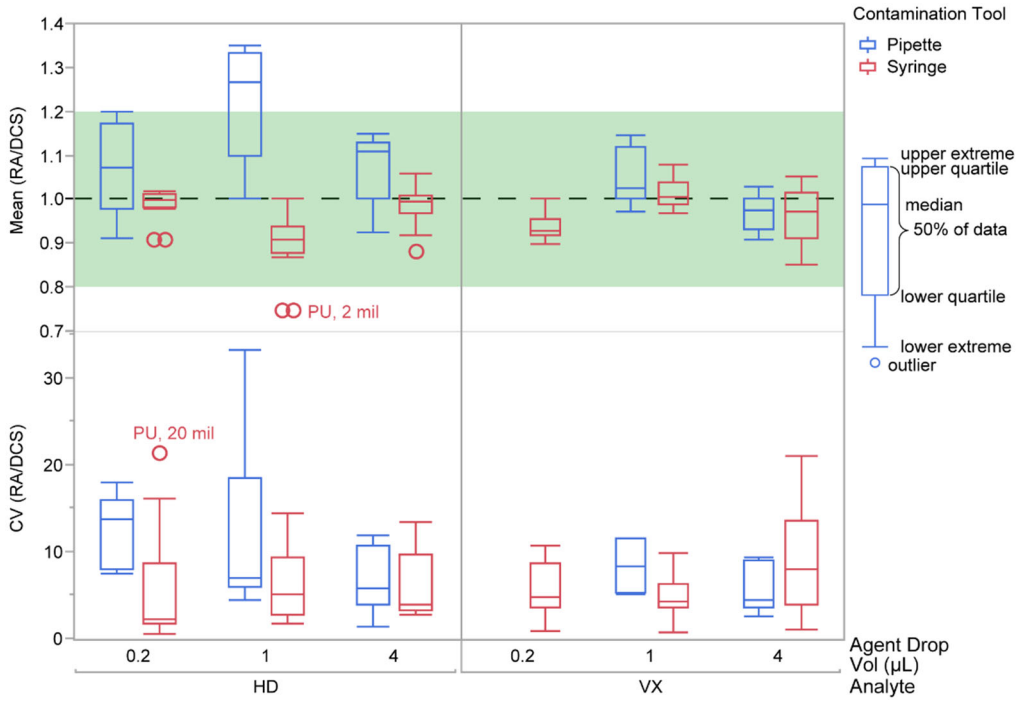


Figure 24. Summary of RA/DCS responses and CV for all positive control samples as a box and whisker plots comparing pipette and syringe delivery.

5. SENSITIVITY ANALYSIS FOR MATERIAL SELECTION

The calculation of an asset-level output requires that corresponding laboratory data is available to represent the asset. However, acquiring data for all materials in all orientations for all features (e.g., flat and shim panel) may present significant test burden and *may not be necessary*. A few material-feature combinations may be responsible for the vast majority of retained agent on an asset.[3] However, it is not known *a priori* which material-feature combinations may be the significant contributors. It has been observed on flat panels that the materials identified as the most significant agent sources can vary across agents.[18] A material prioritization process to identify the most significant agent sources was introduced into the methodology as part of a test guide. The identification of a high priority material-feature is a combination of source term magnitude, abundance of the material-feature on the asset of interest, and probability of contamination.

The following testing is conducted to help identify approaches to reduce the number of material-feature combinations that must be specifically tested. For example, shim panels may produce similar results in both horizontal and vertical orientations if capillary flow within the shim panel gap is more influential than gravity driven flow effects (for the agent and/or treatment processes). If it can be established that certain results will be similar in magnitude, then one condition can be tested and used to represent multiple conditions. The cost to acquire data for *all* conditions may not be consistent with program funding and schedule constraints, and may not provide significantly better accuracy than testing a subset of conditions and using these results to represent multiple conditions. This sensitivity study was conducted to identify the magnitude of differences in RA responses between orientation, shim panel features, and liquid spray variations to identify which test conditions are the most critical.

The introduction of shim panels and their flow restrictions provide a case where both panel orientation and specific decontaminant application procedures may significantly influence the results. An overview of previous orientation testing is provided in Appendix E, which shows that for many agent-materials combinations, RA for *horizontal* flat panels tends to be similar to or greater than that of *vertically* oriented panels. If the horizontal panel data produces similar or greater RA than vertical orientations, then these data represent the worst-case condition and horizontal panel data could be used to represent both horizontal and vertical surfaces in asset level calculations. This kind of simplification can significantly reduce test burden.

The following test was designed for use as a sensitivity analysis to demonstrate when orientation data or decontaminant spray variations may be *necessary* to enable accurate asset-level outputs. The purpose of the test is to gain an approximate order of magnitude effect of the variations on RA. This can be used to prioritize the data to be acquired, or to provide an ability to eliminate some tested conditions. The MAP identified an initial threshold of features with greater than 0.4 LD as being sufficiently significant to warrant collection in future test efforts.

5.1 Test Design

The test design includes two agents (HD, VX), two panel orientations (horizontal, vertical), both flat and shim panels, two spray angles relative to horizontal (30°, 90°) and two spray variations. A spray time of 4 corresponds to spraying the panel for 4 s at the point of contamination while 2 × 2 corresponds to spraying the side of the shim where the agent was directly applied for 2 s, then spraying the opposite side of the shim panel for an additional 2 s.

Comparison of the RA data for panels in a horizontal versus vertical orientation is one of the primary aspects of the sensitivity test. The sensitivity test is aimed at identifying approximate order of magnitude effects. It is proposed that a significant effect is defined as having an LD between RA responses from both orientations of ± 0.4, therefore horizontal panels can be used as a worst-case approximation if the LD between horizontal and vertical conditions is statistically significant and the mean is ≥ 0.4. The LD calculation is defined in Appendix B.3. If results are statistically similar, it may be possible to reduce future testing. Conversely, identifying cases where the measured response is significantly different enables the identification of factors that should be considered in test designs to provide either more accurate results, or an acknowledgement of a worst-case characterization of a technology.

Quantitative comparisons of the RA resulting from soapy water spray between the two spray angles and between the two spray variations were conducted to demonstrate how these aspects influenced efficacy and repeatability (e.g., via CV). A supplementary analysis of these parameters was conducted using a statistical model and is provided in Appendix D. Because of the number of permutations being evaluated and the order of magnitude sensitivity goal, condition replication was set to 4 samples and only one drop volume was selected. Table 30 provides the detailed test conditions.

Table 30. Test parameters to characterize effects of decontaminant application orientation and panel orientation.

Parameter	Conditions	Comments
Materials	Steel (impermeable), PU Coating (wetting, sorptive), Alkyd coating (sessile, sorptive)	Range of materials with different interactions for sorption and wetting characteristics
Agent	HD, VX	HD and VX present different chemistries and interactions with materials, both agents are selected here to demonstrate the ability to recover different contaminants
Age Time	60 min	Typical contamination duration used in previous testing and documentation.[16]
Shim Thickness and Panel Orientation	Flat Panel (horizontal, vertical), 0.002, 0.020 in (horizontal, vertical)	All combinations are tested here to determine whether the conditions are different enough to warrant the expense of always characterizing multiple orientations.
Spray Application	30° and 90° (normal to panel) spray application	Two conditions to indicate the effect of spray angle on efficacy and reproducibility
Drop count × volume (µL)	1 ×1 µL applied via M10 pipette	Single drop application using a standard PDP; single drop volume specified due to number of panel conditions being investigated
Temperature	21 °C	Ambient laboratory conditions ±3 °C, RH not controlled in hood environment
Treatments	Flat panels and shim panels:1) Preval soapy water spray at ~30° from parallel to panel surface, 4 second (sec) spray, 30 min duration + post water rinse Shim panels only: 2) Preval soapy water spray normal (top-down) to panel surface, 4 s spray 3) Preval spray at ~30° from parallel to panel surface, ~2 sec spray on front of panel, ~2 sec spray from opposite side of panel	30 min soapy water duration before performing post-rinse. The soapy water is sprayed with the Preval sprayer for about 5 sec from the side of the panel. Water rinse is 3 × 20 mL aliquots at 1 L/min flow rate from a ¼ in OD tube, dispensed by a peristaltic pump.
Analysis Method	RA test	Acceptability criteria for future testing based on comparison of vertical versus horizontal results. If horizontal RA greater than vertical RA then future testing may use horizontal testing as worst case.
Replication	4	Screening level study, lower replication to enable more parameters to be tested

5.2 Results

These data were collected over 6 test sessions, CAP028 through CAP033. DCS was within acceptable limits as shown in Table 30. In some cases, there are data with wide (i.e., orders of magnitude) distributions or present single value outliers that may be orders of magnitude different from the mean. It is recognized that some decontamination treatments (or material-features) may be highly variable, the methodology characterizes this variability and accounts for it in subsequent scale-up calculations. Therefore, data that appear as ‘outliers’ were not removed from the analysis. As a supplementary analysis, a statistical model was used to fit the treatment parameters over the tested conditions to quantify the effects of orientation, material, feature size, and treatment parameters for each agent (Appendix E).

Table 31. DCS accuracy compared to nominal mass

Analyte	Contamination Tool	Agent Drop Vol. (µL)	DCS Mass (µg)				DCS Accuracy		
			N	Mean	Std Dev	CV	Nominal Mass (µg)	Mean DCS / Nominal	% Diff.
HD	10 µL Syringe	1	15	1140.1	52.6	4.6	1242.6	0.917	-8.2
VX	10 µL Syringe	1	15	751.2	46.3	6.2	926.4	0.811	-18.9

5.2.1 HD Data for Steel

The results for the varying orientations and treatment conditions for HD on steel are provided in Figure 25. Following the treatment processes, regardless of orientation, the 2 mil shims retained the majority of HD that was applied. The 20 mil shim results show that RA was reduced by about and order of magnitude, and that the error bars across the conditions overlap. For both the 2 and 20 mil shims *the treatment parameters of panel orientation and spray variations had no effect*. Contrary to the results from previous studies (Appendix D), for flat panels the horizontal orientation resulted in less RA then the vertical orientation. Variations in both orientation and additional treatment conditions will be evaluated in depth in the statistical analysis in Section 5.3.

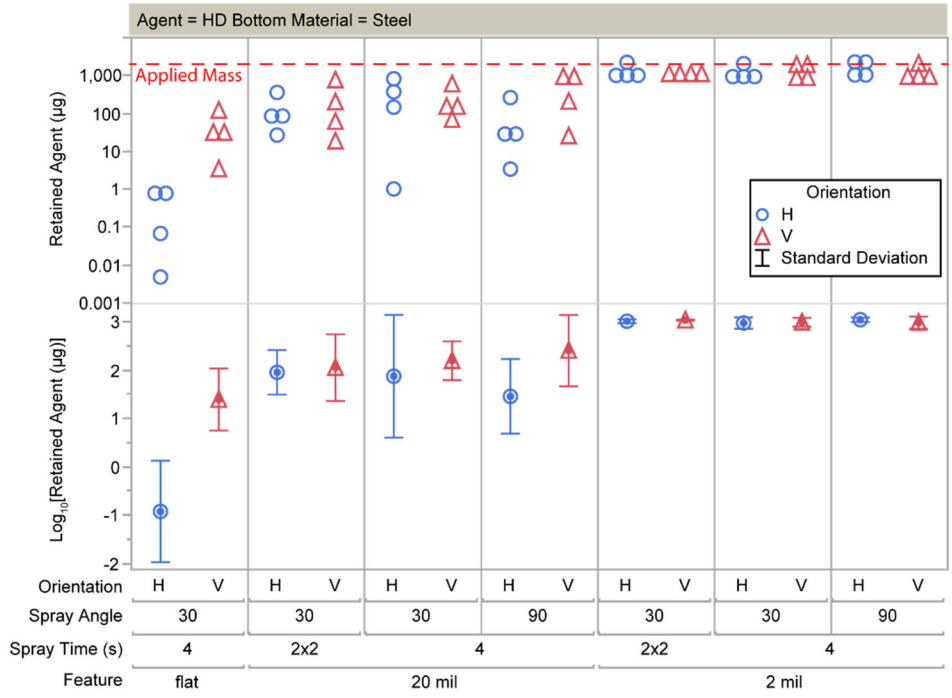


Figure 25. Sensitivity data for HD on steel with soapy water spray.

5.2.2 HD Data for Alkyd Coating

The results of the sensitivity analysis for the varying orientations and treatment conditions for HD on an Alkyd coating are provided in Figure 26. Similar to steel, the retention of HD was high for 2 mil shim sizes and some agent removal was observed for 20 mil shims. Visual inspection of the data indicates that orientation nor spray variations significantly influenced the observed RA, except for the 20 mil vertical orientation using the 2×2 spray pattern. Overall, the horizontal orientation tended to retain slightly more HD, indicating that studying the horizontal orientation alone may provide a worst-case estimation of RA.

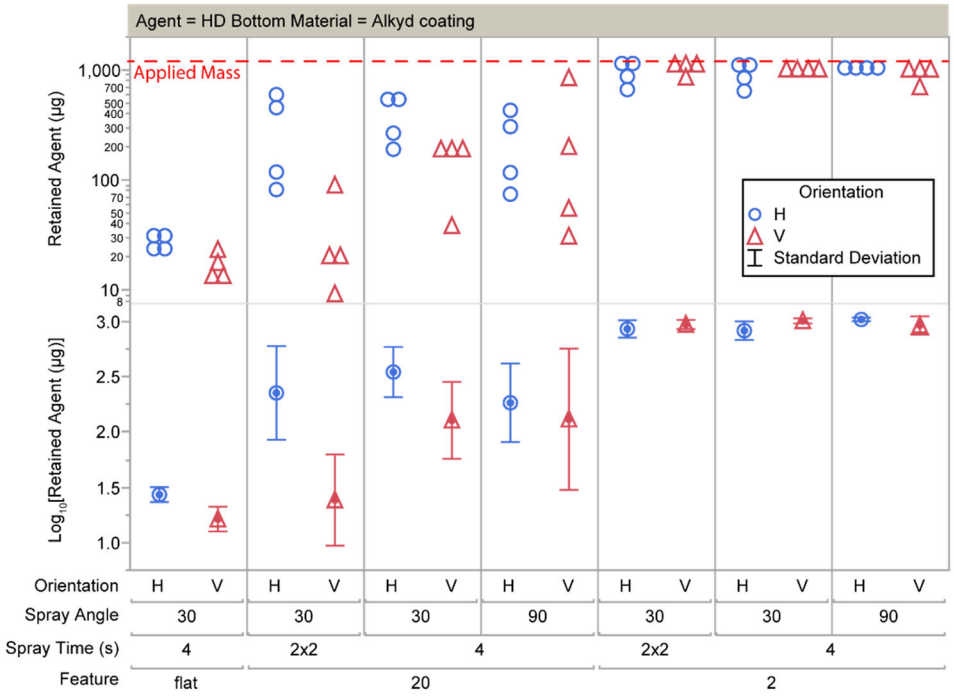


Figure 26. Sensitivity data for HD on Alkyd coating with soapy water spray.

5.2.3 HD Data for Polyurethane Coating

The results of the sensitivity analysis for the varying orientations and treatment conditions for HD on a PU coating are provided in Figure 27. Similar to HD on steel and PU, the retention of HD was significant for 2 mil shims, and somewhat lower for the 20 mil shim size. Visual inspection of the data indicates that orientation nor spray variations significantly influenced the observed RA. However, the 2×2 spray pattern for 20 mil horizontal orientation may have removed slightly more HD.

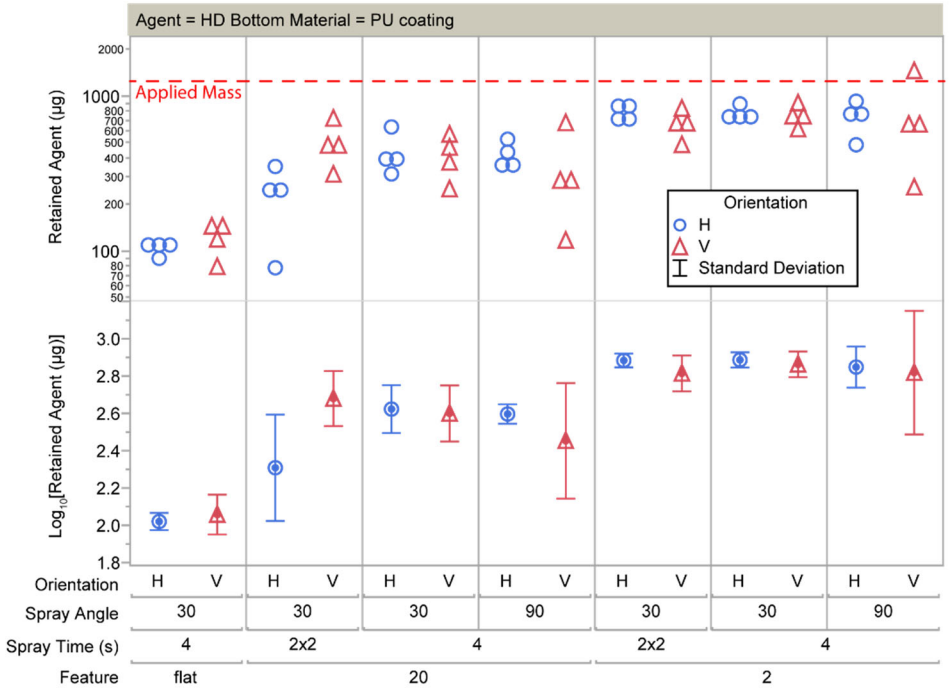


Figure 27. Sensitivity data for HD on PU coating with soapy water spray.

5.2.4 VX Data for Steel

The results of the sensitivity analysis for the varying orientations and treatment conditions for VX on steel are provided in Figure 28. Similar to previous trends, the 2 mil shims retained most of the VX that was applied. There was one out of four replicates for samples in each of the horizontal and vertical orientations that produced a much lower RA. These values may be statistical outliers, but they may also represent a case where the treatment removed some agent from the feature. The 20 mil shims showed greater variability in the results for the horizontal orientation, and the vertical orientation tended to produce greater RA values than the horizontal. For flat panels, there was significantly less variation in the results of the horizontal orientation, though the error bars between orientations overlap.

Like HD on steel, the VX results showed that flat and 20 mil shim panels in the vertical orientation retained more VX than the horizontal, indicating that assessing the vertical orientation would be represent a worst-case. In many of these cases the variability of results is large, producing error bars that span orders of magnitude. This illustrates a challenge that can exist for statistical comparisons and interpretation of decontamination data since variability on some materials is large. In the context of sensitivity analysis, the effects of orientation are somewhat masked by the variability of the results.

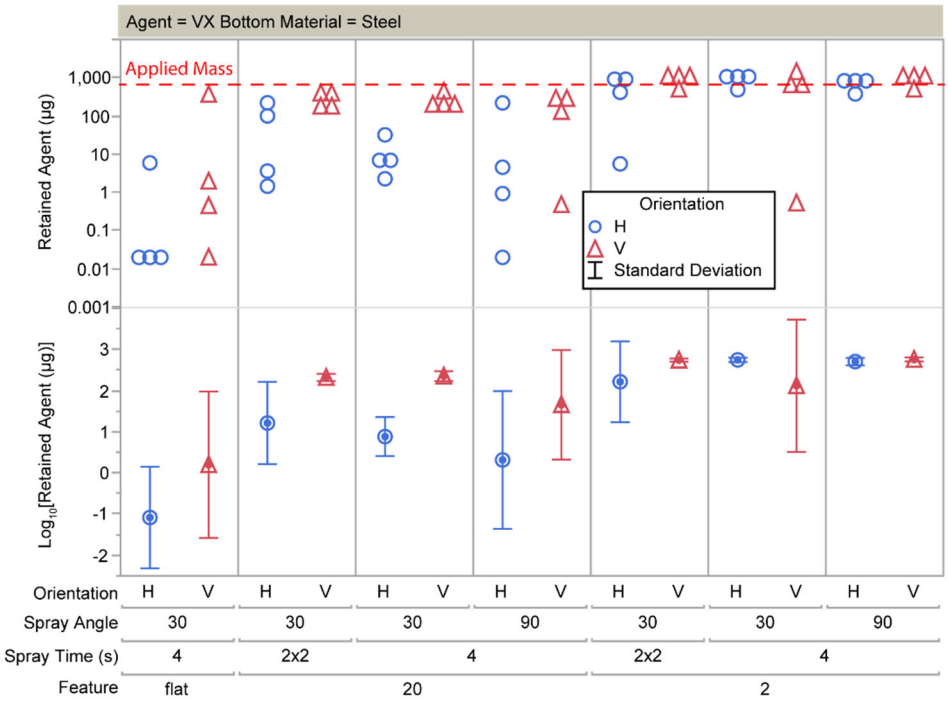


Figure 28. Sensitivity data for VX on steel with soapy water spray.

5.2.5 VX Data for Alkyd Coating

The results of the sensitivity analysis for the varying orientations and treatment conditions for VX on an Alkyd coating are provided in Figure 29. Similar to other results, the retention of VX increased for smaller shim sizes. Visual inspection indicates that orientation nor spray variations significantly influenced the observed RA. The results for the flat and 20 mil shims were similar across all conditions tested. There was also a larger variability in the RA data for the 20 mil shim.

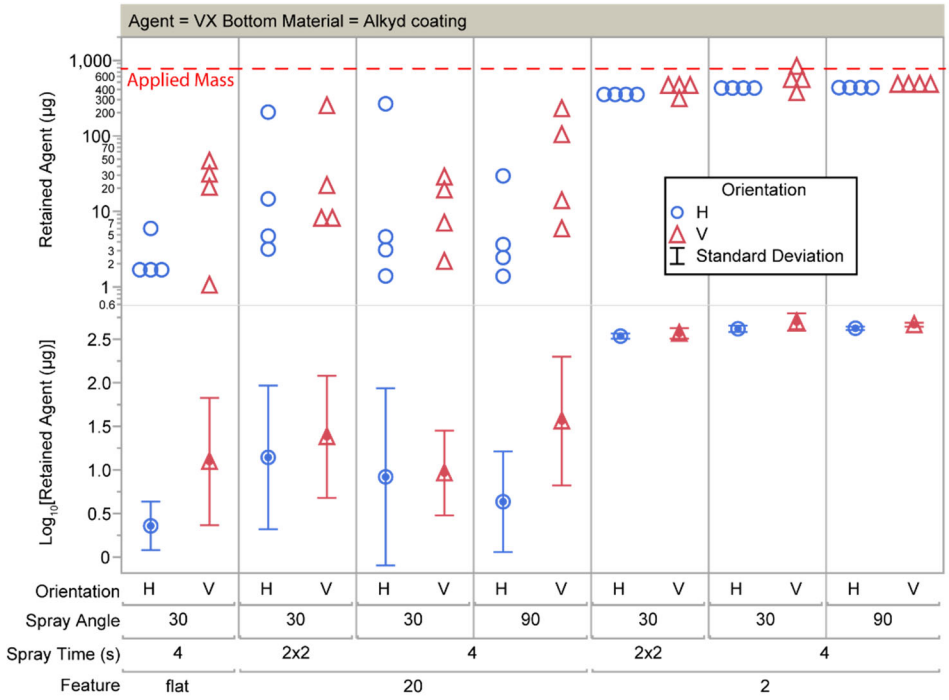


Figure 29. Sensitivity data for VX on Alkyd coating for soapy water spray.

5.2.6 VX Data for PU Coating

The results of the sensitivity analysis for the varying orientations and treatment conditions for VX on a PU coating are provided in Figure 30. The variability of these results is much less than the variability of the steel panels with VX, showing an effect of agent-material interactions. The retention of VX was similar across shim sizes and not significantly different compared to flat panel. In many cases the horizontal condition has a lower mean value than vertical, though the error bars overlap.

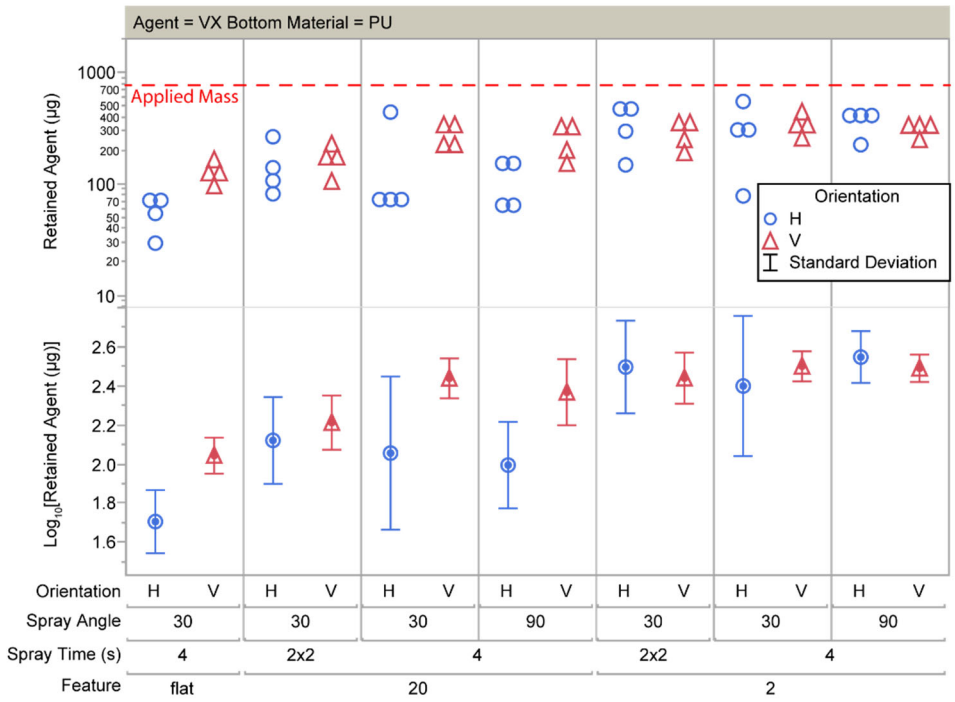


Figure 30. Sensitivity data for VX on PU shim panels. LD is relative to the applied mass.

5.3 Comparison Between Horizontal and Vertical Orientations

The LD in this section is calculated comparing the vertical orientation to the horizontal orientation RA to quantify the effect of orientation. The LD and 95% CI on the difference were calculated according to Appendix B.3. The LD quantifies the order of magnitude difference between the two conditions. If the LD is positive then the horizontal orientation panel retained more agent than the vertical orientation panel, if the LD is negative, then the vertical orientation panel retained more agent. It was postulated that features with LD greater than ±0.4 are sufficiently different (i.e., antilog(0.4) indicates a 2.5× factor difference) that they warrant collection in future test efforts; differences of this magnitude or greater may influence the asset level outputs. These results could be used to identify if the indicated conditions produce a large enough difference to justify testing for the more resource intensive VE and CT testing.

The effect of spray description (i.e., 4 s spray versus 2 × 2s sprays) was found to be insignificant and data were combined across this parameter. The RA was compared between the horizontal and vertical configurations for all the results in this section (Figure 31). The grey shaded region in Figure 31 indicates the range of ± 0.4 LD, where conditions with values outside this range may warrant characterization in future efforts.

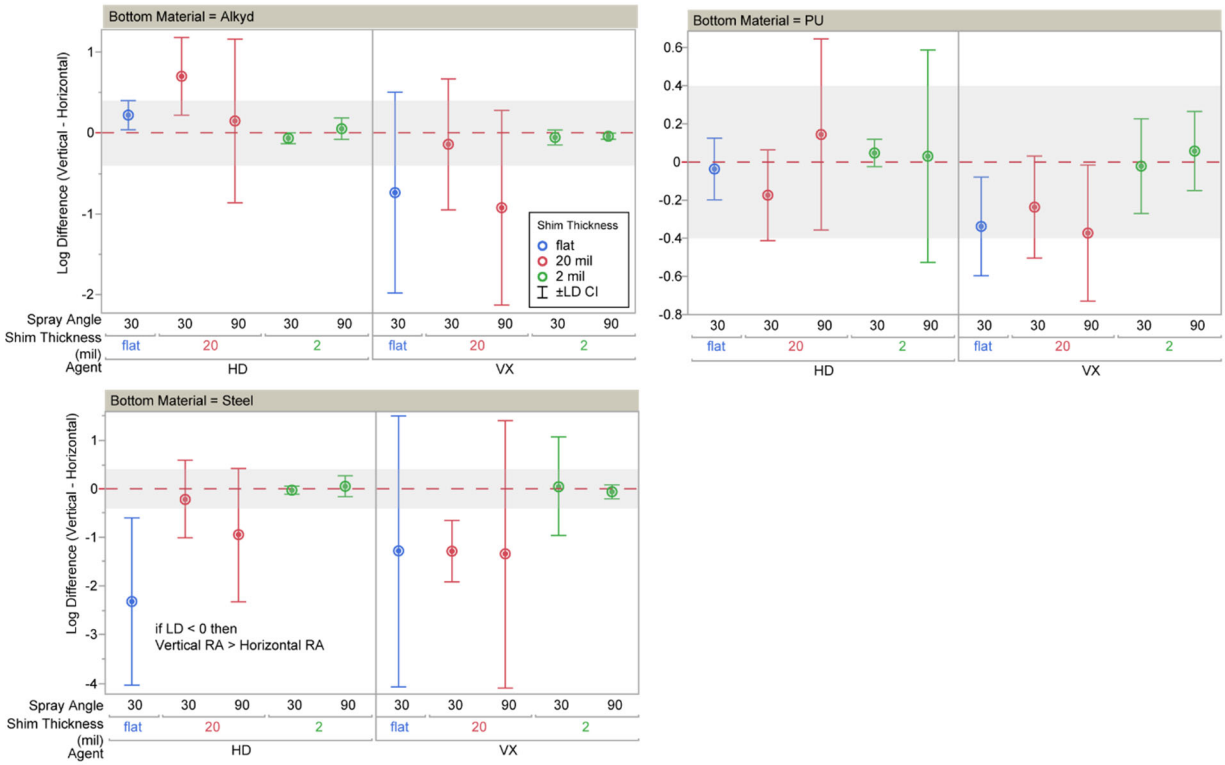


Figure 31. LD comparing RA for the horizontal to vertical orientations for Alkyd, PU, and steel materials with HD and VX. Error bars represent the 95% CI for the LD. Grey shaded region indicates the difference threshold of ± 0.4 LD.

Overall, no significant differences were observed between orientations for the 2 mil shim case; *orientation had no effect on 2 mil shim RA results for this particular spray treatment*. It is anticipated that the capillary forces dominated response and the confined geometry resulted in minimal spray access to the entrained agent independent of the orientation.

For the 20 mil shim there were cases where the LD was more than ± 0.4 between orientations. However, for several of those instances, the CI overlaps zero indicating that the difference is not statistically significant. The two cases with statistically significant results HD on the Alkyd coating with an LD of 0.7 and VX on steel with an LD of -1.3. Sometimes the vertical orientation retained more agent (VX on steel), and other times horizontal orientation retained more agent (HD on Alkyd). This indicates that significant effects greater than ± 0.4 LD (RA different by a factor of antilog(0.4) or 2.5×) may be observed for different orientations in these cases.

For flat panel the HD on steel case was the only condition where there was a statistically significant difference greater than ± 0.4 LD between orientations. The LD of -2.3 (a factor of 200×) is a significant difference, where the geometric mean RA for HD in a vertical orientation was 24.6 µg/drop and horizontal orientation was 0.11 µg/drop (Figure 25). This demonstrates a case where orientation had a significant effect on the RA.

5.4 Summary

A sensitivity analysis was performed to evaluate the effect of orientation and liquid spray parameters for HD and VX on three materials with flat and shim features. Overall, orientation did not have a significant effect on most shim panels when using soapy water spray (other treatment methods such as vaporous technologies may produce different results), but did have some influence on flat panels. It is hypothesized that the capillary forces present in shim panels dominated the response and the confined geometry resulted in minimal spray access to the entrained agent independent of the orientation.

A few cases were observed with more than ± 0.4 LD between orientations. These data do not indicate common factors that can be used to identify what orientations should be tested by default. Some agents spread on some materials, and others (e.g., VX) may have very strong adhesion to many materials that could influence orientation effects on RA. From the hazard mitigation treatment perspective, some technologies may flow off vertical surfaces and reside on horizontal surfaces, while more viscous technologies may stick to vertical surfaces for longer interaction times, vaporous treatments may not exhibit significant effects from orientation. One orientation may not always produce the 'worst case' condition to consider for testing. However, it does not seem practical, due to cost and schedule constraints, to test all conditions in both orientations.

Testing panel orientation for all agent material combinations requires non-trivial resources. Future studies should consider analyzing scaled-up asset level results to identify if the ± 0.4 LD difference at the panel level is too low of a threshold. The ± 0.4 LD range may be small enough that when scaled up, the asset-level results may not show a significant difference if both orientations were used in the calculation compared to testing only one orientation (i.e., the return on investment for expending the resources to test both orientations may not be significant).The threshold value of ± 0.4 LD was an initial estimate to identify a significant effect. Based on lessons learned through this methodology assessment, future variations on a sensitivity analysis are recommended that account for contamination abundance at the asset level that will provide better context towards conditions to prioritize for testing.

Additionally, a proof-of-concept demonstration of the application of statistical models to predict the μ and σ inputs for scale-up calculations is provided in Appendix D. This approach facilitates the use of Design of Experiment tools to enable more efficient testing and a more robust analysis of orientation effects. This section concludes the verification concepts covered as Goal 1 of the report.

6. SOURCE TERM AND SCALE-UP CALCULATIONS

The intent of laboratory-based testing for chemical warfare agent hazard mitigation technologies is to characterize how discrete drops of liquid agents interact with materials to determine whether the technology reduces the quantity of agent retained by the material. It is expected that results from laboratory testing indicate how a technology would perform in an operational environment on actual assets, such as vehicles and other support equipment. As a result, test parameters have previously been configured to directly represent certain aspects of operational conditions. One of these parameters is contamination density (i.e., starting challenge), which specifies the mass of agent per unit of material area (in grams per square meter). Panels have been tested at specific starting challenge levels produced by deposition of discrete liquid agent drops and it has been assumed that the results scale with material area.

It has been demonstrated that starting challenge alone is insufficient for an accurate and conclusive measurement of performance for hazard mitigation technologies.[11] Different drop volumes to generate the same staring challenge on materials may produce significantly different results. This occurs principally because the interaction area between liquid drops and surfaces, which is primarily responsible for RA, varies with droplet volume and is sensitive to the liquid-material surface interactions. This implies that if the laboratory tested agent drop volume does not match the operational drop volumes, the scale-up calculations may not be accurate. A new data analysis approach that accounts for variable liquid drop volume and count was developed and implemented in this methodology. Normalization of the RA mass per drop as a function of the drop volume using an empirical least-squares regression reveals prevailing trends in the assessment of decontamination efficacy that are otherwise not resolvable. This normalization approach captures the effect of drop volume and variable contamination density that is the basis for the scale-up approach to translate from laboratory to operational context. One impact of this approach is that *data acquired on a per drop basis can be used to calculate the asset level response for any contamination density*.

As presented in Section 1.3, one desired output is to represent the post decontamination hazard from an asset that has been randomly contaminated in the operational environment. To achieve this, a new scale-up calculation has been developed that accounts for how each agent drop contributes to the total retained agent mass of the asset; the whole is equal to the sum of the RA per drop. The laboratory generated RA per drop provides a *source term* that is aggregated to represent an asset. This requires that laboratory testing produces the mean and standard deviation of RA on a per drop basis for each material-feature combination found on an asset with which agent interacts. It was identified that the RA data are highly skewed and best described with a log-normal distribution (Appendix B), the ability to describe the variability that may be observed at the asset level cannot be done with standard propagation of uncertainty approaches using log-normal data. Repeated random sampling Monte Carlo (MC) techniques (Appendix F) are better suited to provide the range of response that could be observed at the asset level. Further, this type of MC technique is also ideally suited to enable the random contamination (i.e., random drop volumes interacting with random components of an asset) anticipated for full-scale asset contamination. This work focuses only on the analysis of the highly controlled contamination of an asset in a laboratory. Concepts for how random contamination could be implemented are provided in Appendix C.

The following sections convey how source term functions are defined and implemented in the scale-up calculation as part of the methodology. Three approaches were developed to provide the RA source terms that use different assumptions and require different quantities of data. Each source term calculation approach is analyzed to determine the accuracy for the scale-up calculation.

6.1 Scale-up Calculations

The fundamental premise of the scale-up calculation is that the mass retained by an asset ($M_{Asset,\ Method\ output\ B0}$) is the sum of RA due to each drop that contaminates the various materials and features of the asset. The asset components can be tested for RA in the laboratory with panels. Consider an asset that was contaminated with K drops of agent (e.g., Figure 2). Each agent drop (k) may have a different volume (V_k) and interacts with a specific material feature that generates a specific RA per drop volume ($RA_k(V_k)$). In this case, the RA_k indicates the material-feature associated with drop k. The asset-level RA is calculated as

$$M_{Asset} = \sum_{k=1}^{K} RA_k(V_k) \qquad \text{Eq. 3}$$

Where:

$RA_k(V_k)$	is the laboratory data RA source term as a function of agent drop volume V for the material-feature associated with drop k (µg) for a total number of drops K.
M_{Asset}	total retained agent mass of the asset (µg)

The RA data elements measured in laboratory testing (i.e., the source terms) provide the mass of agent retained by the material-feature for a single drop after the treatment process based on the agent drop volume. This approach was required because efficacy was identified to vary with agent drop volume and count rather than contamination density,[11] and is the reason the laboratory data are normalized on a per drop basis as a function of agent drop volume.

The equation combines the description of how the asset was contaminated with the laboratory data. For example, the laboratory scale assets tested in Section 10 specifically controlled the location and volume of the drops applied to the asset. To conduct the calculation, the drop volume and material-feature source term for each drop k would be identified as shown in Table 32. The features on the asset are not an exact match in gap size with the shim panel, however, the assumption of the shim panel representing the generalized constrained geometry effect only requires the identification of small (2 mil) versus moderate (20 mil) size gaps.

Table 32. Example assignment of drops to specific material-features and drop volumes, based on the Picatinny rail accessory illustrated in Figure 2

Drop ID, k	Description Of Location	Panel Source Material	Panel Source Feature RA_k	Drop Volume (µL) V_k
1	Rail gap	Steel	2 mil shim	0.2
2	Tension knob-screw	Steel	2 mil shim	0.2
3	Flat surface	Steel	Flat Panel	0.2
4	Tension knob-cap	Steel	2 mil shim	0.2
5	Corner	Steel	20 mil shim	0.2

This calculation makes the following assumptions:

1) The RA per drop response is independent of the presence of or interaction with other drops. Whether the drop spreads (and merges with other drops) or remains sessile, the aggregate response of multiple drops is captured by the source term.[11]
2) The response for any complex feature (e.g., threads, seams, closures, gaps, etc.) is represented by the response of the relevant size shim panel.

6.2 Random Sampling Monte Carlo Techniques

The implementation of Eq. 3 using mean value responses for $RA_k(V_k)$ provides an asset-level mean response. However, there is variability associated with the response of each drop associated with measurement uncertainty and treatment process variability. An asset will typically be contaminated by many agent drops, each providing an RA contribution within the variability measured in testing. This requires aggregating the variability of the treatment process for each drop (i.e., propagation of uncertainty). Due to the observed skewed data distributions and log-transform analysis (see Appendix B), techniques using repeated random sampling are ideal to calculate the resulting distributions.[§§] A MC algorithm is used to evaluate Eq. 3 many times, each iteration of Eq. 3 uses a probabilistic per drop RA value for each drop *k,* based on the standard deviation of the laboratory data. This repeated random sampling generates a statistical distribution of M_{Asset} values that describes the asset-level response that may be observed based on the laboratory responses and their variability.

To enable the repeated random sampling for each drop requires inputs that include the mean (μ) and standard deviation (σ) for each RA source term. Appendix B demonstrates that μ and σ calculated on log transformed RA data provide a good description of the observed data distributions. Therefore, the random sampling for each drop will be performed using the log transformed statistics, and then inverse transformed back to the original RA mass units. The $RA_k(V_k)$ of Eq. 3 is randomly sampled as

$$M_{Asset} = \sum_{k=1}^{K} RA_k(V_k) = \sum_{k=1}^{K} antilog_{10}\left(randn(\,) * \sigma_k + \mu_k(V_k)\right) \qquad \text{Eq. 4}$$

Where:

V_k	is the volume of drop *k* (μL)
randn()	is the MATLAB function that provides a random draw from a standard normal distribution (unitless)
σ_k	is the standard deviation of the log-transformed RA result for agent-material-feature-treatment associated with drop *k* ($\log_{10}$(μg))
μ_k	is the mean of the log-transformed RA result for agent-material-feature-treatment associated with drop *k*. ($\log_{10}$(μg))

§§ Standard propagation of experimental uncertainty calculations are appropriate when the data are normal, use of nonlinear log-transforms requires alternate techniques to handle aggregation of data spread, such as MC methods.

The MC algorithm used in this report and a demonstration of random sampling are provided in Appendix F.

As many iterations of this random sampling are performed, the distribution of the output should represent the predicted M_{Asset} distribution based on the laboratory input data and provide an ability to estimate statistical descriptors such as the 95% prediction interval (PI) of the data. For the results presented here, 10,000 iterations were used for all MC calculations. No *a priori* value for a required number of iterations was available, this value was selected to be of sufficient excess size to ensure acceptable sampling and presented acceptable computational load (i.e., personal computers could run the calculations in less than 2 minutes). Future work may identify that significantly fewer iterations are required. Note that MC techniques use random sampling so repeated simulations may provide slightly different results. Repeated runs (not shown) were found to produce small variations in the second significant figure of outputs.

In some cases where the source term has a large standard deviation, it was observed that the randomly sampled mass produced a mass significantly greater than the applied mass for a drop of the specified volume. The MC algorithm (Figure F-1) includes an upper bound limit where the output for a given drop is the applied mass value. In this case, where a randomly selected mass value is greater than the applied mass, the applied mass value is substituted.

Assumptions regarding the Monte Carlo calculations include:

1. The sampled μ and σ are sufficiently accurate representations of the population representing the indicated agent-material-feature-treatment; sample sizes are sufficient to provide accurate statistical descriptions.
2. All data are accurately described using log-transforms.[17]

The use of MC methods can be extended from randomly sampling each drop source term to also include randomly distributing drops across an asset (i.e., randomly contaminating different material-features with each drop having a different volume). A concept of how random contamination may be implemented in future efforts is provided in Appendix C. The volume of the agent drops and the material-feature contaminated can influence the RA source term by orders of magnitude, as a result the random contamination of assets may provide a wide range of potential responses. The methodology assessment focuses on evaluating the accuracy of the scale-up approach, using the highly controlled tested laboratory scale assets, so that approaches such as random drop distributions may be utilized in future efforts.

6.3 Data Normalization and Variable Agent Drop Volume Regression

Previous work has demonstrated that the agent drop volume influences RA and consequently, decontamination efficacy can vary with agent drop volume.[11] The ability to scale-up results to represent an asset requires an equation to provide the post treatment RA mass as a function of the applied agent drop volume. An empirical least squares regression relating the RA mass per agent drop to the agent drop volume[11] is performed according to

$$log_{10}\left(\frac{RA}{n_{tested}}\right) = m\, log_{10}(V) + b \qquad \text{Eq. 5}$$

Where:

m	is the slope for $\log_{10}$ (RA/drop) versus $\log_{10}$(V) analysis
b	is the intercept for $\log_{10}$ (RA/drop) versus $\log_{10}$(V) analysis
RA	is the RA mass for agent-material-treatment-panel (µg)
V	is the agent drop volume (µL)
n_{tested}	is the number of drops applied to a panel in testing (unitless).

Eq. 5 can be solved to provide the inverse-transformed trend of RA mass for an agent drop of volume (V)

$$\mathrm{RA}_{Fit}(V) = antilog_{10}(b) * V^{m} \qquad \text{Eq. 6}$$

Where

$RA_{Fit}(V)$	is the RA mass as a function of agent drop volume (µg)

This empirical relationship (Eq. 5) was derived from observations that drop volume and drop spreading behavior on a material influence measured RA mass. For flat panels, it was empirically observed that for drops that spread, $m \approx 1$, and sessile drops may produce slopes from 0.5–2. This report is the first time this technique is used on agent entrained in shim panel features. Appendix G provides an interpretation of the slope values and their implications on efficacy trends. A log-transform of the RA data also facilitates the statistical analysis of the skewed (i.e., log normal) distributions observed for treatment processes (see Appendix B).

For the data collected in this effort, only one drop was applied per panel, therefore n_{tested} is 1. The regression was conducted using standard linear least squares fit to the log-transformed variables (calculations were conducted using MATLAB 2022b). The adjusted R^2 value is reported as one goodness of fit metric and can be considered a description of the proportion of data variability explained by the regression; a value of 0.5 indicates that 50% of the data variation is explained by the regression. There is no critical value for adjusted R^2 that can be used to indicate a good or poor fit as cases with larger variability may decrease the value. The root mean square error (RMSE) of the fit is provided as an additional goodness of fit parameter and can be thought of as the *standard deviation of the fit*, similar to the standard deviation of a specifically tested agent drop volume.

Assumptions regarding the per drop normalization include:

1. Agent characteristics such as mass purity, viscosity, and liquid surface tension used in testing are representative of the agent that would be encountered in operational context.[1,2,9,10]
2. Decontamination efficacy is not a function of contamination density, but rather agent drop volume and count.[11]

6.4 Source Term Inputs for Monte Carlo Calculations

Three approaches were developed to provide the log-transformed μ and σ values needed for Monte Carlo calculations, each using different assumptions and different quantities of data to calculate the inputs.

6.4.1 Direct Source Terms

The direct source term approach assumes that the drop volume of interest was explicitly tested and the μ and σ values are directly calculated from the log-transformed data. This provides a significant limitation to the approach as only tested drop volumes can be used in the scale-up calculation. Actual operational contamination will produce a continuous distribution of drop volumes making this approach less useful. This approach is reported here as a demonstration of accuracy when no fitting or assumptions about drop volume are included in the calculation. For the data collected in Section 7, 0.2 and 1 μL drops were acquired with five replicates. The log-transformed μ and σ are reported for each of these drop volumes.

6.4.2 Multivolume Fit Source Terms

The multivolume fit calculation technique, referred to as 'fit' throughout the report, uses multi-drop volume laboratory data to fit the per drop regression equation, Eq. 5, to provide the mean value as a function of agent drop volume. Based on the notation used in Eq. 4, the output is tracked with respect to the drop *k* index, indicating the material-feature of interest. The $\mu_k(V)$ input for MC calculations for the agent-material-feature source term is determined using the parameters from the fit

$$\mu_k(V) = m_k \, log_{10}(V) + b_k \qquad \text{Eq. 7}$$

where

μ_k is the mean value output from the fit for agent-material-feature-treatment associated with drop *k* as a function of drop volume ($\log_{10}$(μg))

m_k is the slope parameter from Eq. 5 for agent-material-feature-treatment associated with drop *k*

b_k is the intercept parameter from Eq. 5 for agent-material-feature-treatment associated with drop *k*

Determining σ_k for the regression output requires an analysis of the fit. The RMSE of the fit quantifies both the σ (random error) and the fitting error. If a regression fit is reasonable, then RMSE is an acceptable estimate of σ (random error). As sample size becomes sufficiently large the RMSE will approach the true σ value. When sample sizes are very small the regression fitting error becomes larger and can inflate the RMSE. The RMSE can be considered as the standard deviation of the fit, and for this report, the σ_k input for MC calculations is the RMSE of the fit

$$\sigma_k = RMSE_k \qquad \text{Eq. 8}$$

where

σ_k is the standard deviation of the fit for agent-material-feature-treatment k as a function of drop volume ($\log_{10}(\mu g)$)

The ability to use the fit source terms requires that variable drop volume data was acquired. Slight extrapolation of the results to agent drop volume outside of the test data may be acceptable but should be used with caution. The range of agent drop volumes used in testing should reflect the drop volumes that will be considered for operational scale-up calculations. It is anticipated that M_{asset} calculations using the fit source term will provide best accuracy as the drop volume trend is fit, and more experimental data is utilized. Appendix F provides and interpretation of the regression slope values, their trends across material-features and treatments, and the resulting influence on efficacy.

6.4.3 Indirect Drop Volume Source Terms

The indirect source term uses the laboratory test data for one drop volume to extrapolate the results to all other drop volumes. The extrapolation approach uses the per drop regression (Eq. 5), but assumes that the slope (m) is 1.0. This assumption implies that the efficacy (e.g., log reduction relative to the applied mass) is independent of the agent drop volume. The approach solves for the intercept B in Eq. 5 using the single tested drop volume, then substitutes the value back into Eq. 5, and applies logarithmic identities to produce

$$\mu_{indirect}(V) = \overline{log_{10}\left(\frac{RA}{drop}\right)}_{V_{tested}} + log_{10}\left(\frac{V}{V_{tested}}\right) \quad \text{Eq. 9}$$

where:

$\mu_{indirect}$ is the mean value source term for the agent-material-feature-treatment condition ($\log_{10}$ µg/drop)
V is the agent drop volume for drop k (µL)
V_{tested} is the drop volume used in testing (µL)
$\left(\frac{RA}{drop}\right)_{V_{tested}}$ is the measured RA/drop response for the tested drop volume (µg/drop)

The overbar notation indicates the arithmetic mean of the $\log_{10}$ transformed responses. A slope of 1.0 assumes that the RA is directly proportional to the mass of the drop applied, this is the trend observed for the applied mass measured by the DCS. This also has the implication that efficacy (e.g., LD compared to the applied mass) is constant for all drop volumes.

Using this approach, only one drop volume is tested in the laboratory, which requires fewer resources (e.g., 5 replicates) compared to the regression fit that may use 6–16 replicates per agent-material-feature-treatment condition. The standard deviation MC input for this approach, $\sigma_{indirect,}$ is calculated as the standard deviation of the $\log_{10}$ transformed RA/drop results, and is assumed to be constant for all V.

It is anticipated that M_{Asset} calculations may provide less accuracy with this extrapolation as the slope term may actually deviate from 1.0. This approach provides a cost-accuracy balance of requiring less data to provide a variable agent drop volume capability with potentially less accuracy.

6.5 Quantifying Efficacy with Log Difference

Previous work demonstrated that the efficacy for the same starting challenge generated with different drop volumes could produce different results.[11] This indicated that efficacy can vary with agent drop volume. Where the base 10 LD can be calculated for two specifically tested conditions (Appendix B.3), the LD can also be calculated for variable drop volume results to show how efficacy changes with agent drop volume. One efficacy metric often used is the log difference from the applied mass. In this case the applied mass as a function of agent drop volume is characterize by the DCS ($RA_{Fit,DCS}(V)$, from Eq. 6). The LD of a treatment for a given material-feature ($RA_{Fit,X}(V)$) as a function of agent drop volume can be calculated as

$$LD(V) = log_{10}\left(RA_{Fit,DCS}(V)\right) - log_{10}\left(RA_{Fit,X}(V)\right) \quad \text{Eq. 10}$$

Or equivalently using the MC input parameters already on a log scale

$$LD(V) = \mu_{DCS}(V) - \mu_X(V) \quad \text{Eq. 11}$$

The efficacy will change with agent drop volume if the regression slope *m* deviates from 1.0. Examples of this and the implications of the slope value on efficacy are discussed in Appendix G.

6.6 Confidence, Prediction, and Tolerance Intervals

For the M_{Asset} calculations, the outputs are assumed to follow log-normal distributions. The accuracy of the calculations will focus on the ability to predict similar mean values to the experimentally measured quantities. However, for the end data use, the upper bound of M_{Asset} may be of interest (e.g., for 95% of cases, the asset will be less than a specific value). This type of interval requires a one-sided tolerance interval (TI).[19] Recall, a confidence interval (CI) describes the uncertainty of a parameter like the population mean, a prediction interval (PI) describes the uncertainty of next/future observations, whereas a tolerance interval indicates the range where a proportion of the population likely exists. Tolerance intervals account for sampling error more so than prediction intervals. A one-sided (upper bound) TI is calculated as

$$TI = \mu + k_1\sigma \quad \text{Eq. 12}$$

where

$$k_1 = \frac{z_p + \sqrt{z_p^2 - ab}}{a}$$

$$a = 1 - \frac{z_\alpha^2}{2(n-1)} \quad \text{Eq. 13}$$

$$b = z_p^2 - \frac{z_\alpha^2}{n}$$

This treatment of the TI assumes the data are normally distributed. Based on previous arguments, a log transformation of M_{Asset} is used to generate the μ and σ used to

calculate the TI. If it is desired to have 95% confidence (α) that 95% of all future samples (population percentage, p) are below a threshold, the Z values can be selected from Table 33 to calculate the k_1 value using Eq. 13. The resulting upper-bound TI of the data can be calculated using Eq. 12, to determine if the results are below a requirement level, such as a health-based requirement. As an example, to calculate the k_1 value, using a 95% confidence (α) level for 95% of the population (p), the Z parameters would be Z_α is 1.645, and Z_p is 1.645. The corresponding k_1 value for a range of sample sizes is provided in Table 34. For the calculations produced for asset level outputs later in this report, 10,000 iterations of MC sampling were used.

Table 33. Z values for one-sided tolerance intervals, Z_p is used for probability levels the Z_α value is used for confidence levels

p, α	Z_x
0.999	3.09
0.995	2.576
0.99	2.326
0.975	1.96
0.95	1.645
0.9	1.282

Table 34. Tolerance interval $k_1$values for a range of sample sizes (n) for 95% percent confidence (α) for 95% of the population (p)

a	b	n	k_1
0.662	2.165	5	4.191
0.774	2.319	7	3.355
0.850	2.435	10	2.875
0.929	2.571	20	2.379
0.986	2.679	100	1.924
0.999	2.703	1,000	1.727
1.000	2.706	10,000	1.670

In addition to the TI level outputs, the geometric mean, 95% prediction interval (PI), are calculated from the mean of the $\log_{10}$(RA) for each case. The PI are calculated using the $\log_{10}$ transformed μ and σ of the M_{Asset} output as

$$geometric\ mean = \ antilog_{10}(\mu)$$
$$PI = \ antilog_{10}(\mu \pm z\sigma) \qquad \text{Eq. 14}$$

Where:

PI is the upper and lower prediction interval

z is 1.96 for a two-sided 95% PI

In cases where the sample size is small ($n < 30$), the appropriate $t_{(1-\alpha,df)}$ distribution is used in place of *z*. The resulting PI equation is

$$PI = antilog_{10}\left(\mu \pm t_{1-\alpha,df}\sigma\sqrt{1-1/n}\right) \quad \text{Eq. 15}$$

This occurs when calculating the PI for experimentally measured conditions such as the asset testing using seven replicates. Example t values are provided in Table 35.

Table 35. 95% confidence *t* values used for calculating PI

n	$t_{1-\alpha,\,df}$	$t_{1-\alpha,df}\sqrt{1-1/n}$
4	3.182	3.558
5	2.776	3.041
6	2.571	2.777
7	2.447	2.616
10	2.262	2.373
20	2.086	2.033

The interpretation of statistical moments for log-normal distributions is different than for normal distributions where the mode, median, and mean often coincide. For a log-normal distribution the mode (most frequent value), the median (50th percentile, where half of the distribution is below and half of the distribution is above the value), and the mean (a description of central tendency) are different values as illustrated in Figure 32. For this methodology the geometric mean (i.e., the median) is reported to represent the 50th percentile of the distribution. The μ and σ values are reported for all data, which enables the calculation of any of the desired statistical moments. Figure 32 shows the corresponding 95% prediction interval and TI for the distribution.

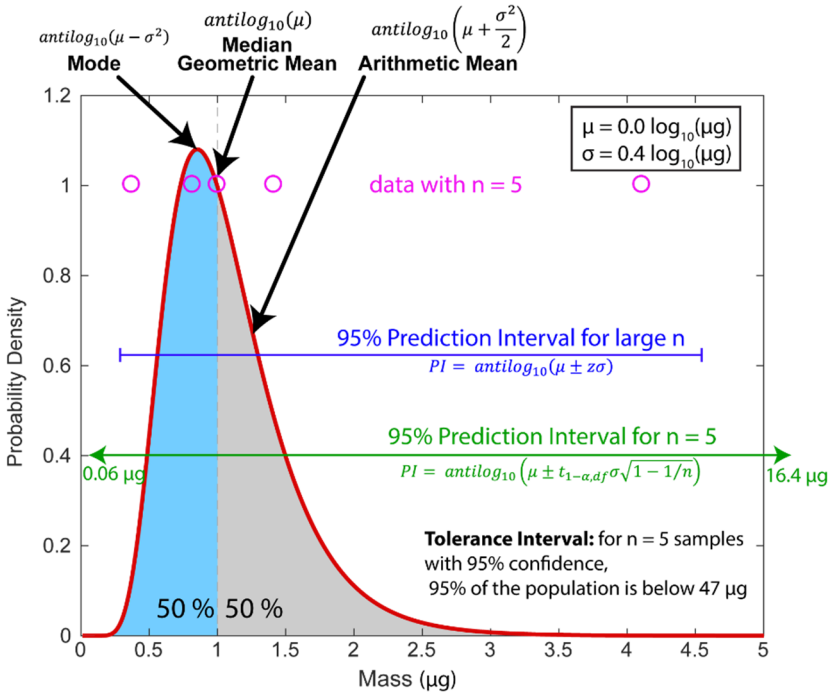


Figure 32. Statistical moments and intervals for a log-normal distribution, pink circles demonstrate an example dataset of five replicates with the indicated μ and σ.

6.7 Relative Contributions

The scale-up calculation enables the ability to quantify the relative contributions of agent contamination from specific features. This kind of information can be used to identify highly contributing sources and optimize the technology for improved performance on these features, or to train personnel to focus mitigation efforts on specific components of assets. The assessment of relative contributions of each source are described as output B_1 of the methodology (Figure A-1). The results presented for shim features demonstrate significant differences in RA compared the flat panel results. For example, if a 2 mil shim produces RA values 100 times that of a flat panel, then one drop interacting with this feature type may present as much exposure risk as 100 drops on a flat panel.

Relative contributions of each feature type are calculated to identify what features are contributing to the calculated M_{Asset} values. The relative contributions of each feature were tracked as

$$C_i = \frac{M_i}{M_{Asset,calculated}} \qquad \text{Eq. 16}$$

Where:

C_i	is the relative contribution of agent-material feature *i*
M_i	is the total mass of RA associated with agent-material feature *i* from per drop RA panel data

$M_{Asset,calculated}$ is the calculated total mass of RA associated with the entire asset based on per drop RA panel data

The C_i results are calculated for each iteration of the MC simulation because each simulation is the result of the sum of randomly sampled drops. The relative contributions can vary significantly based on the random sampling.

6.8 Assessment of Scale-up Accuracy

The accuracy of the asset level RA output is determined by the ratio, R, of the calculated mass, $M_{asset,calculated}$, based on per drop RA responses from panels to the measured RA, $M_{asset,measured}$, for the same number of drops as defined by

$$R = \frac{M_{Asset,calculated}}{M_{Asset,measured}} \quad \text{Eq. 17}$$

Divergence from the ideal value of 1.0 is a quantitative indication of the accuracy of the calculation. *Ultimately for the methodology assessment, it is important to characterize R so the evaluator can understand if the output is a safe-sided over prediction or an underestimate. Even if R is outside of the goal range of 0.1–10, it can be used as an uncertainty factor for MPD in the risk assessment process.*

The mean values for $M_{Asset,measured}$ and $M_{Asset,calculated}$ will be calculated for the log-transformed mass and inverse transformed back to mass units (i.e., the geometric mean will be used to calculate the mass values). Additionally, the 95% TI (or equivalent) for the measured results will be compared to the 95% percentile distribution of the output. The ratio and distribution comparison will be calculated for the three source term calculation approaches described in Section 0.

The ratio R captures *factor* difference if the prediction over (R > 1.0) or underestimated (R < 1.0) the measured value. In assessing the overall accuracy, it is beneficial to consider what is termed the $R_{Absolute}$

$$R_{Absolute} = \begin{cases} R, R \geq 1 \\ 1/R, R < 1 \end{cases} \quad \text{Eq. 18}$$

$$R_{Absolute} = antilog\left(abs\left(log_{10}(R)\right)\right) \quad \text{Eq. 19}$$

Either equation provides a similar $R_{Absolute}$ value. If R had instances of over or underestimating the measured value the summary statistics would tend towards a value of 1. $R_{Absolute}$ is always greater than 1 and the summary statistics describe the overall accuracy of the source term calculation; lower values (i.e., 1.0) indicate better accuracy. This is much like root-mean-square error in statistics where deviations are squared to handle positive and negative values.

The criterion for the accuracy of the calculation are considered acceptable if R is within 0.1–10× (i.e., $R_{Absolute} < 10$). This range may seem wide, however, the lab panel results (per drop) across materials and features can vary by multiple orders of magnitude, and the variability of the results across agent-material-feature-treatment combinations can be a standard deviation of 0.1–1.0 $\log_{10}$ units (i.e., factor of 10^1=10×). For the dynamic range of inputs and

magnitude of variability (i.e., some drops may disproportionally contribute to the signal), the aggregation of per drop contributions approximates the RA response for the tested asset.

6.9 Limit of Quantitation Considerations

One of the end uses of the methodology output is to compare the results to health-based requirements. A question arises as to what analytical sensitivity is required to enable the assessment to 'low level' toxicity values. The asset scale-up approach uses the source term (i.e., laboratory data) combined with an asset and contamination vignette to facilitate the exposure assessment. Therefore, the required sensitivity is a combination of laboratory chromatography sensitivity *and the vignette being considered*.

The data collected in this effort present cases where some materials are decontaminated to below the limit of quantitation (LOQ). The HD LOQ used here corresponds to approximately 10 ng/drop, which is a very small quantity of agent and represents the highest laboratory sensitivity currently achievable. If assets (e.g., a vehicle) were uniformly contaminated to 10 g/m^2 contamination density that could correspond to 200,000 drops. Assuming each drop was decontaminated to the LOQ, the asset RA would translate to a mass of 4 mg of agent distributed over the asset, if it were reasonable to assume all mass from the full asset could contribute to an exposure dose. In the case of contact transfer, as assets become large, such as vehicles, this assumption may be significantly overprotective.

In the case of highly effective treatment processes, non-detection results for source terms will be observed. For the MC scale-up calculations used here, the μ value *is used as reported* at the LOQ or LOD level. *** If all samples provide LOQ or LOD levels the resulting σ is zero. The MC calculations can execute with a zero value σ, however, it produces a point function where all random sampling produces exactly μ. This can influence the distribution of the MC outputs. In this work, if a σ is calculated as zero, then a lower bound $\sigma = 0.05 \log_{10}(\mu g)$ is substituted. As discussed in Appendix B, this value corresponds to a CV equivalent of about 10%. This value is derived from the repeatability analysis for DCS in Section 8.5.

7. DECONTAMINATION TREATMENT SOURCE TERM DATA

Previous method validation efforts, have characterized repeatability and reproducibility of treatment processes and RA analysis methods for a range of agent-material combinations including agents GD, HD, VX, and methyl salicylate, and materials including impermeable metals, glass, polymers, and various coatings.[6-8] Significant effort was involved in evaluating day-to-day and lab-to-lab variations. It is suggested that these previous examples have thoroughly demonstrated the stability and reproducibility of the treatment procedures and RA analysis method, as well as have shown that the reproducibility varies for each agent-material-treatment-temperature condition. In these studies, it was observed that if liquid agent is present when the treatment process occurs, the variability tends to be higher. Additionally, impermeable materials (e.g., glass or metal) tend to have low RA values but high relative variability (e.g., CV > 100%), which is attributed to liquid agent retention in scratches and grooves of the material

*** Other practices such as using ½ of the LOD value can be used, if desired and documented.

surface. Overall, the reproducibility tends to range from 6–58% (CV) across agent-material pairs.[6,7] As a result, only one agent, HD, is used for the reproducibility testing here.

The variability observed in an RA measurement after a treatment process will stem from both the details of the agent distribution in or on the material as well as influence of the treatment process. The methodology was designed to allow treatment configurations and application techniques to be defined by the technology developer or tester. For example, if the spray duration, pressure, or angle were altered for the soapy water spray, a different CV may be observed. It is thought that the variability of RA results after a treatment process primarily reflects the variability of the treatment process being evaluated.

For the asset level calculations conducted in Section 11, it is assumed that the variability of RA data observed in panel data represents the variability that is observed when decontamination is conducted on the asset; the standard deviation is one of the inputs to the calculation. The data from Section 4 demonstrated that the agent can be delivered, extracted, and quantified with an acceptable high degree of repeatability (e.g., average CV of 8%) for most agents and materials. It is recognized that this measurement reflects the quantity of mass measured and does not account for variations in the distribution of agent on or in the material (i.e., variable spreading or flow into the shim panel gap that are not quantified by the method). The test method (treatment and analysis procedures) provides a characterization of the effect of the treatment process (i.e., mean value and the variability of the RA measurement).

For each agent-material-feature-treatment condition there are several factors to investigate from the data and analysis. 1) What is the magnitude and variability of the RA data, 2) how well do the source term calculation techniques describe the experimental data, and 3) What is the efficacy of the treatment process as a function of agent drop volume?

7.1 Test Description

This following test design produces data that will contribute to methodology assessment goals 1.2, 1.3, 2, and 3. The validation test conditions are indicated in Table 36. As part of method validation, the reproducibility (i.e., within test day variability) of the treatment process on flat panels and shim panels will be evaluated. Within-test-day reproducibility will be presented in the form of CV (e.g., standard deviation / arithmetic mean). The contamination settings are designed to enable assessment of one drop volume, two drop volumes, and variable drop volume contamination configurations (Table 37). In this case, there are five replicates of 0.2 and 1 μL drops, and a sampling of other drop volumes to enable the per drop volume regression to evaluate validation goal 2. The sampling of 5 replicates per drop volume is based on previous testing; however, for this variable drop volume configuration a total of 16 samples per condition have been collected. This sampling will enable the panel data to be analyzed according to the three approaches identified in Section 6.3.

Table 36. Test parameters to characterize the reproducibility of the RA method on different panel types for two different decontamination treatments.

Parameter	Conditions	Comments
Materials	Steel, PU coating, Alkyd coating, Silicone	Representative materials from assets to be used in calculations including impermeable, wetting, and non-spreading
Agent	HD	
Age Time	60 min	Typical contamination duration.
Panel variations	Flat Panel, 0.002 , 0.020 inch	Flat panels are 'bottom washers' with holes in the middle
Drop count ×volume (µL)	matrix in Table 37	All drops are delivered with a syringe pump
Contamination Temperature	21 °C	Standard ambient temperature
Treatment	1) soapy water spray followed by water rinse (30 min residence) 2) CHAD treatment (2 h at 76 °C, 95% RH)	Soapy water is sprayed with the Preval sprayer for about 4 s from the side of the panel at about 30°.
Analysis Method	RA test	Output includes RA for each panel and DCS per test session.
DCS	3 replicates per drop volume applied	Due to number of drop volumes used this consumes significant resources, and previous efforts have demonstrated accuracy and reproducibility. DCS confirms mass of agent applied to each panel
Replication	16, see Table 37	Replication designed to enable methodology assessment goals 2.1, 2.2, and 2.3.

Table 37. Contamination conditions for validation testing per agent-material-treatment.

Drop Vol (µL)	Number of drops	Total Volume Applied (µL)	HD Mass Applied (mg)	Equivalent Contamination Density (g/m^2) on 2-inch Diameter Flat Panel*	Replication
0.2	1	0.2	0.216	0.108	5
0.6	1	0.6	0.648	0.324	2
1	1	1	1.08	0.54	5
2	1	2	2.16	1.08	2
4	1	4	4.32	2.16	2

*Contamination density is provided here as context to previous methodology test conditions.

7.1.1 Material Selection

Table 36 provides information regarding the test materials used. For the asset level calculation demonstration in Section 11, the steel data collected here will be used as the source terms to represent the Multi-tool and Picatinny rail accessory.

7.1.2 Treatment Processes

A liquid treatment process and a vaporous treatment process are used here to demonstrate how hazard mitigation approaches with and without a dependence on liquid fluid flow perform on the selected assets/materials using flat and shim panels. The details of the

treatment processes were provided in Section 2.3. Note that the CHAD treatment condition was significantly shortened to 2 h to ensure measurable signal; longer treatment durations are highly effective and produce below detection signals that do not enable variability analysis.

7.1.3 Criteria for Validation

Regarding Validation Goal 1.3, these data produce results for multiple agent-material-feature-treatment combinations. The primary data element is the RA result, normalized per drop (RA/drop). There is sufficient replication (i.e., 5 replicates) to indicate the variability for 0.2 µL and 1 µL drops in the form of a CV. Additionally the RMSE of the fit can be produced to show a relative variability across the variable volume regression. The CV may significantly exceed 100%, and does not indicate a poor methodology, rather the treatment process produces a wide range of responses. Previous multi-laboratory efforts demonstrated repeatability standard deviations between 15–74% and reproducibility standard deviations of 42–89%, depending on agent and timing conditions used (see Appendix A). DCS will have a mean value within 20% of the target delivered mass.

7.2 Soapy Water Treatment

The results for these conditions were obtained over three test sessions in tests CAP035–CAP037.

7.2.1 DCS Results

The DCS data for each test is provided in Table 38. There are a few conditions below the 20% of nominal delivered mass (< 0.80). Nominal mass assumes the liquid density of 1.268 g/mL and a HD mass purity of 0.98. Based on these data, the 4 µL setting for the syringe pump delivered slightly less mass than anticipated. Most decontamination efficacy results demonstrate order of magnitude reductions such that the ~2–5% less than anticipated delivered mass should have a minimal impact on results.

Table 38. DCS statistics for each test for soapy water treatment

Test ID	Agent Drop Vol (µL)	DCS (µg)			Nominal Mass (µg)	DCS / Nominal
		N	Mean	Std Dev		
CAP035	0.2	4	200.8	21.6	248.9	0.81
	0.6	3	665.9	13.9	746.8	0.89
	1	4	1000.2	151.6	1244.6	0.80
	2	3	2241.6	26.0	2489.2	0.90
	4	3	3744.2	117.5	4978.4	0.75
CAP036	0.2	3	226.9	9.1	248.9	0.91
	0.6	3	666.9	80.6	746.8	0.89
	1	4	1209.8	285.1	1244.6	0.97
	2	3	2087.7	165.0	2489.2	0.84
	4	5	4382.1	309.7	4978.4	0.88
CAP037	0.2	3	210.0	69.5	248.9	0.84
	0.6	3	597.0	20.0	746.8	0.80
	1	3	1018.9	29.0	1244.6	0.82
	2	3	1930.5	115.8	2489.2	0.78
	4	3	3737.7	151.1	4978.4	0.75

Red colored text indicates results out of acceptance criterion (1 ± 0.2)

7.2.2 Steel

The results for HD on Steel with Soapy Water Spray treatment are provided in Figure 33 with fits provided using the variable drop log transformed RA responses. Note that the results for this agent-material-treatment combination span five orders of magnitude. Additionally, impermeable materials like bare metals or glass tend to produce higher variability RA data.

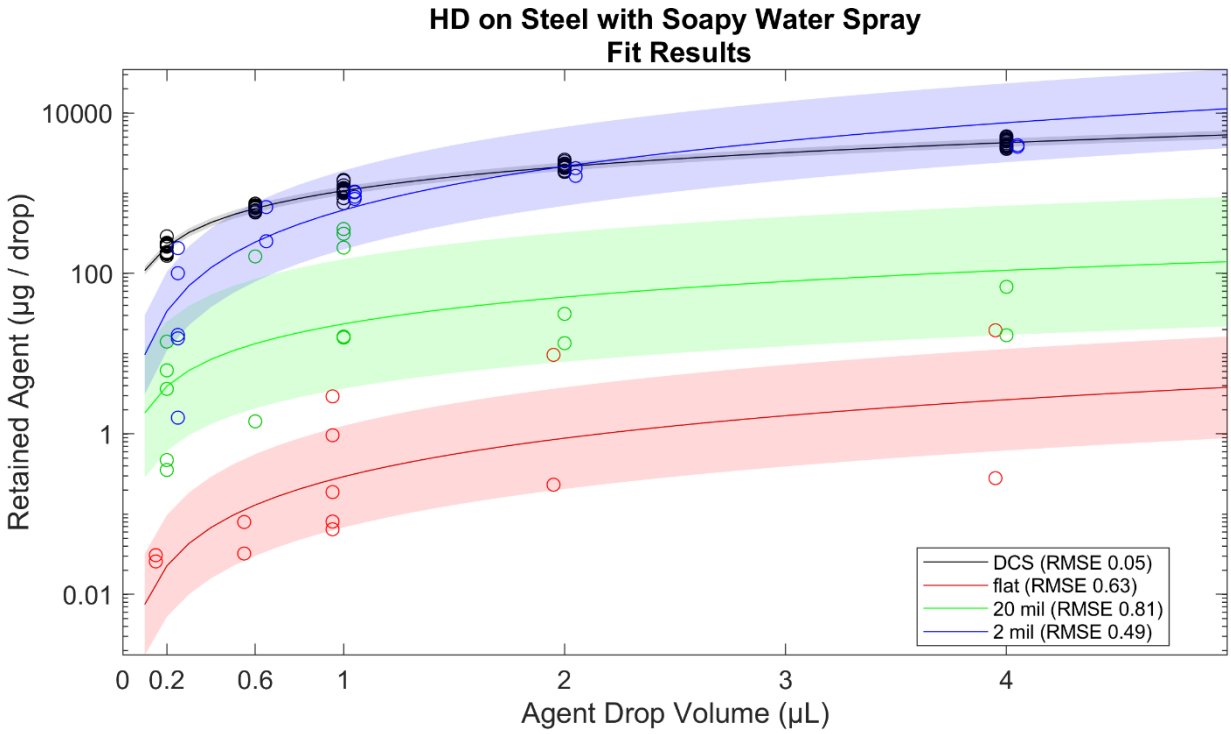


Figure 33. Variable agent drop volume results for HD on Steel with Soapy Water Spray treatment. The shaded region represents antilog($\mu_{fit}(V)$ ± RMSE). Data values are scattered in the x axis to facilitate visualization.

The variable agent drop volume regression described in Section 6.3 was used to fit the data. The regression results for all features (i.e., panel configurations of flat, 20 mil, 2 mil, and the DCS results) are provided in Table 39. Each regression uses the 16 samples generated with the drop distribution in Table 37, providing 15 degrees of freedom for each regression. The adjusted R^2 value is reported as one goodness of fit metric. The adjusted R^2 value can be considered a description of the proportion of data variability explained by the regression; a value of 0.5 indicates that 50% of the data variation is explained by the regression. There is no critical value for adjusted R^2 that can be used to indicate a good or poor fit as cases with larger variability may decrease the value. The shaded region in each graph corresponds to the 95% confidence interval (CI) for the fit.

Table 39. Variable agent drop volume regression results for HD on Steel with Soapy Water Spray treatment.

Treatment	Material	Feature	Slope, m (log_{10}[µg/µL])	Intercept, b (log_{10}[µg])	RMSE, (log_{10}[µg])	Adj. R^2
Soapy Water Spray	Steel	DCS	0.995	3.031	0.052	0.987
		flat	1.592	-0.533	0.633	0.559
		20	1.110	1.371	0.806	0.250
		2	1.805	2.791	0.491	0.736

The RMSE is provided as an estimate of the standard deviation of the data across all drop volumes. Smaller RMSE indicates a more reproducible RA output, Appendix B provides an overview of the characteristics of the log-transformation where the RMSE provides a multiplicative description of the variability (times or divided by) rather than additive (plus or minus). The inverse transformed RMSE, antilog(RMSE), is the multiplicative standard deviation of the data; the DCS data are typically within a factor of antilog(0.05) = 1.12× of the fit, the flat panel data are typically within antilog(0.63) = 4.2× of the fit. The RMSE is used as the σ term supplied as an input to the MC calculations for the 'fit' technique.

Overall, the fit regressions visually trend with the data. As shown in Table 37, The adjusted R^2 results span from 0.25–0.98. The lowest adjusted R^2 value was for the 20 mil shim feature, however, this does not necessarily indicate a poor fit, but rather that the fit relating RA/drop to the drop volume accounts for 25% of the variability of the data, the other 75% of the variability was not captured by the regression and could be a result of the variability of the RA data. Recall that the RMSE value captures the combination of the fitting error and random error (i.e., contamination and treatment process variability observed in the RA data). Investigation of the variability for the 1 µL drop volume shows CV values > 80% (Table 40). The lower adjusted R^2 values are likely a result of the variability of agent distribution and the treatment condition. Note that the standard deviation for individual drop volumes for the log_{10}(RA/drop) correlates with the RMSE for the model fits.

The 0.2 and 1 µL drop conditions were collected with 5 replicates enabling a statistical analysis (Table 40). The statistical summaries are reported for the log transformed RA/drop results and the corresponding inverse transforms as calculated using Eq. 14. The lower and upper bound were calculated with Eq. 15, using the multiplier ($\boldsymbol{t_{1-\alpha,df}\sqrt{1-1/n}}$) of 3.041 for n=5 samples (from Table 35).

Table 40. Statistical summary for HD on Steel with Soapy Water Spray treatment for 0.2 and 1 µL agent drops

Feature	Agent Drop Vol (µL)	n	$\log_{10}$(RA [µg/drop])			Inverse Transform (µg/drop)		
			µ	σ	CV_{log} (%)	Geo. Mean	Lower Bound PI	Upper Bound PI
DCS	0.2	19	2.33	0.06	9.1%	213	161	283
	1	20	3.02	0.07	10.6%	1,047	754	1,453
flat	0.2	5	-1.57	0.03	5.3%	0.03	0.02	0.03
	1	5	-0.51	0.72	149.9%	0.31	0.00	47
20 mil	0.2	5	0.35	0.70	145.6%	2.24	0.02	301
	1	5	1.96	0.69	141.2%	91	0.73	11,436
2 mil	0.2	5	1.39	0.82	193.7%	24	0.08	7,649
	1	5	2.99	0.04	6.2%	977	738	1,293

The fit for the 2 mil data in Figure 33 exceeds the RA/drop output for DCS results; the fit indicates results greater than the applied mass. The laboratory acquired data shows that the 2 mil results were similar to the DCS results. When the scale-up calculation is performed using the 2 mil fit, the reported output response will be limited (i.e., substituted) to the applied mass as described in Section 6.2.

The 0.2 and 1 µL agent drop volume results were used for the indirect fit extrapolation (Eq. 9), shown in Figure 34. The intended use of the indirect fit is when only one agent drop volume is tested and alternate drop volumes are not available. In the methodology assessment, multiple drop volumes were used to calculate the RMSE for the fit. The RMSE values are comparable across all fit techniques for a given data set, and are used to compare the accuracy between the fit and indirect approaches (lower RMSE indicates a more accurate fit). For example, using the 0.2 µL results for flat panel data provided $\sigma = 0.03$ $\log_{10}(\mu g)$, but the RMSE was 0.7 $\log_{10}(\mu g)$, indicating a potentially significant fitting error. Whereas the 1 µL flat panel results produced $\sigma = 0.72$ $\log_{10}(\mu g)$, and the RMSE was 0.66 $\log_{10}(\mu g)$, indicating a likely smaller fitting error and that the extrapolated results have a similar random error. A more in-depth comparison of the fit techniques and their advantages and disadvantages is provided in Appendix G. The MC input using indirect fits is based on the σ value for the tested data.

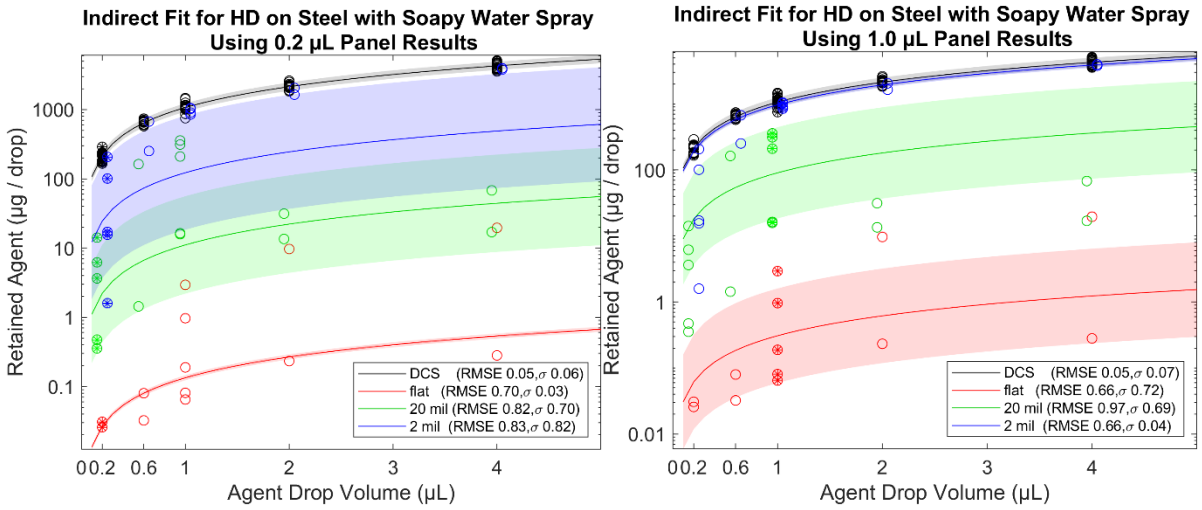


Figure 34. Inverse transformed indirect fits for HD on steel with soapy water spray for 0.2 µL drops (left) and 1 µL drops (right). Asterisks (*) indicate the specific data values used to produce each indirect fit. The RMSE of the indirect fit is indicated in the legend, the σ indicates the log transformed standard deviation of the specific drop volume data used (Table 40). The shaded region corresponds to antilog($\mu_{Indirect}(V) \pm \sigma$).

A comparison of the RMSE values across the fitting techniques is provided in Table 41. A smaller RMSE value indicates a better fit, with the limit that the RMSE cannot be lower than the inherent variability of the data. For reference the σ value for the five replicates of 1 µL data is provided in the table. Overall, the fit technique tends to provide the lower RMSE values that tend to be of similar magnitude as the σ value. The slope of the fit regression, *m*, is provided in the table. The indirect method assumes *m* equals 1, it is expected that when *m* diverges from 1.0 the RMSE of the indirect fits would increase. The RMSE represents the average error for all data, whereas the σ value represents only the single condition. The 2 mil data for 1 µL produced a small σ of 0.04 $\log_{10}$(µg), however the overall RMSE was larger for all fit techniques. The RMSE for the indirect technique using 1 µL drops often provides a lower RMSE than the indirect technique using 0.2 µL. A comparison of fit techniques using all material-feature-treatment combinations is provided in Appendix G.

Table 41. Comparison of RMSE (in $\log_{10}$(µg) units) for each of the source term fitting techniques for HD on steel with soapy water spray

Treatment	Material	Feature	Indirect, V=0.2 µL	Indirect, V=1 µL	Fit		RA/drop, V=1 µL
			RMSE	RMSE	m	RMSE	σ
Soapy Water Spray	Steel	DCS	0.05	0.05	0.99	0.05	0.07
		flat	0.70	0.66	1.59	0.63	0.72
		20 mil	0.82	0.97	1.11	0.81	0.69
		2 mil	0.83	0.66	1.81	0.49	0.04

LD was not included as a metric or specific goal for the methodology assessment, but is a common method output (Output A, Appendix Table A-1) used to quantify efficacy for a specific agent-material-feature-treatment process and is demonstrated here. The LD of the

treatment process (calculated using Eq. 10) using each fit type is provided in Figure 35, using the DCS fit as the reference for the applied mass. The LD of the fit model may vary with drop volume if the slope of the fit regression deviates from 1. If the fit slope is greater than 1 (e.g., the 2 mil shim slope is 1.8), then efficacy will *decrease* for larger drop volumes. If the regression slope is less than 1 (cases are observed in later data), then efficacy will *increase* for larger drop volumes. If the slope is exactly 1.0, as assumed by the indirect fit, then the efficacy is constant for all drop volumes. The LD was also calculated using discrete agent drop volumes directly calculated from the laboratory data as $\mu_{DCS} - \mu_{treatment}$ for each drop volume. Only the 0.2 and 1 µL data had sufficient replication to calculate the CI (error bar), as defined in Appendix B.3.

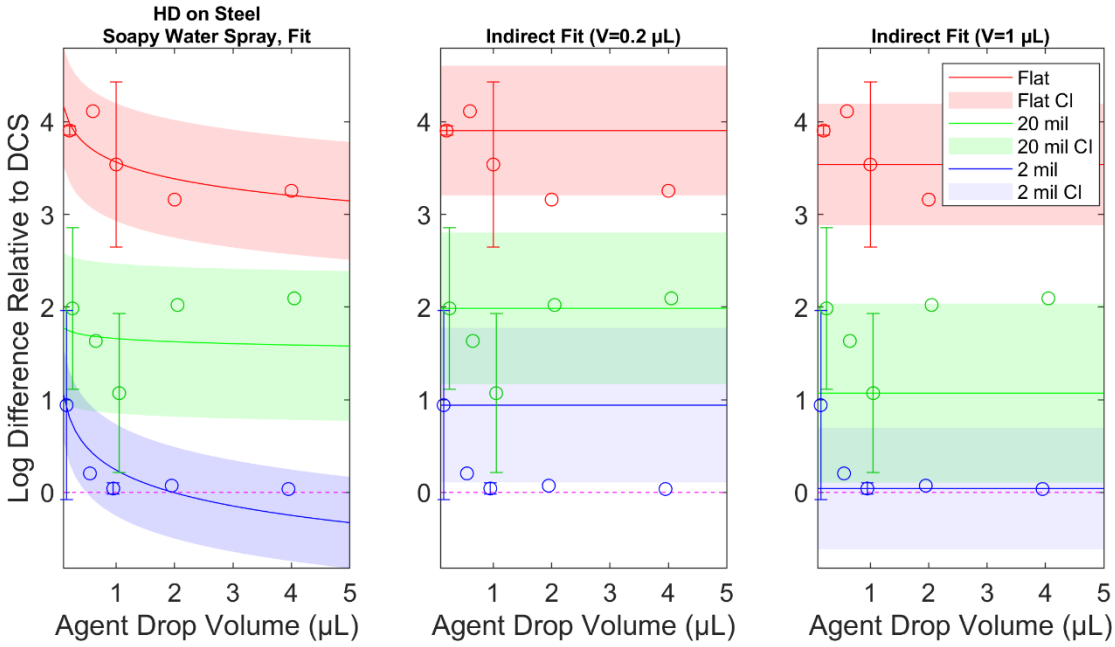


Figure 35. LD as a function of agent drop volume compared to DCS for HD on steel with soapy water spray. Graphs for the fit (left), indirect with 0.2 µL data (middle), and indirect with 1 µL data (right). Data points with error bars are discretely calculated LD values with 95% CI for 1 and 0.2 µL drops. Data points are slightly offset in x to facilitate visualization.

Visual inspection of the LD graphs show that the efficacy calculated with the fit best agrees with the LD calculated from the data (open circles). The deviations in LD across techniques follow the same trend as the RMSE values for each technique (Table 41). Further, the fit technique allows the efficacy to vary with the agent drop volume. The indirect techniques assume that m=1, and as a result the LD must be constant across drop volumes.

7.2.3 Alkyd Coating

The results for HD on Alkyd coating with Soapy Water Spray treatment are provided in Figure 36, and fit summary in Table 42. Note that the results for this material span five orders of magnitude. The statistical summary for 0.2 and 1 µL drops are provided in Table 43.

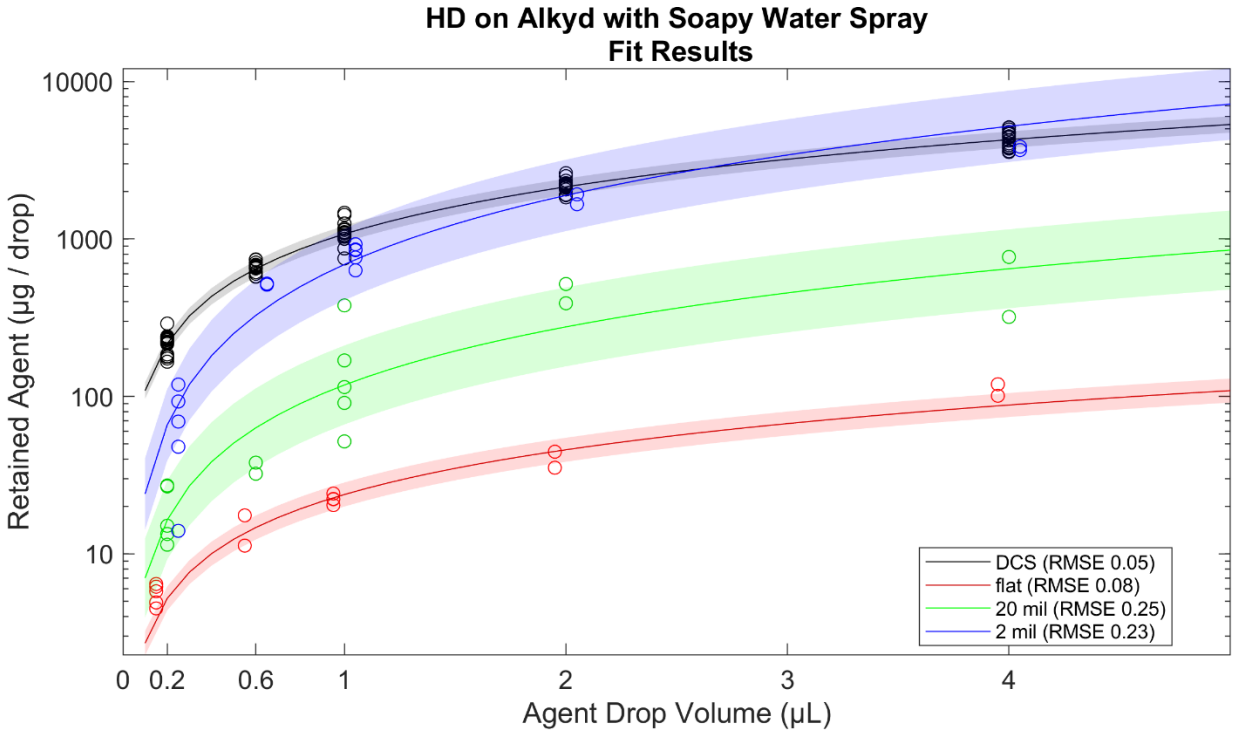


Figure 36. Variable agent drop volume results for HD on Alkyd coating with Soapy Water Spray. The shaded region represents antilog($\mu_{fit}(V)$ ± RMSE). Datapoints have been slightly offset in the x-axis to facilitate visualization.

Table 42. Variable agent drop volume regression results for HD on Alkyd coating with Soapy Water Spray treatment.

Treatment	Material	Feature	Slope, s (log_{10}[µg/µL])	Intercept, b (log_{10}[µg])	RMSE, (log_{10}[µg])	Adj. R^2
Soapy Water Spray	Alkyd	DCS	0.995	3.031	0.052	0.987
		flat	0.944	1.377	0.076	0.970
		20	1.226	2.072	0.250	0.833
		2	1.457	2.838	0.227	0.896

Table 43. Statistical summary for HD on Alkyd coating with Soapy Water Spray treatment for 0.2 and 1 µL agent drops.

Feature	Agent Drop Vol (µL)	n	$\log_{10}$(RA [µg/drop])			Inverse Transform (µg/drop)		
			µ	σ	CV_{log} (%)	Geo. Mean	Lower Bound PI	Upper Bound PI
DCS	0.2	19	2.33	0.06	9.1%	214	161	283
	1	20	3.02	0.07	10.6%	1,047	755	1,453
flat	0.2	5	0.74	0.07	10.1%	5	3	9
	1	5	1.35	0.03	3.9%	22	18	28
20 mil	0.2	5	1.24	0.18	27.1%	17	5	61
	1	5	2.11	0.32	51.9%	129	14	1,211
2 mil	0.2	5	1.74	0.36	59.8%	55	4	684
	1	5	2.90	0.06	9.8%	794	522	1,209

The 0.2 and 1 µL agent drop volume results were used for the indirect fit extrapolation, shown in Figure 34. The 1 µL flat panel results produced σ = 0.72 $\log_{10}$(µg), and applying Eq. 9 to this condition produced an indirect fit with RMSE of 0.66 $\log_{10}$(µg). Similar analysis was conducted for each material-feature and agent drop volume combination. The RMSE for all fits are provided in Table 44. In general, the fit technique provided the lowest RMSE, followed by indirect with V=1 µL, and 0.2 µL, respectively.

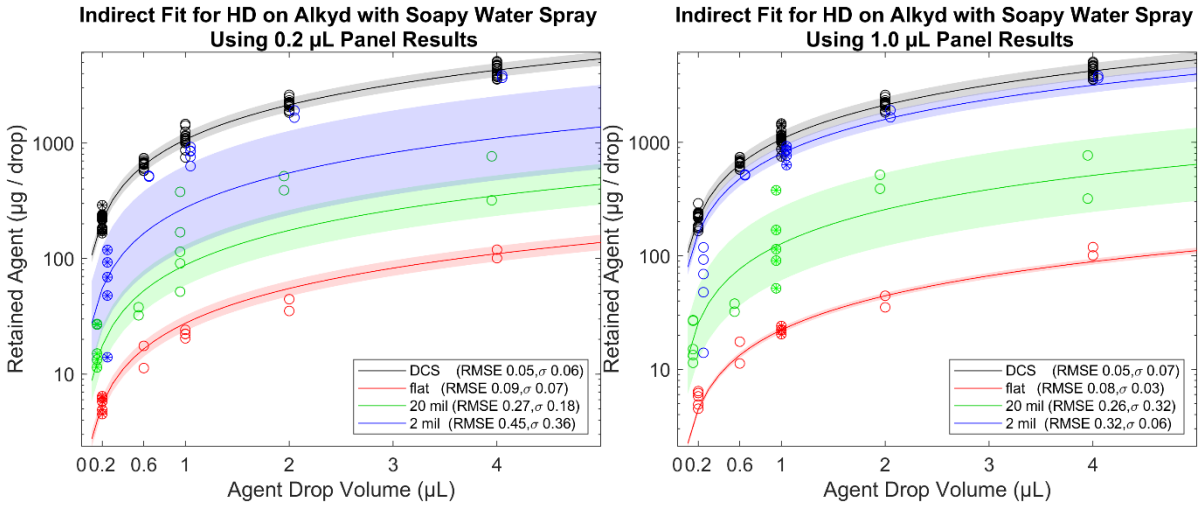


Figure 37. Inverse transformed indirect fits for HD on Alkyd coating with soapy water spray for 0.2 µL drops (left) and 1 µL drops (right). Asterisks (*) indicate the specific data values used to produce each indirect fit. The shaded region corresponds to antilog($\mu_{Indirect}(V) \pm \sigma$).

Table 44. Comparison of RMSE (in $\log_{10}(\mu g)$ units) for each of the source term fitting techniques for HD on Alkyd coating with soapy water spray

Treatment	Material	Feature	Indirect, V=0.2 µL	Indirect, V=1 µL	Fit		RA/drop, V=1 µL
			RMSE	RMSE	m	RMSE	σ
Soapy Water Spray	Alkyd coating	DCS	0.05	0.05	0.99	0.05	0.07
		flat	0.09	0.08	0.94	0.08	0.03
		20 mil	0.27	0.26	1.23	0.25	0.32
		2 mil	0.45	0.32	1.46	0.23	0.06

The LD comparing the response to the DCS (i.e., reduction from applied mass) is provided in Figure 38. These data show that the flat panel results have an LD of about 1.6, the 20 mil shim samples have a lower LD of about 1, and the 2 mil shim has an LD ranging from 0.5 at small drop volumes with decreasing efficacy to 0 LD at larger drop volumes. If the line in Figure 38 flat, then efficacy is constant across agent drop volumes, if the line has a positive or negative slope then the efficacy of the treatment process varies with the drop volume. Similar to previous results, the fit technique tends to show better agreement with the data.

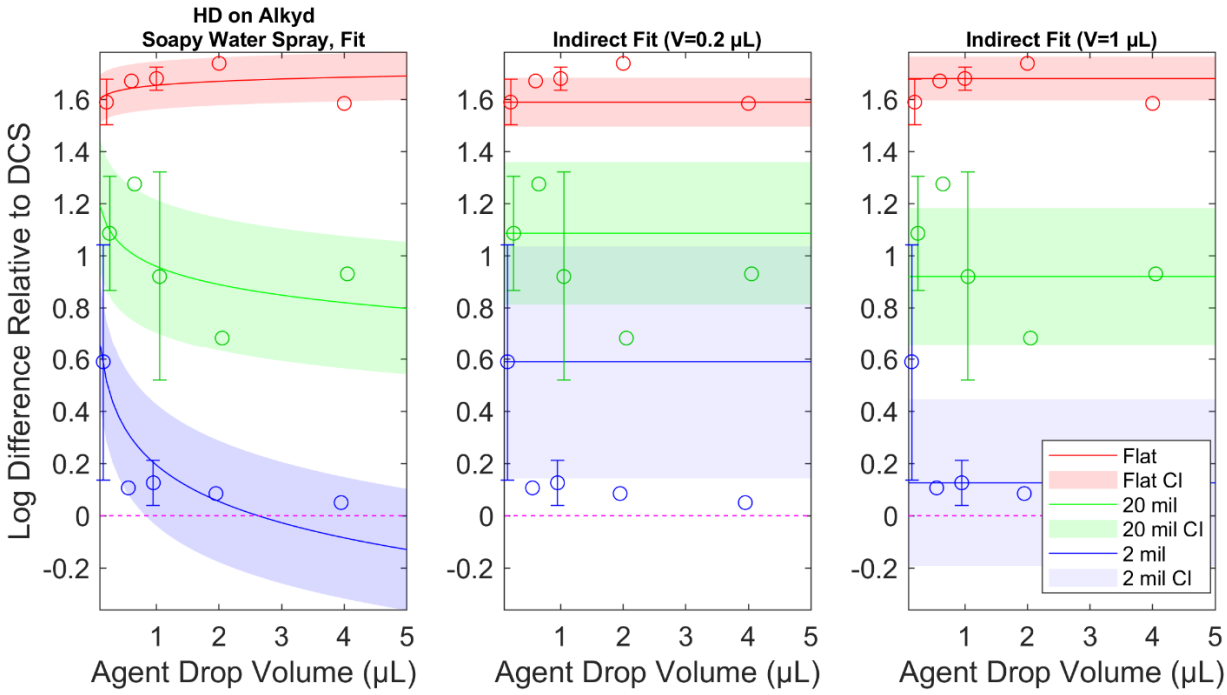


Figure 38. LD as a function of agent drop volume compared to DCS for HD on Alkyd coating with soapy water spray. Data points with error bars are specifically calculated LD values with 95% CI for 1 and 0.2 µL drops, data are slightly offset in x to facilitate visualization.

7.2.4 Polyurethane Coating

The results for HD on PU coating with Soapy Water Spray treatment are provided in Figure 39, and fit summary in Table 45. Note that the results for this material span five orders of magnitude. The statistical summary for 0.2 and 1 µL drops are provided in Table 46.

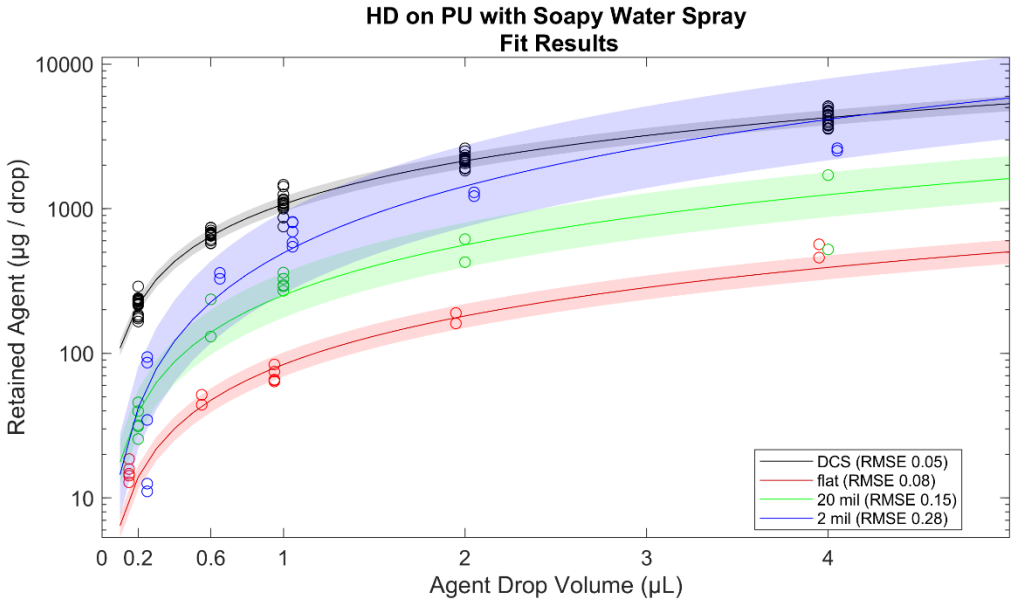


Figure 39. Variable agent drop volume results for HD on PU coating with Soapy Water Spray. The shaded region represents antilog($\mu_{fit}(V)$ ± RMSE). Datapoints have been slightly offset in the x-axis to facilitate visualization.

Table 45. Variable agent drop volume regression results for HD on PU coating with Soapy Water Spray treatment.

Treatment	Material	Feature	Slope, s (log_{10}[µg/µL])	Intercept, b (log_{10}[µg])	RMSE, (log_{10}[µg])	Adj. R^2
Soapy Water Spray	PU coating	DCS	0.995	3.031	0.052	0.987
		flat	1.115	1.923	0.083	0.975
		20	1.155	2.402	0.155	0.921
		2	1.533	2.694	0.282	0.860

Table 46. Statistical summary for HD on PU coating with Soapy Water Spray treatment for 0.2 and 1 µL agent drops.

Feature	Agent Drop Vol (µL)	n	Retained Agent (µg/drop)			Inverse Transform (µg/drop)		
			µ	σ	CV_{log} (%)	Geo. Mean	Lower Bound PI	Upper Bound PI
DCS	0.2	19	2.33	0.06	9.1%	214	161	283
	1	20	3.02	0.07	10.6%	1,047	755	1,453
flat	0.2	5	1.18	0.06	9.1%	15	10	23
	1	5	1.85	0.05	7.7%	71	50	100
20 mil	0.2	5	1.53	0.10	15.1%	34	17	68
	1	5	2.49	0.05	7.5%	309	218	439
2 mil	0.2	5	1.52	0.44	75.5%	33	2	721
	1	5	2.83	0.08	12.0%	676	386	1,184

The 0.2 and 1 µL agent drop volume results were used for the indirect fit extrapolation, shown in Figure 40. Similar analysis was conducted for each material-feature and agent drop volume combination. The RMSE for all fits are provided in Table 47. In general, the fit technique provided the lowest RMSE, followed by indirect with V=1 µL, and 0.2 µL, respectively.

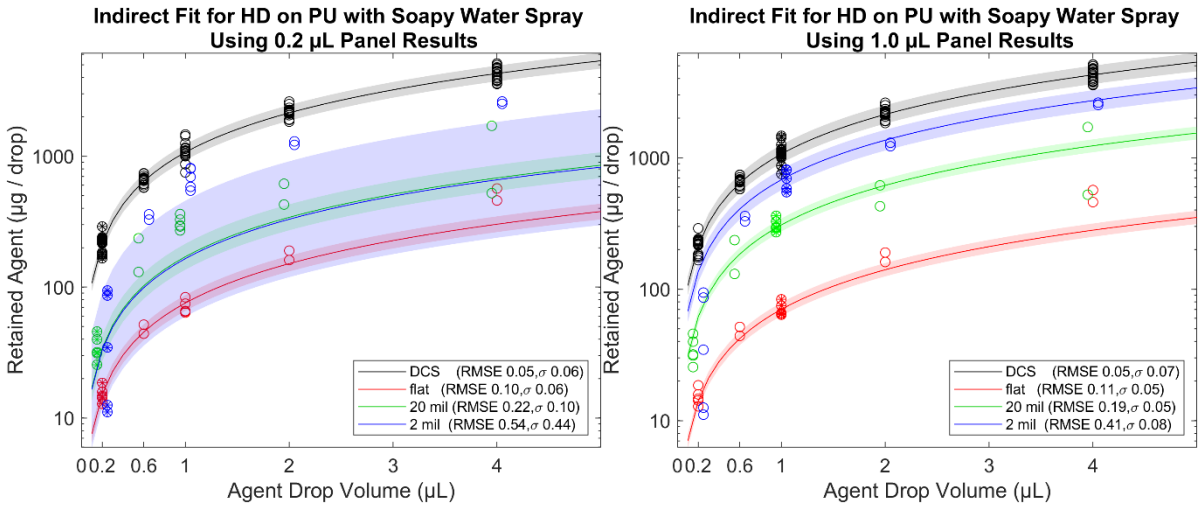


Figure 40. Inverse transformed indirect fits for HD on PU coating with soapy water spray for 0.2 µL drops (left) and 1 µL drops (right). Asterisks (*) indicate the specific data values used to produce each indirect fit. The shaded region corresponds to antilog($\mu_{Indirect}(V) \pm \sigma$).

Table 47. Comparison of RMSE (in $\log_{10}(\mu g)$ units) for each of the source term fitting techniques for HD on PU coating with soapy water spray

Treatment	Material	Feature	Indirect, V=0.2 µL	Indirect, V=1 µL	Fit		RA/drop, V=1 µL
			RMSE	RMSE	m	RMSE	σ
Soapy Water Spray	PU coating	DCS	0.05	0.05	0.99	0.05	0.07
		flat	0.10	0.11	1.12	0.08	0.05
		20 mil	0.22	0.19	1.16	0.15	0.05
		2 mil	0.54	0.41	1.53	0.28	0.08

The LD comparing the response to the DCS (i.e., reduction from applied mass) is provided in Figure 41. This graph shows that agent entrainment in features alters efficacy compared to flat panel testing. These data show that the flat panel results have an LD of about 1.1, the 20 mil shim samples have a lower LD of about 0.6, and the 2 mil shim has an LD ranging from 0.8 at small drop volumes with decreasing efficacy to 0 LD at larger drop volumes. Similar to previous results, the fit technique tends to show better agreement with the data.

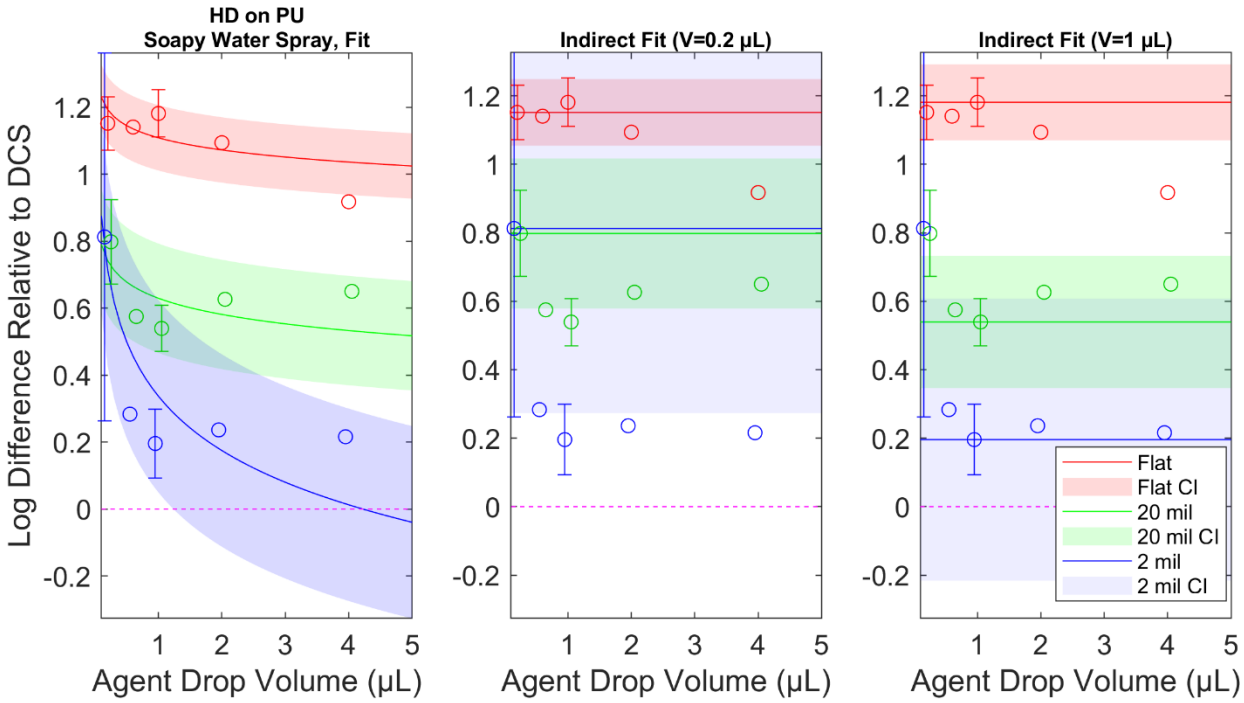


Figure 41. LD as a function of agent drop volume compared to DCS for HD on PU with soapy water spray. Data points with error bars are specifically calculated LD values with 95% CI for 1 and 0.2 µL drops, data are slightly offset in x to facilitate visualization.

7.2.5 Silicone Elastomer

The results for HD on Silicone with Soapy Water Spray treatment are provided in Figure 42, and fit summary in Table 48. Note that the results for this material span two orders of magnitude. Overall, these data show that soapy water spray had almost no effect towards the removal of HD from silicone. The statistical summary for 0.2 and 1 µL drops are provided in Table 49.

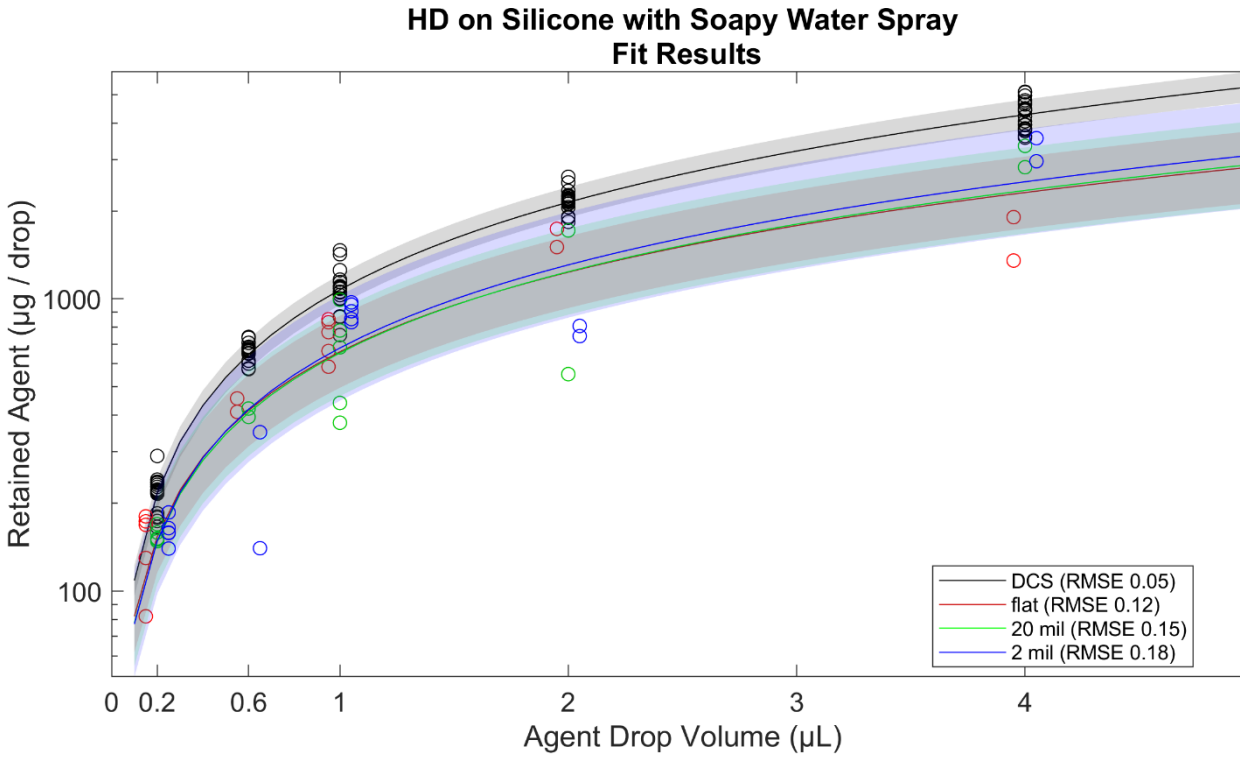


Figure 42. Variable agent drop volume results for HD on Silicone with Soapy Water Spray treatment. The shaded region represents antilog($\mu_{fit}(V)$ ± RMSE). Datapoints have been slightly offset in the x-axis to facilitate visualization.

Table 48. Variable agent drop volume regression results for HD on Silicone with Soapy Water Spray treatment.

Treatment	Material	Feature	Slope, s (log_{10}[µg/µL])	Intercept, b (log_{10}[µg])	RMSE, (log_{10}[µg])	Adj. R^2
Soapy Water Spray	Silicone	DCS	0.995	3.031	0.052	0.987
		flat	0.905	2.819	0.123	0.919
		20	0.923	2.815	0.147	0.892
		2	0.944	2.832	0.180	0.851

Table 49. Statistical summary for HD on silicone-steel with Soapy Water Spray treatment for 0.2 and 1 µL agent drops.

Feature	Agent Drop Vol (µL)	n	$\log_{10}$(RA [µg/drop])			Inverse Transform (µg/drop)		
			µ	σ	CV_{log} (%)	Geo. Mean	Lower Bound PI	Upper Bound PI
DCS	0.2	19	2.33	0.06	9.1%	214	161	283
	1	20	3.02	0.07	10.6%	1,047	755	1,453
flat	0.2	5	2.15	0.14	22.0%	141	53	376
	1	5	2.87	0.07	10.5%	741	454	1,210
20 mil	0.2	5	2.19	0.02	3.6%	155	135	178
	1	5	2.79	0.17	27.0%	617	188	2,028
2 mil	0.2	5	2.21	0.04	6.7%	162	123	215
	1	5	2.96	0.03	4.5%	912	739	1,125

The 0.2 and 1 µL agent drop volume results were used for the indirect fit extrapolation, shown in Figure 43. Similar analysis was conducted for each material-feature and agent drop volume combination. The RMSE for all fits are provided in Table 50. In general, the fit technique provided the lowest RMSE, followed by indirect with V=1 µL, and 0.2 µL, respectively.

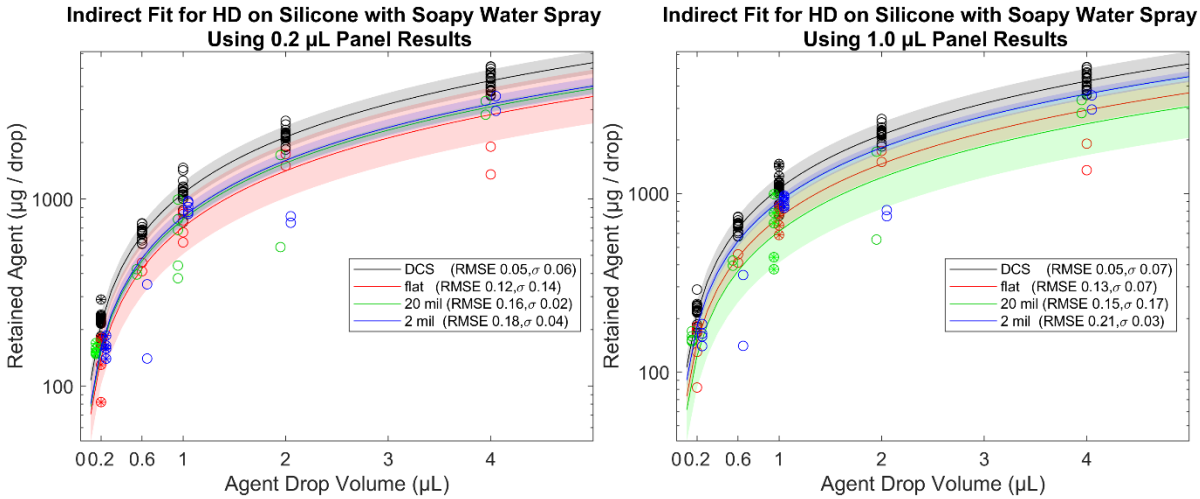


Figure 43. Inverse transformed indirect fits for HD on silicone with soapy water spray for 0.2 µL drops (left) and 1 µL drops (right). Asterisks (*) indicate the specific data values used to produce each indirect fit. The shaded region corresponds to antilog($\mu_{Indirect}(V) \pm \sigma$).

Table 50. Comparison of RMSE (in $\log_{10}(\mu g)$ units) for each of the source term fitting techniques for HD on silicone with soapy water spray

Treatment	Material	Feature	Indirect, V=0.2 µL	Indirect, V=1 µL	Fit		RA/drop, V=1 µL
			RMSE	RMSE	m	RMSE	σ
Soapy Water Spray	Silicone	DCS	0.05	0.05	0.99	0.05	0.07
		flat	0.12	0.13	0.90	0.12	0.07
		20 mil	0.16	0.15	0.92	0.15	0.17
		2 mil	0.18	0.21	0.94	0.18	0.03

The LD comparing the response to the DCS (i.e., reduction from applied mass) is provided in Figure 44. These data show that all of the samples have an LD of about 0.15. In this case, the results are rather uniform across drop volume (i.e., the fit slopes are close to 1), and all techniques produce similar fits.

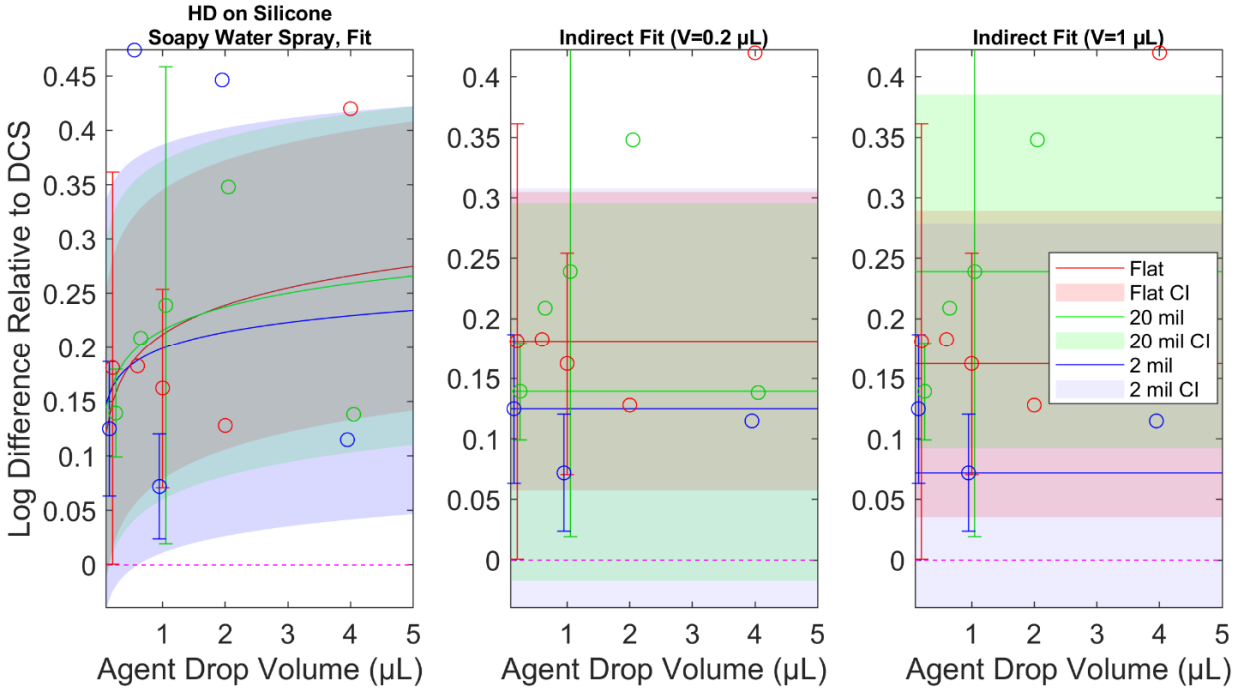


Figure 44. LD as a function of agent drop volume compared to DCS for HD on silicone with soapy water spray. Data points with error bars are specifically calculated LD values with 95% CI for 1 and 0.2 µL drops, data are slightly offset in x to facilitate visualization.

7.3 CHAD Treatment Results

The results for these conditions were obtained over three test days in tests CAP038–CAP040.

7.3.1 DCS Results

The DCS data for each test is provided in Table 38. There are a few conditions below the 20% of nominal delivered mass (< 0.80). Nominal mass assumes the liquid density of 1.27 g/mL and a HD mass purity of 98%.

Table 51. DCS statistics for each test for soapy water treatment

Test ID	Agent Drop Vol (µL)	DCS (µg)			Nominal Mass (µg)	DCS / $M_{Nominal}$
		n	Mean	Std Dev		
CAP038	0.2	3	208.7	27.3	248.9	0.84
	0.6	3	665.4	18	746.8	0.89
	1	3	1116.5	21.5	1244.6	0.90
	2	3	2152.7	38.1	2489.2	0.87
	4	3	3911.3	152.4	4978.4	0.79
CAP039	0.2	3	237.4	3.2	248.9	0.95
	0.6	3	695.3	41	746.8	0.93
	1	3	995.5	212.3	1244.6	0.80
	2	3	2487.5	138	2489.2	1.00
	4	3	4994.3	185.7	4978.4	1.00
CAP040	0.2	3	218.8	2.2	248.9	0.88
	0.6	3	676.4	28.1	746.8	0.91
	1	3	1114.3	121.7	1244.6	0.90
	2	3	2167.5	13.7	2489.2	0.87
	4	3	4630.2	286.1	4978.4	0.93

Red colored text indicates results out of acceptance criterion (1 ± 0.2)

7.3.2 Steel

The results for HD on Steel with CHAD treatment are provided in Figure 45, with fit results provided in Table 52. Note that the results for this material span five orders of magnitude. Most of the flat panel and 20 mil shim results are below quantitation limits (< 10 ng/drop). This resulted in low adj. R^2 values for flat panel and the 20 mil shim. The 2 mil shim data is highly variant with the 1 µL drop spanning a four order of magnitude range. This is reflected in the very low adj. R^2 value and large RMSE. The statistical summary for 0.2 and 1 µL drops is provided in Table 53.

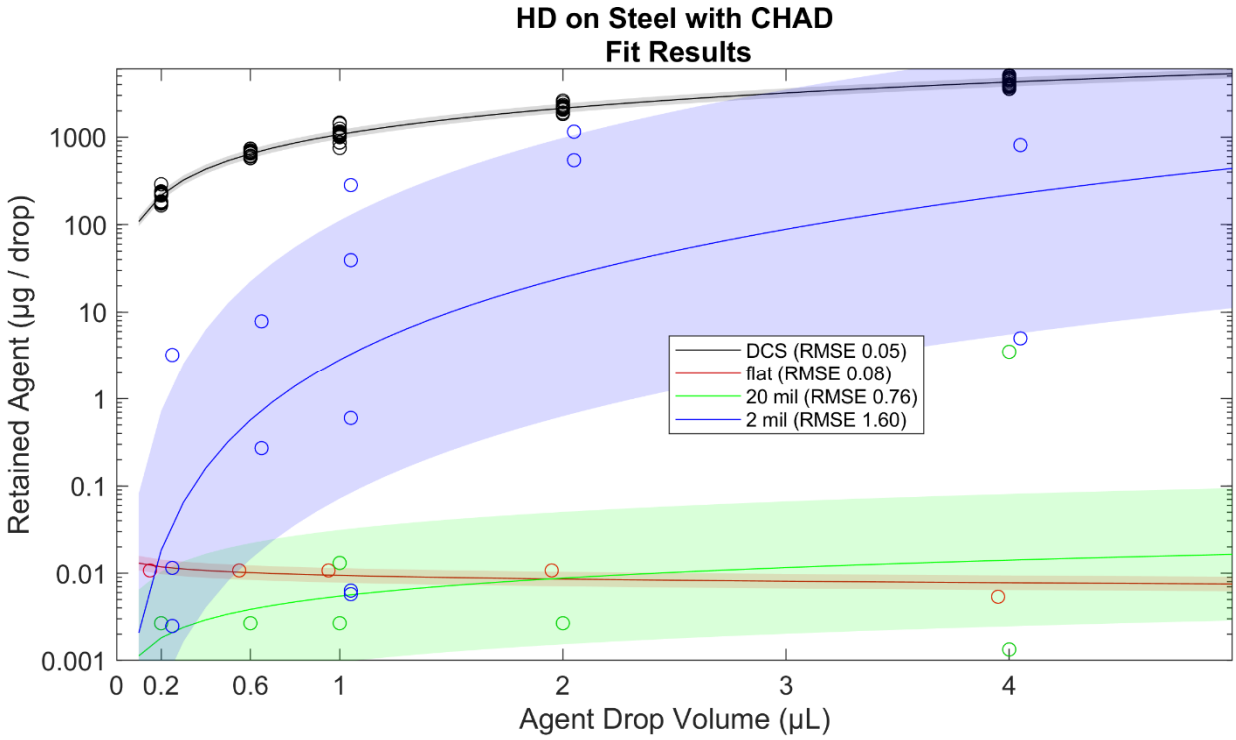


Figure 45. Variable agent drop volume results for HD on Steel with CHAD treatment. The shaded region represents antilog($\mu_{fit}(V)$ ± RMSE). Datapoints have been slightly offset in the x-axis to facilitate visualization.

Table 52. Variable agent drop volume regression results for HD on Steel with CHAD treatment

Treatment	Material	Feature	Slope, s (log_{10}[µg/µL])	Intercept, b (log_{10}[µg])	RMSE, (log_{10}[µg])	Adj. R^2
CHAD	Steel	DCS	0.995	3.031	0.052	0.987
		flat	-0.140	-2.025	0.083	0.347
		20	0.684	-2.262	0.758	0.095
		2	3.135	0.452	1.596	0.428

This steel data presents several characteristics that can influence data use. The 2 mil data was above the LOQ and, as is often observed on impermeable materials, provides significant variability. The lower-than-normal goodness of fit parameters are more influenced by the data variability, rather than the fit not describing the data trend. Recall that this variability is a characteristic of the treatment process quantified by the methodology. The advantage of the MC approach used for scale-up is that this variability will be translated through the scale-up process to indicate how this variability would manifest at the asset level. Both the flat and 20 mil shim features produced mostly below LOQ results. Some samples were also below detection levels, and the LOQ or LOD value is reported.

The RA mass per drop in this case can vary based on the dilution factor and that the LOQ and LOD levels are calculated for each chromatography calibration run. The data are

used ‘as is’ for data fitting. This produced low adjusted R^2 values for the flat and 20 mil shim cases. However, as noted previously, there is an acceptable fit to the data. Additionally, the LOQ values produce standard deviations of zero, which will cause non-normal distributions in the scale-up calculations demonstrated in Section 11.

Table 53. Statistical summary for HD on Steel with CHAD treatment for 0.2 and 1 µL agent drops

Feature	Agent Drop Vol (µL)	n	$\log_{10}$(RA [µg/drop])			Inverse Transform (µg/drop)		
			µ	σ	CV_{log} (%)	Geo. Mean	Lower Bound PI	Upper Bound PI
DCS	0.2	19	2.33	0.06	9.1%	214	161	283
	1	20	3.02	0.07	10.6%	1,047	755	1,453
flat	0.2	5	-1.97	0.00	0.0%	0.011	0.011	0.011
	1	5	-1.97	0.00	0.0%	0.011	0.011	0.011
20 mil	0.2	5	-2.57	0.00	0.0%	0.003	0.003	0.003
	1	5	-2.43	0.31	49.5%	0.004	0.000	0.033
2 mil	0.2	5	-1.72	1.29	666.8%	0.019	0.000	160
	1	5	-0.12	2.14	19871.3%	0.759	0.000	2,441,968

The 0.2 and 1 µL agent drop volume results were used for the indirect fit extrapolation, shown in Figure 46. Similar analysis was conducted for each material-feature and agent drop volume combination. The RMSE for all fits are provided in Table 54. In general, the fit technique provided the lowest RMSE, followed by indirect with V=1 µL, and 0.2 µL, respectively. The LD as a function of V (using Eq. 11) was calculated for each fitting technique as shown in Figure 47.

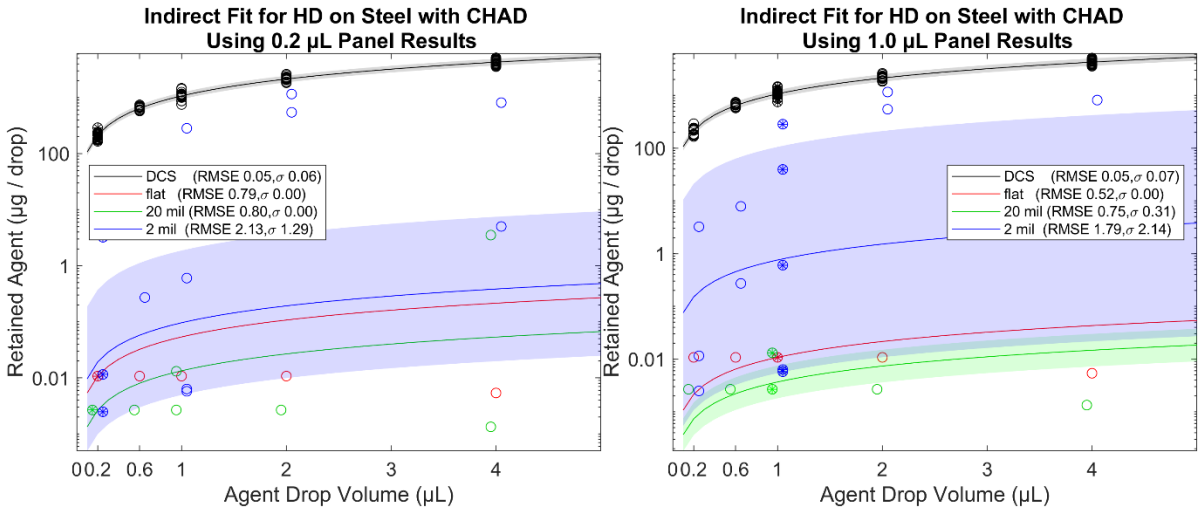


Figure 46. Inverse transformed indirect fits for HD on steel with CHAD for 0.2 µL drops (left) and 1 µL drops (right). Asterisks (*) indicate the specific data values used to produce each indirect fit. The shaded region corresponds to antilog($\mu_{Indirect}(V) \pm \sigma$).

Table 54. Comparison of RMSE (in $\log_{10}$(µg) units) for each of the source term fitting techniques for HD on steel with CHAD

Treatment	Material	Feature	Indirect, V=0.2 µL	Indirect, V=1 µL	Fit		RA/drop, V=1 µL
			RMSE	RMSE	m	RMSE	σ
CHAD	Steel	DCS	0.05	0.05	0.99	0.05	0.07
		flat	0.79	0.52	-0.14	0.08	0.00
		20 mil	0.80	0.75	0.68	0.76	0.31
		2 mil	2.13	1.79	3.14	1.60	2.14

The LD comparing the response to the DCS (i.e., reduction from applied mass) is provided in Figure 47. This graph shows that agent entrainment in features alters efficacy compared to flat panel testing. These data show that the flat panel results have an LD of about from 46, the 20 mil shim samples have a LD of about 5, and the 2 mil shim has an LD ranging from 4 at small drop volumes with decreasing efficacy to 1 LD at larger drop volumes. Similar to previous results, the fit technique tends to show better agreement with the data.

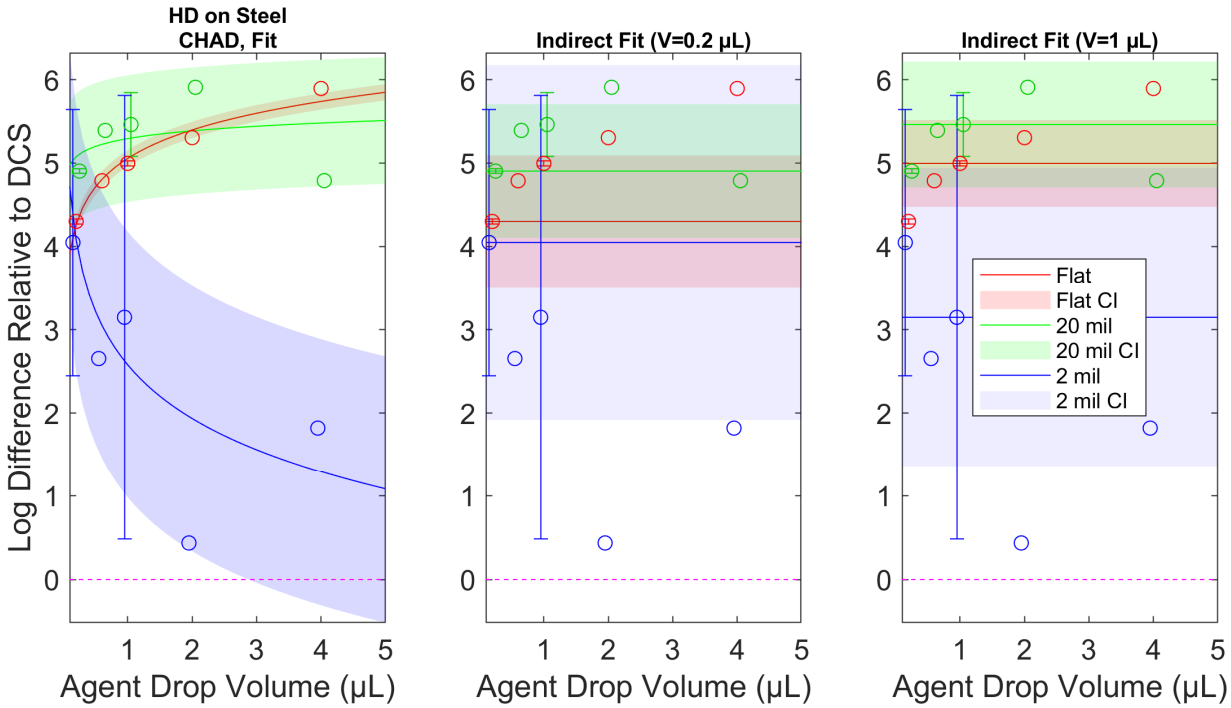


Figure 47. LD as a function of agent drop volume compared to DCS for HD on steel with CHAD. Data points with error bars are specifically calculated LD values with 95% CI for 1 and 0.2 µL drops, data are slightly offset in x to facilitate visualization.

7.3.3 Alkyd Coating

The results for HD on Alkyd coating with CHAD treatment are provided in Figure 48, with the regression results provided in Table 55. Note that the results for this material span three orders of magnitude. The statistical summary for 0.2 and 1 µL drops are provided in Table 56.

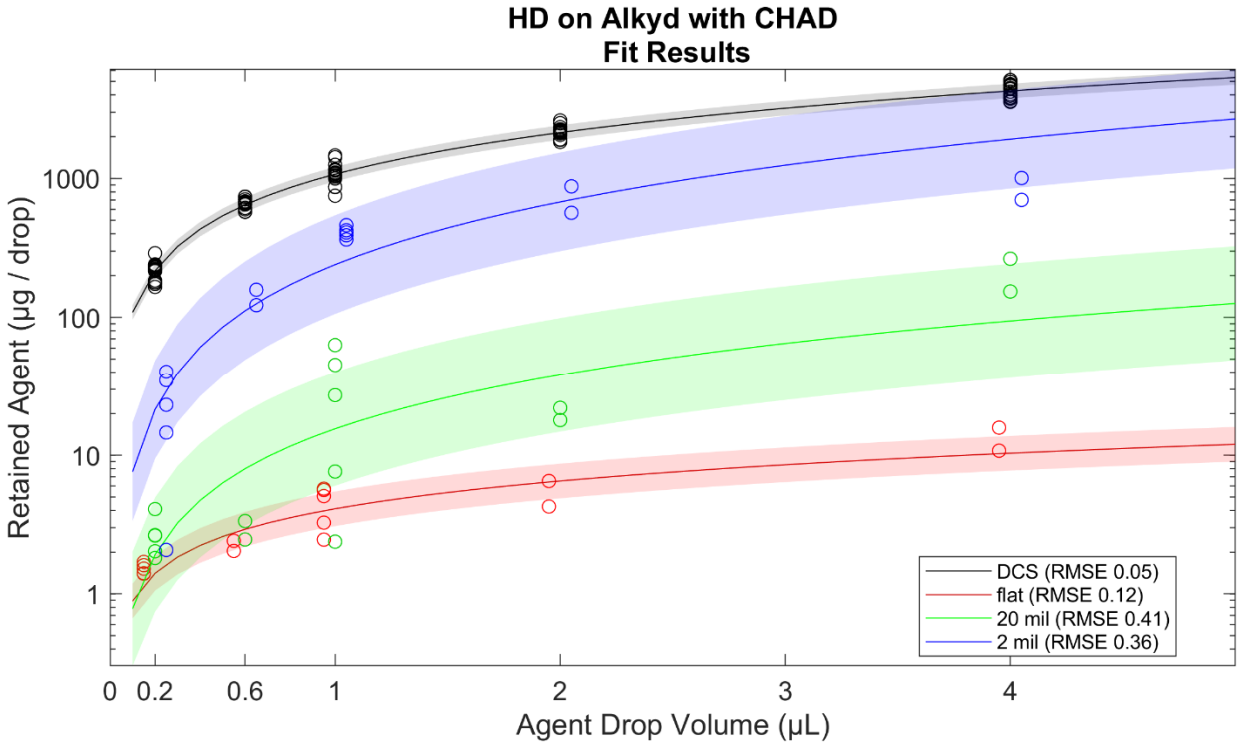


Figure 48. Variable agent drop volume results for HD on Alkyd coating with CHAD. The shaded region represents antilog($\mu_{fit}(V)$ ± RMSE). Datapoints have been slightly offset in the x-axis to facilitate visualization.

Table 55. Variable agent drop volume regression results for HD on Alkyd coating with CHAD treatment.

Treatment	Material	Feature	Slope, s (log_{10}[µg/µL])	Intercept, b (log_{10}[µg])	RMSE, (log_{10}[µg])	Adj. R^2
CHAD	Alkyd	DCS	0.995	3.031	0.052	0.987
		flat	0.666	0.614	0.125	0.856
		20	1.300	1.192	0.411	0.671
		2	1.500	2.380	0.355	0.787

Table 56. Statistical summary for HD on Alkyd coating with CHAD treatment for 0.2 and 1 µL agent drops.

Feature	Agent Drop Vol (µL)	n	$\log_{10}$(RA [µg/drop])			Inverse Transform (µg/drop)		
			µ	σ	CV_{log} (%)	Geo. Mean	Lower Bound PI	Upper Bound PI
DCS	0.2	19	2.33	0.06	9.1%	214	161	283
	1	20	3.02	0.07	10.6%	1,047	755	1,453
flat	0.2	5	0.18	0.04	5.6%	2	1	2
	1	5	0.62	0.16	25.2%	4	1	13
20 mil	0.2	5	0.40	0.14	20.9%	3	1	7
	1	5	1.23	0.59	111.2%	17	0.3	1,057
2 mil	0.2	5	1.20	0.52	93.5%	16	0.4	604
	1	5	2.61	0.04	5.9%	407	308	539

The 0.2 and 1 µL agent drop volume results were used for the indirect fit extrapolation, shown in Figure 49. Similar analysis was conducted for each material-feature and agent drop volume combination. The RMSE for all fits are provided in Table 57. In general, the fit technique provided the lowest RMSE, followed by indirect with V=1 µL, and 0.2 µL, respectively.

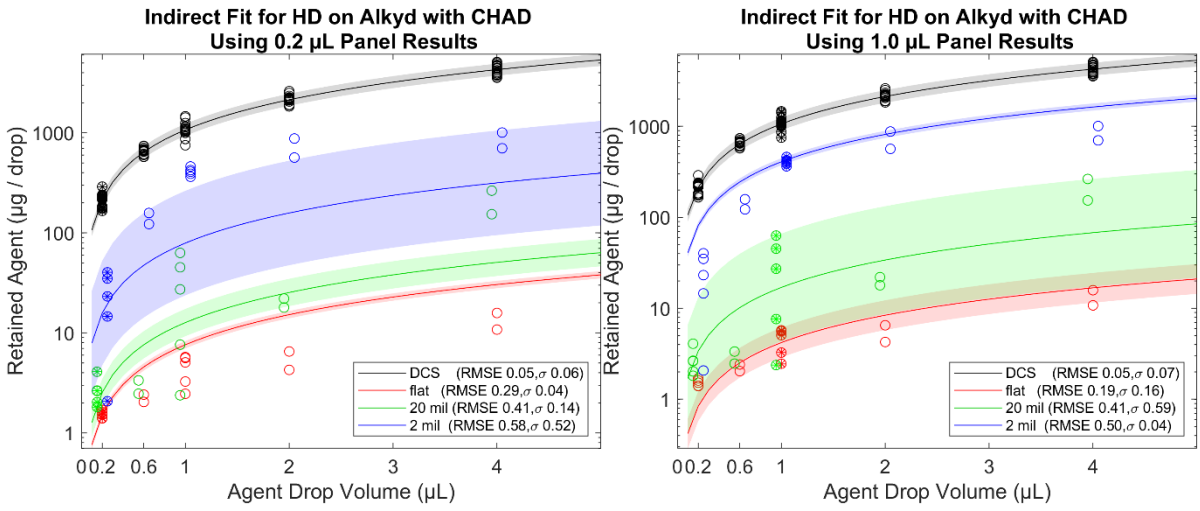


Figure 49. Inverse transformed indirect fits for HD on alkyd coating with CHAD for 0.2 µL drops (left) and 1 µL drops (right). Asterisks (*) indicate the specific data values used to produce each indirect fit. The shaded region corresponds to antilog($\mu_{Indirect}(V) \pm \sigma$).

Table 57. Comparison of RMSE (in $\log_{10}(\mu g)$ units) for each of the source term fitting techniques for HD on Alkyd coating with CHAD

Treatment	Material	Feature	Indirect, V=0.2 µL	Indirect, V=1 µL	Fit		RA/drop, V=1 µL
			RMSE	RMSE	m	RMSE	σ
CHAD	Alkyd coating	DCS	0.05	0.05	0.99	0.05	0.07
		flat	0.29	0.19	0.67	0.12	0.16
		20 mil	0.41	0.41	1.30	0.41	0.59
		2 mil	0.58	0.50	1.50	0.36	0.04

The LD comparing the response to the DCS (i.e., reduction from applied mass) is provided in Figure 50. This graph shows that agent entrainment in features alters efficacy compared to flat panel testing. These data show that the flat panel results have an LD of about 2–2.5, the 20 mil shim samples have a lower LD of about 1.8, and the 2 mil shim has an LD ranging from 1 at small drop volumes with decreasing efficacy to 0.3 LD at larger drop volumes. Similar to previous results, the fit technique tends to show better agreement with the data.

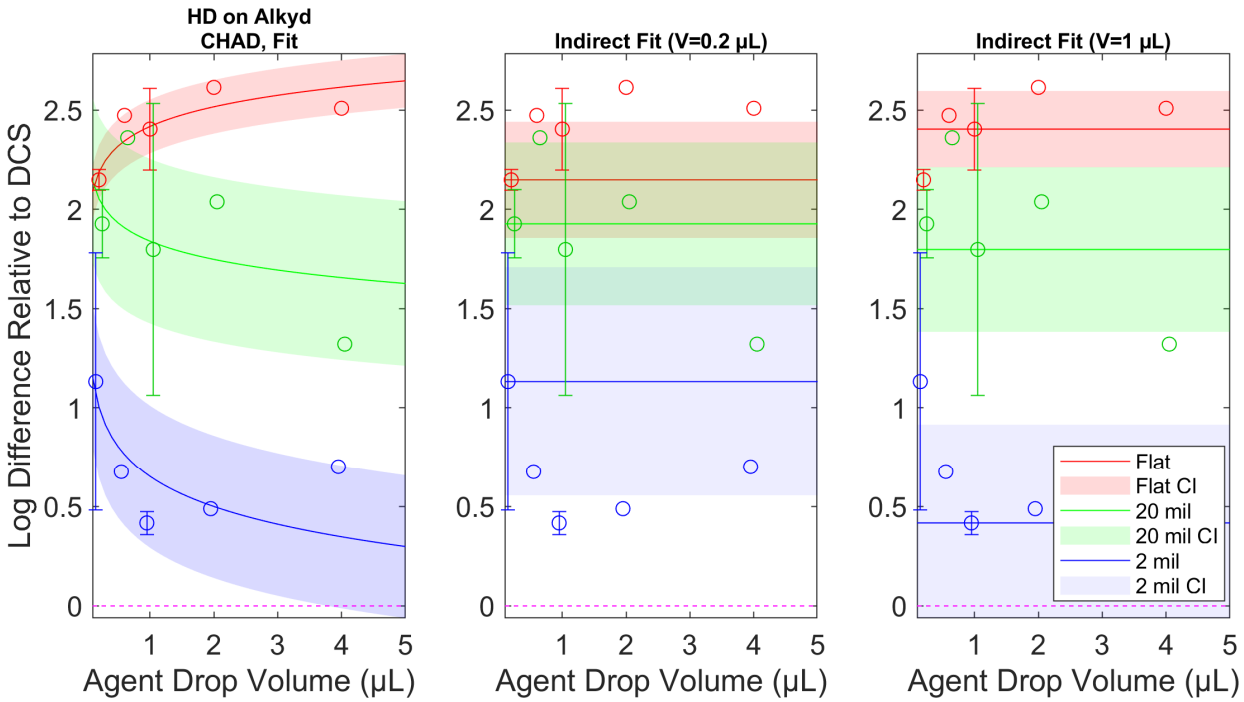


Figure 50. LD as a function of agent drop volume compared to DCS for HD on Alkyd with CHAD. Data points with error bars are specifically calculated LD values with 95% CI for 1 and 0.2 µL drops, data are slightly offset in x to facilitate visualization.

7.3.4 Polyurethane Coating

The results for HD on PU coating with CHAD treatment are provided in Figure 51, with the regression results provided in Table 58. Note that the results for this material span five orders of magnitude. The statistical summary for 0.2 and 1 µL drops are provided in Table 59.

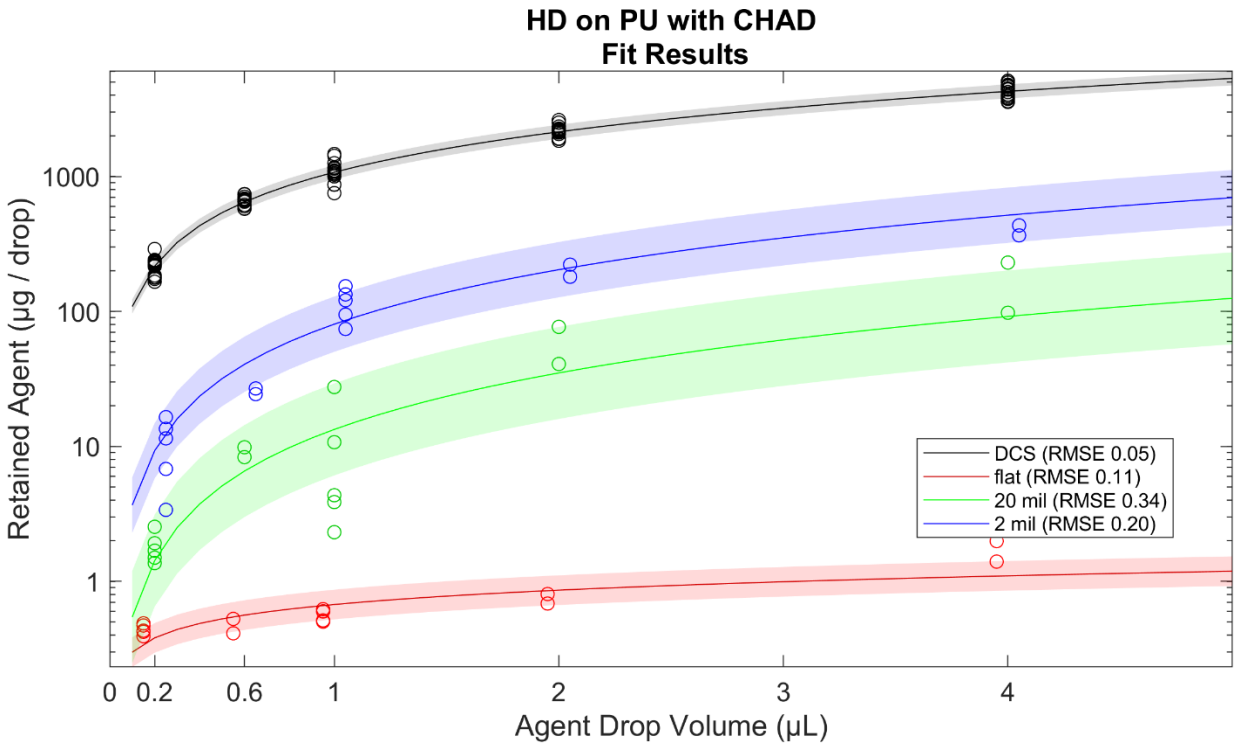


Figure 51. Variable agent drop volume results for HD on PU coating with CHAD. The shaded region represents antilog($\mu_{fit}(V)$ ± RMSE). Datapoints have been slightly offset in the x-axis to facilitate visualization.

Table 58. Variable agent drop volume regression results for HD on PU coating with CHAD treatment.

Treatment	Material	Feature	Slope, s (log_{10}[µg/µL])	Intercept, b (log_{10}[µg])	RMSE, (log_{10}[µg])	Adj. R^2
CHAD	PU	DCS	0.995	3.031	0.052	0.987
		flat	0.353	-0.171	0.110	0.680
		20	1.390	1.126	0.340	0.775
		2	1.341	1.906	0.205	0.900

Table 59. Statistical summary for HD on PU coating with CHAD treatment for 0.2 and 1 µL agent drops.

Feature	Agent Drop Vol (µL)	n	$\log_{10}$(RA [µg/drop])			Inverse Transform (µg/drop)		
			µ	σ	CV_{log} (%)	Geo. Mean	Lower Bound PI	Upper Bound PI
DCS	0.2	19	2.33	0.06	9.1%	214	161	283
	1	20	3.02	0.07	10.6%	1,047	755	1,453
flat	0.2	5	-0.36	0.04	5.8%	0.4	0.3	1
	1	5	-0.25	0.04	6.5%	0.6	0.4	1
20 mil	0.2	5	0.25	0.10	16.0%	2	0.9	4
	1	5	0.81	0.43	71.9%	6	0.3	131
2 mil	0.2	5	0.95	0.28	43.9%	9	1.3	63
	1	5	2.05	0.13	19.3%	112	45	279

The 0.2 and 1 µL agent drop volume results were used for the indirect fit extrapolation, shown in Figure 52. Similar analysis was conducted for each material-feature and agent drop volume combination. The RMSE for all fits are provided in Table 60. In general, the fit technique provided the lowest RMSE, followed by indirect with V=1 µL, and 0.2 µL, respectively.

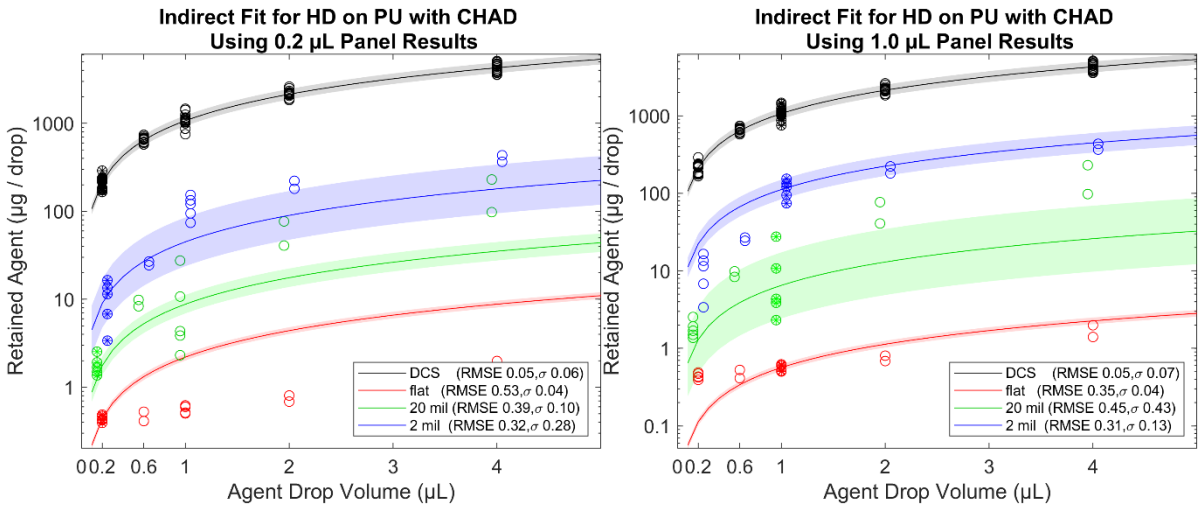


Figure 52. Inverse transformed indirect fits for HD on PU coating with CHAD for 0.2 µL drops (left) and 1 µL drops (right). Asterisks (*) indicate the specific data values used to produce each indirect fit. The shaded region corresponds to antilog($\mu_{Indirect}(V) \pm \sigma$).

Table 60. Comparison of RMSE (in $\log_{10}(\mu g)$ units) for each of the source term fitting techniques for HD on Alkyd coating with CHAD

Treatment	Material	Feature	Indirect, V=0.2 µL	Indirect, V=1 µL	Fit		RA/drop, V=1 µL
			RMSE	RMSE	m	RMSE	σ
CHAD	PU coating	DCS	0.05	0.05	0.99	0.05	0.07
		flat	0.53	0.35	0.35	0.11	0.04
		20 mil	0.39	0.45	1.39	0.34	0.43
		2 mil	0.32	0.31	1.34	0.20	0.13

The LD comparing the response to the DCS (i.e., reduction from applied mass) is provided in Figure 53. This graph shows that agent entrainment in features alters efficacy compared to flat panel testing. These data show that the flat panel results have an LD ranging from 2.6–3.5, the 20 mil shim samples have a lower LD of about 2.1–1.6, and the 2 mil shim has an LD ranging from 1.5 at small drop volumes with decreasing efficacy to 0.9 LD at larger drop volumes. Similar to previous results, the fit technique tends to show better agreement with the data.

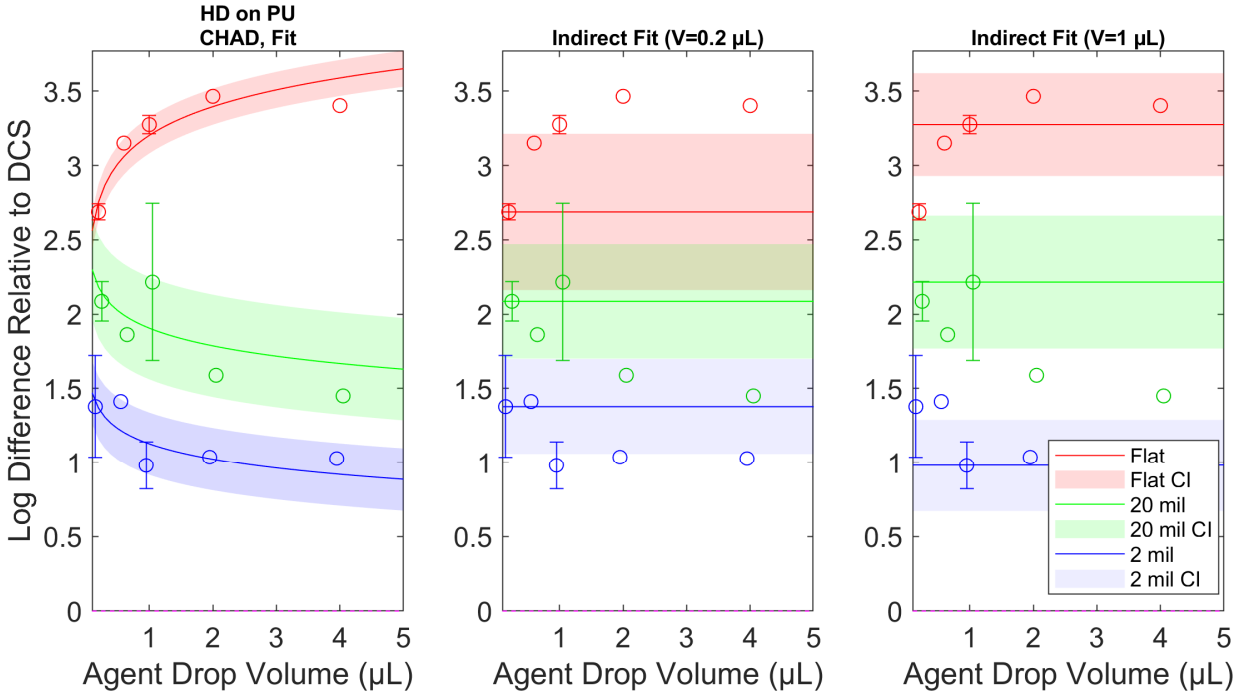


Figure 53. LD as a function of agent drop volume compared to DCS for HD on PU coating with CHAD. Data points with error bars are specifically calculated LD values with 95% CI for 1 and 0.2 µL drops, data are slightly offset in x to facilitate visualization.

7.3.5 Silicone Elastomer

The results for HD on Silicone with CHAD treatment are provided in Figure 54 with the regression results provided in Table 61. Note that the results for this material span two orders of magnitude. The statistical summary for 0.2 and 1 µL drops are provided in Table 62.

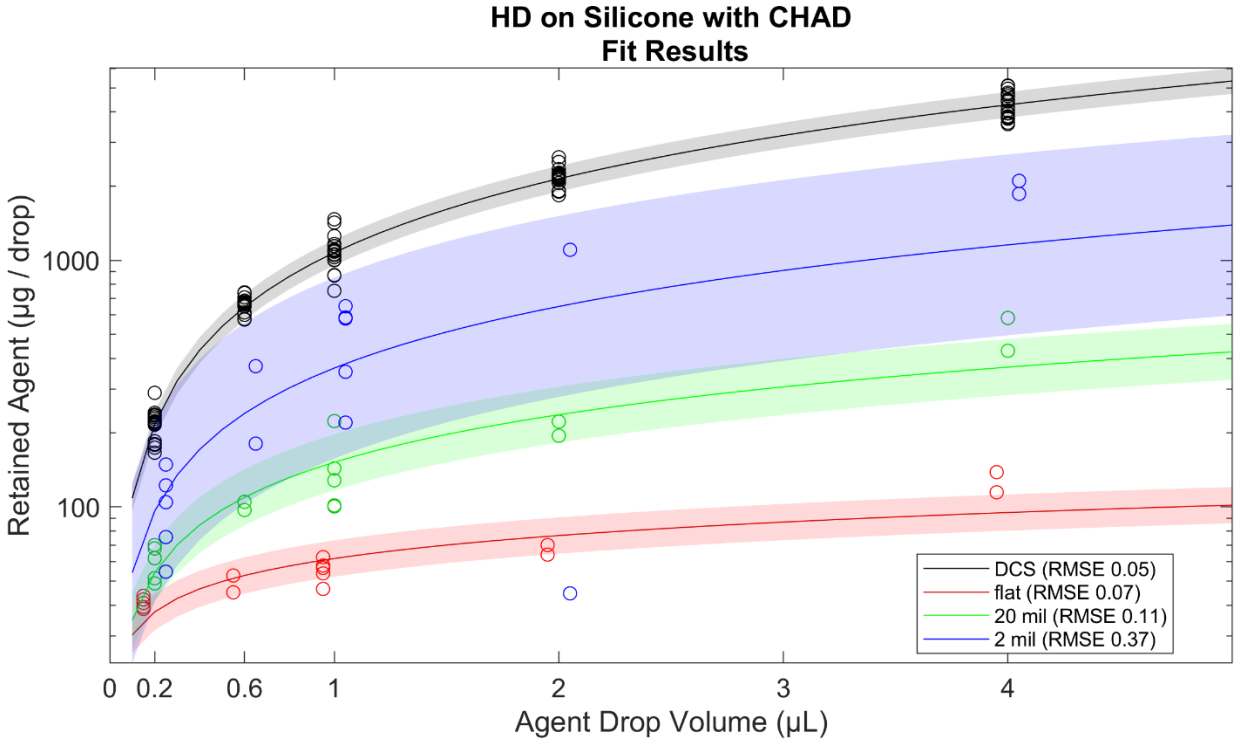


Figure 54. Variable agent drop volume results for HD on Silicone with CHAD treatment. The shaded region represents antilog($\mu_{fit}(V)$ ± RMSE). Datapoints have been slightly offset in the x-axis to facilitate visualization.

Table 61. Variable agent drop volume regression results for HD on Silicone with CHAD treatment.

Treatment	Material	Feature	Slope, s (log_{10}[µg/µL])	Intercept, b (log_{10}[µg])	RMSE, (log_{10}[µg])	Adj. R^2
CHAD	Silicone	DCS	0.995	3.031	0.052	0.987
		flat	0.310	1.791	0.073	0.787
		20	0.640	2.181	0.113	0.870
		2	0.829	2.563	0.367	0.503

Table 62. Statistical summary for HD on silicone-steel with CHAD treatment for 0.2 and 1 µL agent drops.

Feature	Agent Drop Vol (µL)	n	$\log_{10}$(RA [µg/drop])			Inverse Transform (µg/drop)		
			µ	σ	CV_{log} (%)	Geo. Mean	Lower Bound PI	Upper Bound PI
DCS	0.2	19	2.33	0.06	9.1%	214	161	283
	1	20	3.02	0.07	10.6%	1,047	755	1,453
flat	0.2	5	1.61	0.02	3.3%	41	35	47
	1	5	1.74	0.05	7.3%	55	39	78
20 mil	0.2	5	1.77	0.07	10.7%	59	36	96
	1	5	2.12	0.14	21.8%	132	49	351
2 mil	0.2	5	1.98	0.17	26.6%	95	29	314
	1	5	2.65	0.20	31.0%	447	110	1,812

The 0.2 and 1 µL agent drop volume results were used for the indirect fit extrapolation, shown in Figure 55. Similar analysis was conducted for each material-feature and agent drop volume combination. The RMSE for all fits are provided in Table 63. In general, the fit technique provided the lowest RMSE, followed by indirect with V=1 µL, and 0.2 µL, respectively.

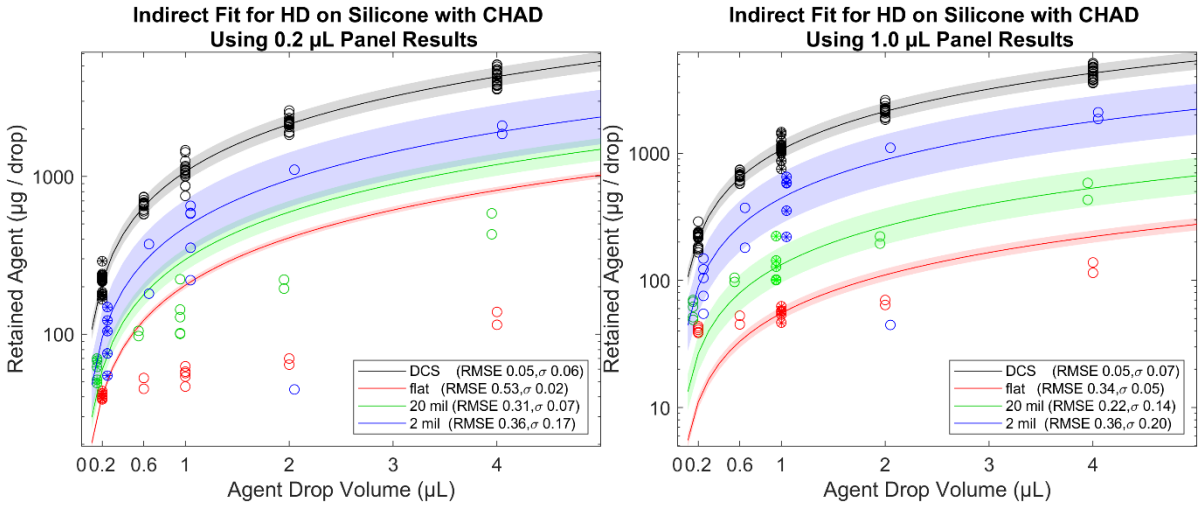


Figure 55. Inverse transformed indirect fits for HD on silicone with CHAD for 0.2 µL drops (left) and 1 µL drops (right). Asterisks (*) indicate the specific data values used to produce each indirect fit. The shaded region corresponds to antilog($\mu_{Indirect}(V) \pm \sigma$).

Table 63. Comparison of RMSE (in $\log_{10}(\mu g)$ units) for each of the source term fitting techniques for HD on silicone with CHAD

Treatment	Material	Feature	Indirect, V=0.2 µL	Indirect, V=1 µL	Fit		RA/drop, V=1 µL
			RMSE	RMSE	m	RMSE	σ
CHAD	Silicone	DCS	0.05	0.05	0.99	0.05	0.07
		flat	0.53	0.34	0.31	0.07	0.05
		20 mil	0.31	0.22	0.64	0.11	0.14
		2 mil	0.36	0.36	0.83	0.37	0.20

The LD comparing the response to the DCS (i.e., reduction from applied mass) is provided in Figure 56. This graph shows that agent entrainment in features alters efficacy compared to flat panel testing. These data show that the flat panel results have an LD of about 0.6–1.6, the 20 mil shim samples have a lower LD of about 0.6–1.0, and the 2 mil shim has an LD ranging from 0.4 at small drop volumes with decreasing efficacy to 0.6 LD at larger drop volumes. Similar to previous results, the fit technique tends to show better agreement with the data.

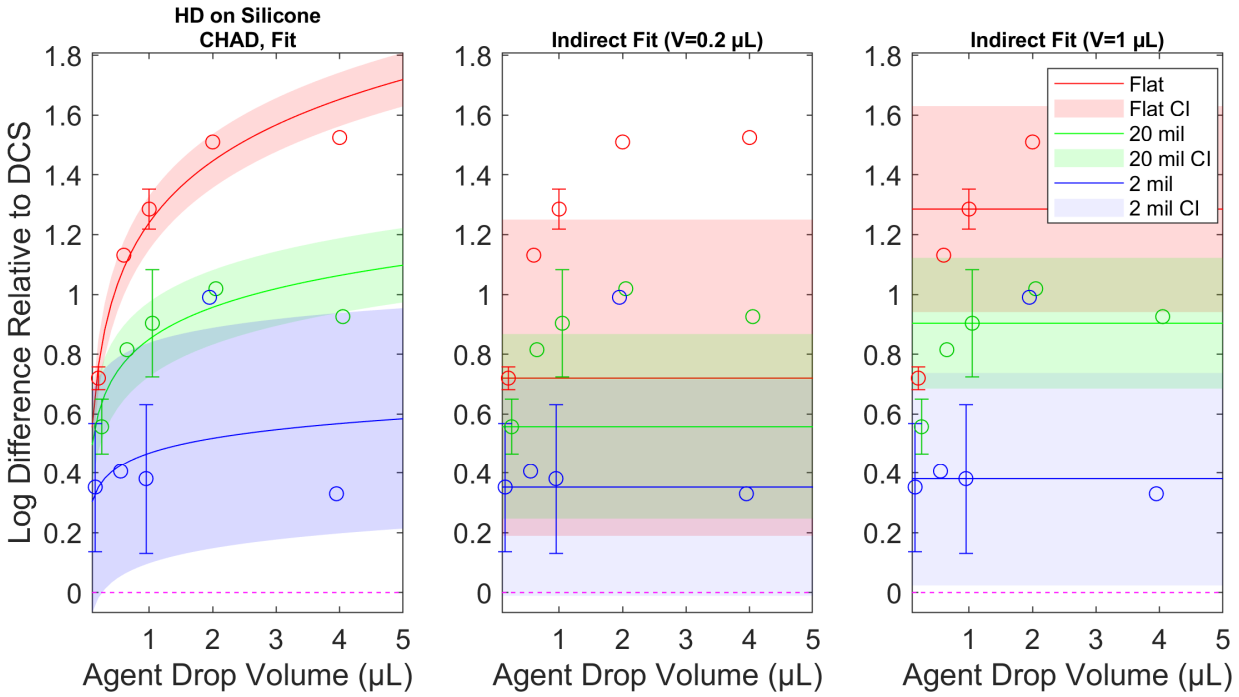


Figure 56. LD as a function of agent drop volume compared to DCS for HD on Silicone with CHAD. Data points with error bars are specifically calculated LD values with 95% CI for 1 and 0.2 µL drops, data are slightly offset in x to facilitate visualization.

7.4 Trends in RA Variability

Validation goal 1.3 was to demonstrate the single day repeatability of different treatment procedures (i.e., the CV). There was no threshold criterion established for CV. To illustrate the results for validation goal 1.3, five replicates were collected for 0.2 and 1 µL drops for each material, feature, treatment combination. The CV_{Log} was calculated for each of these conditions to indicate the variability (i.e., repeatability) of the treatment conditions as measured by the standard deviation of the log transformed RA results, σ, shown in Figure 57. As discussed in Appendix B, the σ value is the multiplicative standard deviation that may be observed for the RA data (i.e., the RA data will be within a standard deviation of antilog(σ) times or divided by the geometric mean),[17,20,21] and is comparable even when the mean values for each condition are significantly different. Across the conditions, the flat panel results tend to produce RA data with smaller σ, around 0.1 $\log_{10}$(µg) or within a factor of 1.25× (see antilog scale on right side of Figure 57. Both shim panel feature sizes produce significantly larger σ, ranging from 0.1–0.8 $\log_{10}$(µg); usually within a factor 2.5×, but up to 6.3× (and for steel 2 mil shim with CHAD as high as σ of 3). The variability (as measured by σ) of the RA response is a characteristic of the agent-material-feature and the treatment process.

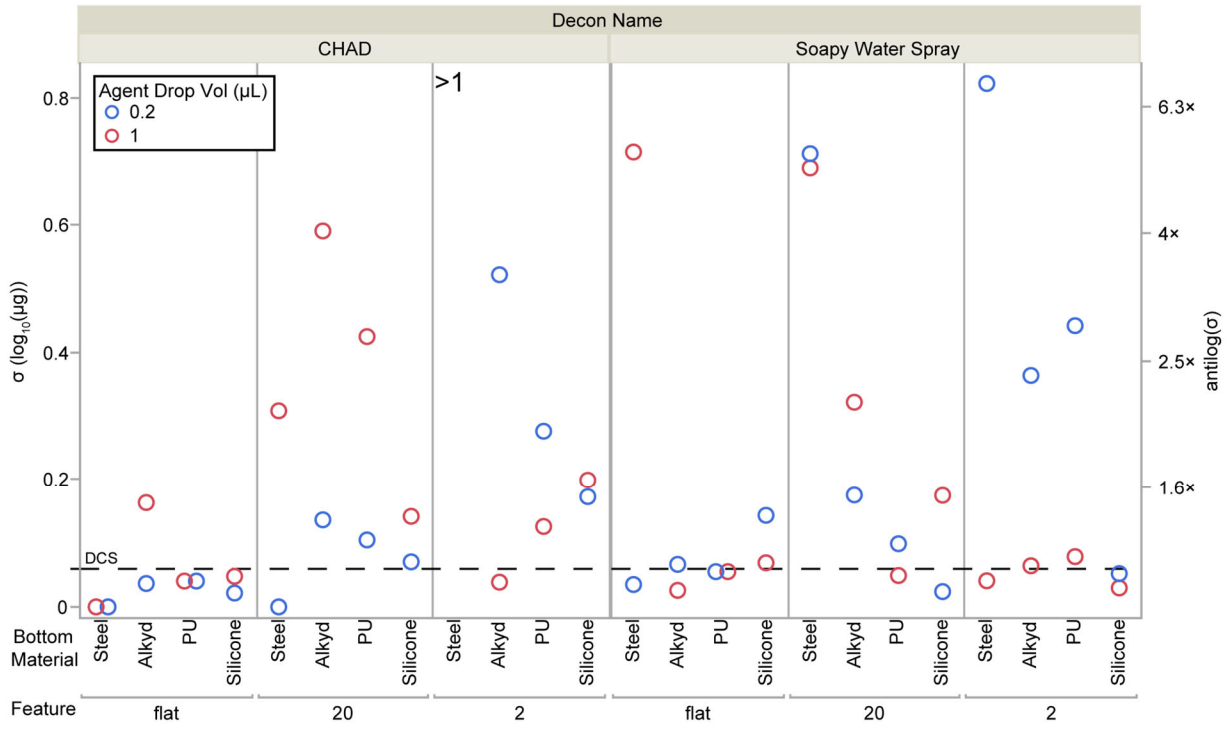


Figure 57. Standard deviation of the log transformed RA data (σ), for 0.2 and 1 µL drops across materials, features, and treatment procedures. The dashed line represents the average σ for DCS of 0.06 $\log_{10}$(µg).

The σ can be transformed to provide a coefficient of variation, CV_{log}, as described in Appendix B. Some of the CV_{log} values are very large and their context is best interpreted in conjunction with µ ± σ, as seen in Figure 58. The larger CV_{log} values are associated with steel. A

generalized observation from hazard mitigation testing is that efficacy and variability are related to the agent distribution when the treatment is performed. For steel, the agent does not sorb into the material but rather resides as a liquid on the surface. Most decontaminants remove the majority of agent from the material, but agent interactions with surface roughness and scratches may retain agent and produce significant variability. In contrast, the other more sorptive coatings and elastomer have (reproducibly) retained some quantity of agent in the material. The same interactions with surface features may occur for any liquid agent on any material, but the (more reproducibly) sorbed agent dominates the RA mass, providing much lower variability.

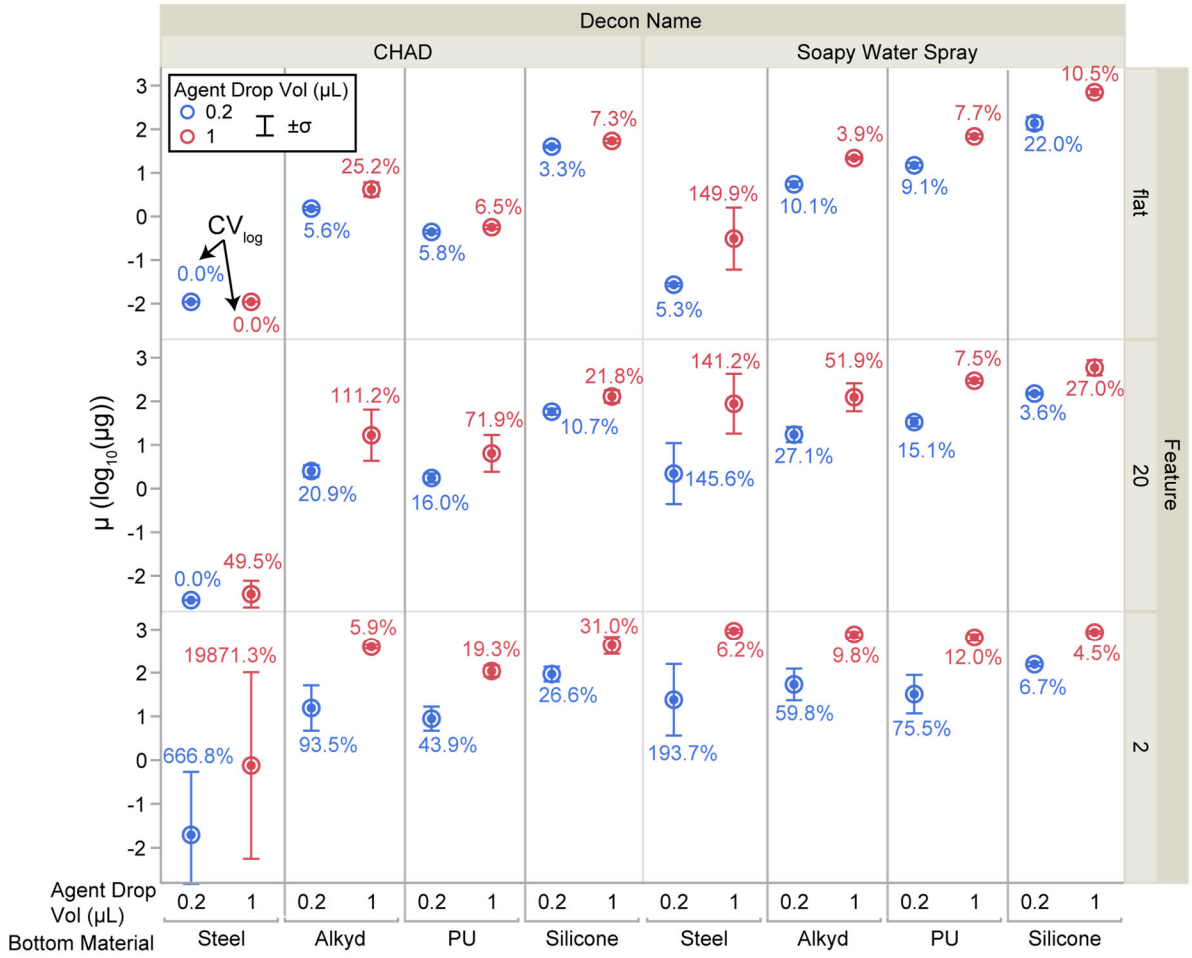


Figure 58. Magnitude and variability of the RA data shown as μ ± σ, text values provide the CV_{log}.

As a reference, the average CV for the positive control samples (Section 4.3) was 8%. This represented the variability of delivering agent to the sample, extracting the sample, and quantifying the mass of agent using chromatography. The difference between the positive control samples and the treatment process samples is primarily the treatment process, the change in RA variability is associated with the generation of a reproducible agent distribution and the effect of the treatment process.

The variability of the RA data may be different for each agent-material-feature-treatment process combination. This is associated with how the agent interacts with the material

and features if present, and how the treatment process accesses the resulting agent distribution when the treatment process is conducted. For example, if the spray duration, pressure, or angle were altered for the soapy water spray, a different CV may be observed. If the internal air velocity of the CHAD treatment were altered or if a different treatment duration were used, a different CV may be observed. The methodology was designed to allow variable treatment configurations and application techniques to be defined (or optimized) by the technology developer or tester.

7.5 Significance of Entrained Agent

These data provide a quantitative demonstration of the influence that entrained agent distributions have on post decontamination exposure sources. As a data summary, Figure 59 shows the RA data for the 1 µL drop volume across materials and features. The values in parentheses show the factor difference between the shim panel conditions and flat panel results. For example, for steel with soapy water spray, the 2 mil shim retains 1973 times the quantity of HD as the flat panel. This illustrates the importance of characterizing entrained agent effects. On a per drop basis entrained agent distributions generate up to 1973 times the quantity of agent observed on flat panels. It is vital to capture these effects to provide more accurate representations of the residual agent that may be present for full-scale asset contamination.

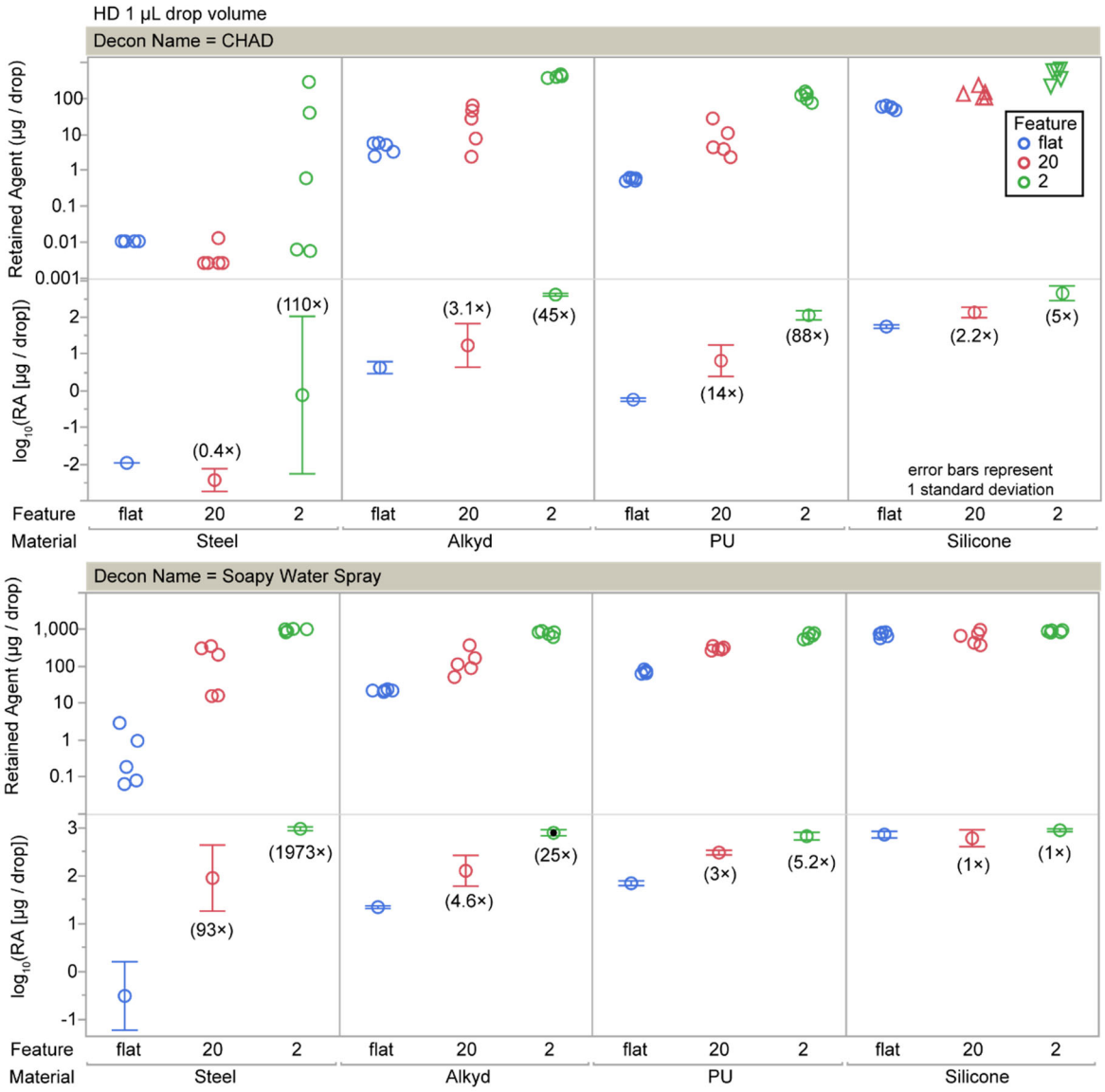


Figure 59. Source term summary for 1 µL drops across materials and feature sizes for CHAD (top) and Soapy Water Spray (bottom). Values in parenthesis are factor differences (i.e., how many times greater) for the shim panels from the equivalent flat panel condition.

7.6 Source Term Fitting Accuracy - RMSE

The RMSE provides a way to compare the ability of the fit techniques to describe the laboratory data. The RMSE describes the average error between the fit and the data that is a composite of fitting error and the variability of the data. A better fit will have lower RMSE values, with a lower bound of σ of the RA data. A comparison of the RMSE of the fit and indirect techniques using different drop volumes is provided in Figure 60. The σ value (purple) represents the standard deviation for the 1 µL drop volume, whereas the RMSE represents the average deviation across all drop volumes. As a general trend the blue line representing the fit technique tends to be the lowest RMSE value, usually close to the σ value. The indirect

technique using 1 µL drops tends to have similar or slightly greater RMSE than the fit, and the indirect technique using 0.2 µL drops tends to have the highest RMSE. A statistical summary of the RMSE values group by treatment and feature size show these trends in Table 64. From these trends, the fit technique typically provides the more accurate description of the data (lower RMSE), and the fit technique provides values very close to the 1 µL drop σ value (i.e., produces values similar to the data). The indirect technique using 1 µL drop volumes tends to provide larger RMSE values, with the 0.2 µL drop volume data producing the largest RMSE (worst fit). A more detailed investigation is provided in Appendix G.

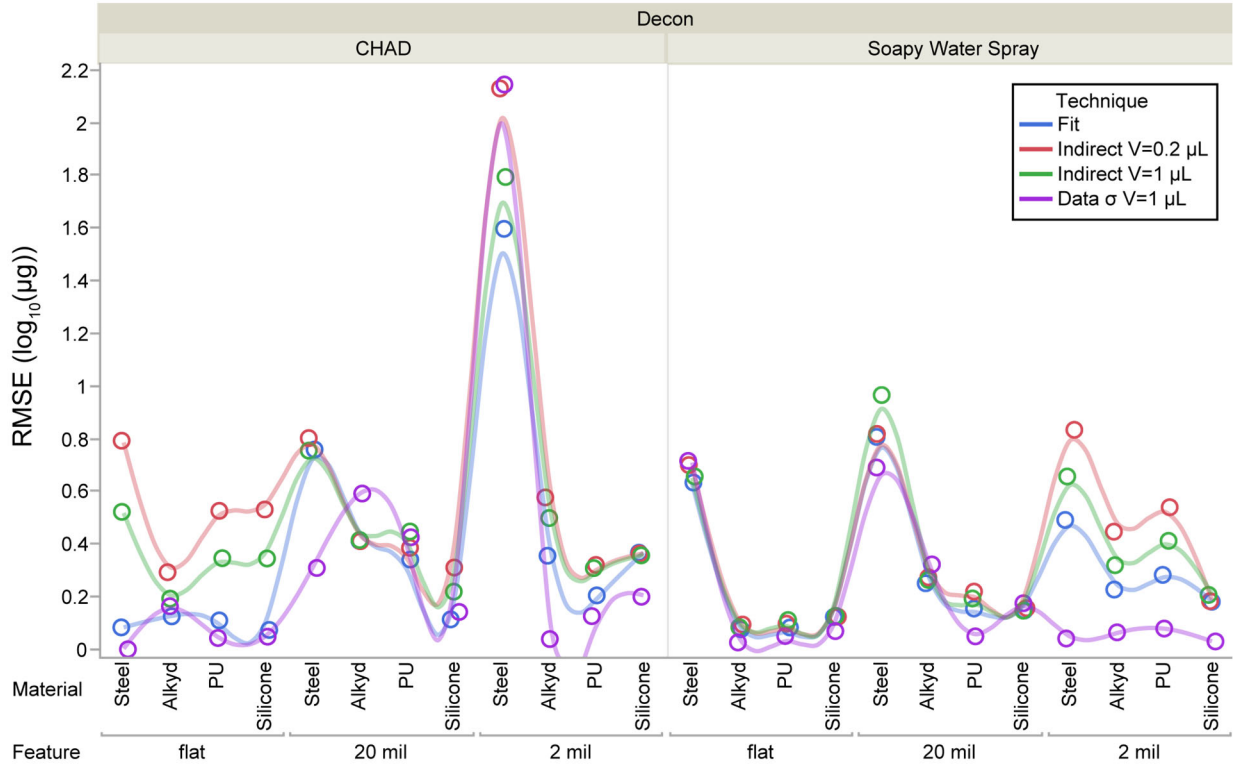


Figure 60. RMSE trends for each analysis technique across materials and features (lower RMSE indicates a better fit).

Table 64. Summary of mean RMSE values ($\log_{10}(\mu g)$) across the materials for each treatment-feature combination for the different analysis techniques

Treatment	Feature	Indirect V=0.2 µL	Indirect V=1 µL	Fit	Data σ V=1 µL
Applied Mass	DCS	0.05	0.05	0.05	0.07
CHAD	flat	0.54	0.35	0.10	0.06
	20 mil	0.48	0.46	0.41	0.37
	2 mil	0.85	0.74	0.63	0.63
Soapy Water Spray	flat	0.25	0.24	0.23	0.22
	20 mil	0.37	0.39	0.34	0.31
	2 mil	0.50	0.40	0.30	0.05

7.7 Summary

This section conducted a variable agent drop volume analysis for two treatment procedures on three materials with flat and shim panels. The introduction of the shim panel methodology provides a first-time capability to measure variable drop volume effects on flat panels and entrained agent in shim panel features. These data demonstrated that the per drop regression approach provides a good fit to both flat panel and shim feature results. The output of the source term fit techniques will be used in the asset-level scale-up calculations in Section 11.

The fit and indirect analysis was conducted for each material-feature-treatment combination and the per drop normalization was shown to provide a good empirical fit to the data. These data illustrated validation goal 1.2 to perform shim panel testing on multiple agent-material combinations.

The CV_{log} was characterized for each material-feature-treatment combination and identified trends related to shim size, with flat panels having the least variability (e.g., CV_{log} of 4–25% for coatings or silicone), with steel presenting larger CV_{log} values up to 150%. Shim panel results had higher RA values and sometimes larger CV_{log} values from 6–100% for coatings or silicone. In the case of the steel 2 mil shim with CHAD (CV_{log} of 19000%) it seems the vaporous technology either completely removed agent from the feature, or did not access the agent in all cases. In contrast, with soapy water spray the steel 2 mil shim was much more reproducible (CV_{log} of 6.2% for the 1 µL drop) but minimal efficacy was observed (i.e., most of the agent remained in the feature).

These types of observations are a demonstration of how the variability of the RA output of the methodology may be different across agent-material-feature-treatment combinations. The variability (as measured by σ) of the RA response is a characteristic of the agent-material-feature the ability to generate a reproducible agent distribution and the treatment process.

8. REPRODUCIBILITY ESTIMATION VIA INTERMEDIATE PRECISION STANDARD DEVIATION

One part of implementing a test methodology is to evaluate the ability to generate the same value upon repeated measurements. The method was evaluated by determining the repeatability and intermediate precision standard deviation (IPSD) for each sample type (DCS, and RA) at multiple test conditions for tests conducted over multiple days within the same laboratory. The output of this analysis provides a value that indicates how similar test results were within a test day and from day-to-day.

Standard statistical methods were used to calculate the single-laboratory precision for the RA analysis method using the intermediate-precision standard deviation (IPSD) method, detailed in ISO 5725-3:1994.[22] The IPSD method was used to calculate the standard deviation of the RA data when executed by a single laboratory, where certain parameters were held constant and others were allowed to vary. Parameters held constant were the laboratory, operators, and test equipment. Parameters allowed to vary were the test day and the analytical calibration curves, because a new calibration curve was generated for each test day. This IPSD calculation approach has been used in several recent assessments for coatings agent resistance[6] and the low volatility agent permeation (LVAP)[23] methods. The IPSD calculation was developed for normal distribution data, the use of log transformation for method outputs has required additional analysis not covered in ISO 5725.

The methodology enables the characterization of the efficacy of hazard mitigation technologies implemented as prescribed by technology developers. The prescribed application procedure may or may not produce the same variability for all panel configurations. The methodology will estimate the magnitude (μ) and the variability (σ) of the RA for the prescribed application procedure so that the asset level response and variability may be estimated. Alternately, the methodology could be used to refine the technology application process or formulation to improve efficacy *and* variability. The results from Section 4 demonstrate that agent contamination can be reproducibly applied, extracted, and quantified. The results presented here focus on the IPSD of measurements for evaluation of the soapy water spray and CHAD treatment procedures for 0.2 and 1 µL drops on the materials of alkyd coating and steel for each feature type (i.e., flat, 20 mil, and 2 mil) for HD. Testing was conducted such that the 0.2 and 1 µL drop conditions were tested with five replicates each, on five test days.

8.1 Definitions for IPSD Calculations

To facilitate discussion of the standard deviation of the test method, the definitions of the specific technical terms are provided here, as they apply to this test.

> **Repeatability (S_r)** is the within-test-day standard deviation of responses for measurements made under repeatability conditions. Repeatability conditions are multiple responses (i.e., replicates) from within the same test day, where operators, laboratory, equipment, and calibration remain constant between measurements. The S_r calculated for this study was generated using only a single laboratory and is therefore referred to as a "single laboratory" S_r to distinguish it from the more comprehensive S_r estimate that may be obtained from multi-laboratory studies.

Between-test-day standard deviation (S_L) is the between-test-day standard deviation for measurements made in a single laboratory across test days and calibrations. The conditions that remained constant between test days were the operator and test equipment. The S_L captures the variability in measurements attributable to changes in testing from day to day and does not account for variability within the same test day, such as random error.

Intermediate-precision standard deviation (IPSD) is the standard deviation of responses for measurements made where some factors are allowed to vary, but the laboratory remains constant. Conditions that remained constant were the operator and test equipment. The IPSD accounts for variability of measurements within a single test day and day to day, based on the following relationship:

$$IPSD = \sqrt{S_r^2 + S_L^2} \qquad \text{Eq. 20}$$

Reproducibility (S_R) is the standard deviation of responses for measurements made *from different laboratories*. The data in this methodology assessment report is from a single laboratory. The day-to-day variations measured in this report correspond only to IPSD.

8.2 Calculations

The validation data was consistent with the ISO 5725 procedures for estimation of IPSD as described in ISO 5725-3,[22] Section 8.0, "Within-Laboratory Study and Analysis of Intermediate Precision Measures", and subsection 8.2, "An Alternative Method". This study focused on the effect of test day, whereas equipment and operators were not changed. In ISO 5725 terminology, the IPSD would be labeled as $IPSD_{(T)}$, with the subscript referring to test days.

Equation 11 in ISO 5725-3, Section 8.2.2 for the calculation of IPSD requires balanced data sets, with the same number of replicates per day. This formula is not applicable to non-balanced data sets because it is unable to account for variable degrees of freedom per test day. Equation 11 and the other ISO 5725-3 formulas for the calculation of S_r, and S_L are derived from the basic statistical model given in ISO 5725-3, Section 6.1. This basic statistical model in ISO 5725-3 is a random-effects model with the laboratory, test day, operator, calibration, and equipment serving as random factors. Therefore, the precision estimates are calculated directly by fitting this random-effects model to the data using the residual maximum-likelihood (REML) method. The REML method is more appropriate than the expected mean squares (EMS) method described in ISO 5725-3 because the validation data set is unbalanced.[17] The precision estimates are given by the variance components after a random-effects model is fit using JMP 17 (SAS Institute; Cary, NC) "Fit Model", with the test day designated as a random effect. For each material-feature-treatment set in this study, the following equations were used†††:

$$S_r = \sqrt{Residual\ Variance\ Component} \quad \text{Eq. 21}$$

$$S_L = \sqrt{Test\ Day\ Variance\ Component} \quad \text{Eq. 22}$$

$$IPSD_T = \sqrt{Total\ Variance\ Component} \quad \text{Eq. 23}$$

The ISO 5725-3 methods assume the data are normal. The skewed distributions observed in RA testing would cause inflated estimates of variance. Therefore, the IPSD calculations were conducted on the log-transformed RA data. The IPSD and related values are often presented as a percentage of the mean value response, similar to a CV. However, the use of log-transformation of the data alters the interpretation. The standard deviation of log transformed data is proportional to the CV without dividing by the mean value (see Appendix B). Therefore, the S_r, S_L, and IPSD are provided as standard deviation values in log units, the CV_{Log} (Eq. B-6) is provided as a proportional indication of data variability.

††† The ISO method normalizes the variance components by dividing by the mean value. The use of log-transformation for the inputs results in using the variance without dividing the variance by the mean.

8.3 Statistical Outliers and IPSD Results

The IPSD was calculated with and without outliers. ISO 5725-3 guidance specifies using a Grubbs method to remove statistical outliers. Given the limited number of test days, the IPSD was also calculated with all data included. This approach was used to maintain compliance with the ISO method. The estimates are in terms of percentage of the average response and the results treated as a normal distribution (using arithmetic means). Note: other analysis conducted in this work assumes log-normal distributions.

8.4 IPSD Testing Over Multiple Days

Validation goal 1.4 is to quantify the IPSD of the same condition repeated over multiple days (i.e., a single laboratory reproducibility assessment). To assess IPSD, a test involving different types of panels made of steel and an Alkyd coating (Table 65) contaminated with HD and decontaminated with two different treatments was repeated for a total of five (5) test sessions. Based on the positive control results from Section 4, the ability to deliver drops across days is reproducible.

Table 65. Test conditions for IPSD determination across multiple test sessions

Parameter	Conditions	Comments
Materials	Steel, Alkyd coating	Representative materials from assets to be used in calculations including impermeable, wetting, and non-spreading
Agent	HD	
Age Time	60 min	Typical contamination duration.
Panel variations	Flat Panel, 0.002, 0.020 in	Sample flat panel and relevant features
Drop count ×volume (µL)	1 × 1 µL	single drop application using syringe pump
Contamination Temperature	21 °C	Standard ambient temperature
Treatment	1) Soapy water spray followed by water rinse (30 min residence) 2) CHAD treatment (2 h at 76 °C, 95% RH)	The soapy water spray resided on the panel for 30 min before the rinse procedure. The soapy water was sprayed with the Preval sprayer for about 5 s from the side of the panel.
Analysis Method	RA test	Output would include RA for each panel, and DCS per test session.
DCS	5 replicates	Single drop volume test used 5 replicates to confirm mass delivered
Replication	5	5 panels per test condition
Test Sessions	5	Each condition of 5 replicates was repeated in a total of 5 different test sessions

The results for the CHAD and soapy water spray treatment process are provided in Figure 61 and Figure 62, respectively. The results are tabulated for CHAD in Table 66 and for soapy water spray in Table 67. Visual inspection of the data shows overall similar results across test days and while some cases show significant variability (e.g., steel 2 mil shim with CHAD), there is *similar variability* across test days. Other cases, such as the alkyd 2 mil shim with soapy water show low variability within and across test days, likely because the agent was inaccessible to the treatment. The similar small variance in some cases and large variance in other conditions

illustrates how the methodology is characterizing the variability of the treatment process for different material-feature combinations.

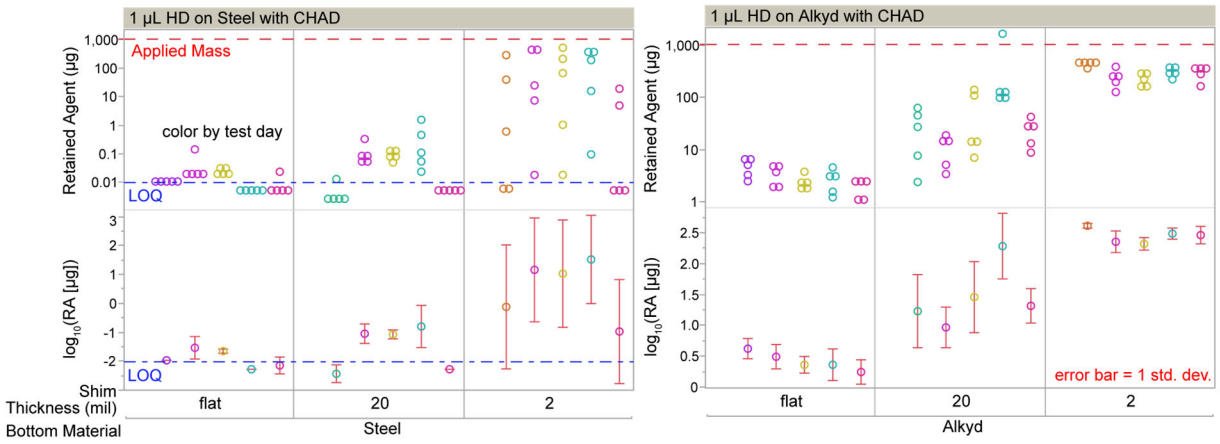


Figure 61. RA across test days for CHAD with 1 µL HD drop volume. Each color represents results collected on a different test session. Individual sample RA results are provided in the top graph, the bottom portion of each graph illustrates the arithmetic mean of the log-transformed RA. Error bars represent one standard deviation of the log-transformed RA.

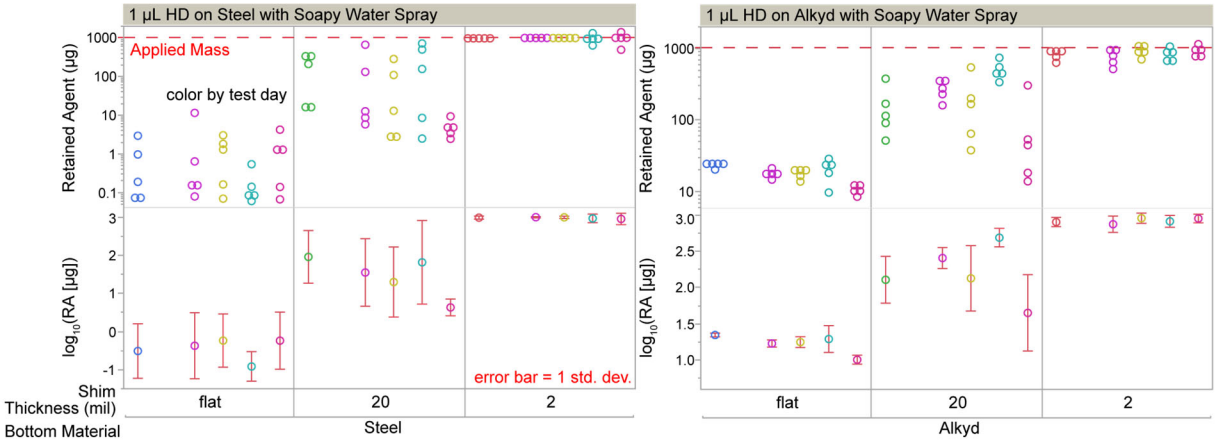


Figure 62. RA across test days for Soapy Water Spray with 1 µL HD drop volume. Each data grouping/color represents results collected on a different test session. Error bars represent one standard deviation of the log-transformed RA.

Table 66. Statistical summary of multi-day data for CHAD treatment

Bottom Material	Shim Thickness (mil)	Test ID	n	Retained Agent (µg/drop)			log_{10}(RA [µg/drop])		
				Mean	Std. Dev.	CV (%)	Mean	Std. Dev.	CV_{log} (%)
Steel	flat	CAP038	5	0.01	0.00	0.0	-1.97	0.00	0.0
		CAP041	5	0.04	0.06	124.4	-1.53	0.39	64.8
		CAP042	5	0.02	0.00	16.9	-1.65	0.07	10.7
		CAP043	5	0.01	0.00	0.0	-2.28	0.00	0.0
		CAP044	5	0.01	0.01	92.0	-2.15	0.29	46.2
	20	CAP040	5	0.004	0.00	98.0	-2.43	0.31	49.8
		CAP041	5	0.12	0.12	100.5	-1.05	0.34	55.2
		CAP042	5	0.09	0.03	32.2	-1.07	0.15	23.1
		CAP043	5	0.44	0.65	147.1	-0.8	0.73	155.3
		CAP044	5	0.01	0.00	0.0	-2.28	0.00	0.0
	2	CAP039	5	64.71	123.52	190.9	-0.12	2.14	19489.2
		CAP041	5	181.79	239.67	131.8	1.16	1.80	4167.5
		CAP042	5	158.5	216.63	136.7	1.03	1.86	5366.9
		CAP043	5	187.36	178.98	95.5	1.52	1.52	1426.1
		CAP044	5	4.77	8.20	171.7	-0.97	1.80	4167.5
Alkyd	flat	CAP038	5	4.43	1.48	33.4	0.62	0.16	24.6
		CAP041	5	3.36	1.38	41.1	0.49	0.20	31.1
		CAP042	5	2.4	0.81	33.7	0.36	0.13	19.9
		CAP043	5	2.63	1.41	53.7	0.36	0.26	41.0
		CAP044	5	1.9	0.75	39.6	0.25	0.20	31.1
	20	CAP040	5	29.13	25.49	87.5	1.23	0.59	110.9
		CAP041	5	11.29	6.69	59.3	0.97	0.33	53.4
		CAP042	5	54.48	58.92	108.2	1.46	0.58	108.2
		CAP043	5	425.77	701.43	164.7	2.28	0.53	95.4
		CAP044	5	24.05	13.46	56.0	1.32	0.28	44.5
	2	CAP039	5	409.83	36.61	8.9	2.61	0.04	6.1
		CAP041	5	240.6	95.84	39.8	2.35	0.18	27.8
		CAP042	5	213.35	48.68	22.8	2.32	0.10	15.3
		CAP043	5	310.21	62.37	20.1	2.48	0.09	13.7
		CAP044	5	299.84	80.95	27.0	2.46	0.14	21.5

Table 67. Statistical summary for multi-day data for Soapy Water Spray treatment

Bottom Material	Shim Thickness (mil)	Test ID	n	Retained Agent (µg/drop)			log_{10}(RA [µg/drop])		
				Mean	Std. Dev.	CV (%)	Mean	Std. Dev.	CV_{log} (%)
Steel	flat	CAP035	5	0.85	1.23	144.8	-0.51	0.72	151.6
		CAP041	5	2.49	5.01	200.9	-0.37	0.87	217.1
		CAP042	5	1.21	1.20	99.6	-0.24	0.7	144.6
		CAP043	5	0.18	0.20	114.3	-0.92	0.39	64.8
		CAP044	5	1.40	1.68	120.6	-0.24	0.75	162.8
	20	CAP037	5	182.49	161.06	88.3	1.96	0.69	141.2
		CAP041	5	164.24	283.70	172.7	1.55	0.89	227.9
		CAP042	5	82.36	121.28	147.3	1.30	0.92	245.4
		CAP043	5	276.14	317.41	114.9	1.81	1.10	390.1
		CAP044	5	4.79	2.70	56.5	0.63	0.22	34.3
	2	CAP036	5	971.22	89.05	9.2	2.99	0.04	6.1
		CAP041	5	993.69	36.69	3.7	3.00	0.02	3.0
		CAP042	5	988.75	67.76	6.9	2.99	0.03	4.6
		CAP043	5	949.94	239.22	25.2	2.97	0.11	16.8
		CAP044	5	932.67	257.67	27.6	2.95	0.15	23.1
Alkyd	flat	CAP035	5	22.28	1.32	5.9	1.35	0.03	4.6
		CAP041	5	17.09	1.98	11.6	1.23	0.05	7.6
		CAP042	5	17.97	2.95	16.4	1.25	0.07	10.7
		CAP043	5	20.86	7.35	35.2	1.29	0.18	27.8
		CAP044	5	10.25	1.47	14.4	1.01	0.06	9.1
	20	CAP037	5	160.91	128.68	80.0	2.11	0.32	51.6
		CAP041	5	266.45	84.53	31.7	2.41	0.14	21.5
		CAP042	5	201.77	202.36	100.3	2.13	0.45	77.1
		CAP043	5	504.16	152.61	30.3	2.69	0.13	19.9
		CAP044	5	86.73	123.02	141.8	1.65	0.53	95.4
	2	CAP036	5	804.54	112.89	14.0	2.90	0.06	9.1
		CAP041	5	764.62	185.86	24.3	2.87	0.11	16.8
		CAP042	5	911.13	138.82	15.2	2.96	0.07	10.7
		CAP043	5	827.44	153.09	18.5	2.91	0.08	12.2
		CAP044	5	900.47	121.22	13.5	2.95	0.06	9.1

8.5 IPSD Results

The $IPSD_T$ results were calculated using the REML method on the log transformed RA results. The within test day (S_r), between test day (S_L), and total $IPSD_T$ are provided in Table 68 as variance (σ^2) and standard deviation (σ).‡‡‡ The $IPSD_T$ standard deviation was used to calculate CV_{Log} (using Eq. B-6) to indicate the variability as a proportion. The *goal* (Table 4) for the method $IPSD_T$ was < 60% (not an acceptance criterion). Four of the 13 conditions investigated met the goal. These results provide insight regarding the method outputs, some material-feature-treatments are more reproducible than others.

Table 68. REML method output for $IPSD_T$ on log-transformed RA data

Treatment Process	Bottom Material	Feature	Variance σ^2 ($log_{10}(\mu g)^2$)			Variance Contribution (%)		Standard Deviation σ ($log_{10}(\mu g)$)			$IPSD_T$ CV_{Log} (%)
			S_r	S_L	$IPSD_T$	S_r	S_L	S_r	S_L	$IPSD_T$	
Applied Mass	-	-	0.003	0.000	0.003	96.6	3.4	0.05	0.01	0.05	7.9
CHAD	Alkyd	2 mil	0.014	0.011	0.025	57.4	42.6	0.12	0.10	0.16	24.2
		20 mil	0.184	0.114	0.298	61.7	38.3	0.43	0.34	0.55	99.3
		flat	0.037	0.013	0.051	73.7	26.3	0.19	0.12	0.23	35.2
	Steel	2 mil	3.368	0.402	3.769	89.3	10.7	1.84	0.63	1.94	7666.9
		20 mil	0.152	0.557	0.710	21.5	78.5	0.39	0.75	0.84	203.2
		flat*	0.002	0.089	0.091	2.0*	98.0*	0.04*	0.30*	0.30*	48.3*
Soapy Water Spray	Alkyd	2 mil	0.006	0.000	0.006	100.0*	0.0*	0.08*	-	0.08*	12.2*
		20 mil	0.124	0.124	0.248	49.9	50.1	0.35	0.35	0.50	87.7
		flat	0.009	0.015	0.024	38.2	61.8	0.10	0.12	0.16	24.0
	Steel	2 mil	0.008	0.000	0.008	100.0*	0.0*	0.09*	-	0.09*	13.2*
		20 mil	0.671	0.139	0.810	82.9	17.1	0.82	0.37	0.90	233.5
		flat	0.379	0.036	0.415	91.3	8.7	0.62	0.19	0.64	126.5

*indicates that one or more data points were below quantitation, limiting this analysis
Values in red indicate $IPSD_T$ CV_{Log} was greater than the 60% goal.

The REML calculation quantifies the contributions of variability enabling a characterization of day-to-day stability versus the variability within a test day. Based on an empirical observation of thousands of method validation precision studies over many years, for a stable methodology producing normal data, the S_r value should be about half of the IPSD (within day and between day variability should be of similar magnitudes).[24] If S_r dominates the $IPSD_T$ (variance contribution is much greater than 50%) it is an indication that the particular test condition is highly variable, if S_L dominates the $IPSD_T$ variance contribution then the results

‡‡‡ Note that variance is additive to determine IPSD, whereas standard deviations accumulate as the square root of the sum of the squared standard deviation. Therefore, the relative contributions of S_r and S_L are compared using variance, rather than standard deviation.

may be inconsistent from day-to-day. There was no criterion for the variance contributions, these data are provided as a characterization of the methodology.

For example, the applied mass (i.e., DCS) demonstrated an IPSD CV_{Log} of 7.9%, which is highly reproducible for these control samples. The variance contribution indicates that day-to-day variance contribution (S_L) is very small (3.4%), the same answer is produced from day to day. The majority (96.6%) of the IPSD is from within day variability (S_r). This shows the IPSD and day-to-day stability for delivering and quantifying agent mass.

For the 2 mil Alkyd coating treated with CHAD, the IPSD CV_{Log} was 24.2%. The variance contribution shows similar magnitudes for S_r and S_L, indicating a reproducible and stable method. Changing the shim thickness to 20 mil produced an IPSD CV_{Log} of 99.3% (did not meet the goal). Inspecting the data (Figure 61), the σ for each day is larger and the mean value across days is visually different. These trends are reflected in the larger σ values for S_r and S_L, producing a larger IPSD of 0.55 $\log_{10}(\mu g)$. The variance contribution shows that the majority of variance (61.7%) is observed within the test day and that 38.3% of the variance is observed across test days. The other conditions follow similar trends and are not discussed in detail.

Some conditions illustrate some specific features of the data. The flat panel steel treated with CHAD produced a S_L value indicating very poor day-to-day agreement. Figure 61 shows that this case is dominated by below quantitation limit values. The S_L value reflects slight changes in the quantitation limit that are calculated for each chromatography calibration. The other condition with a larger S_L variance contribution (61.8) was the flat panel alkyd coating with soapy water spray. Inspection of Figure 62 and the S_r and S_L standard deviations show that this condition had unusually small standard deviation within a test day, allowing a S_L of 0.12 log10(µg) to more significantly influence the IPSD.

Figure 63 shows the $IPSD_T$ standard deviation grouped by material-feature. This shows that in many cases, the IPSD is similar across the liquid and vaporous treatment processes. The material-feature combinations tend to dominate the magnitude of the $IPSD_T$. Recall that $IPSD_T$ focuses only on variability, and not the magnitude of the RA (i.e., efficacy). The 2 mil shim may be reproducible, but also tends to present significant quantities of RA (Figure 62). The 20 mil shim tended to exhibit the highest $IPSD_T$. The moderate shim size may be in a regime where the agent distribution generated from contamination and the ability of the treatment process are more sensitive to subtle difference in the application process.

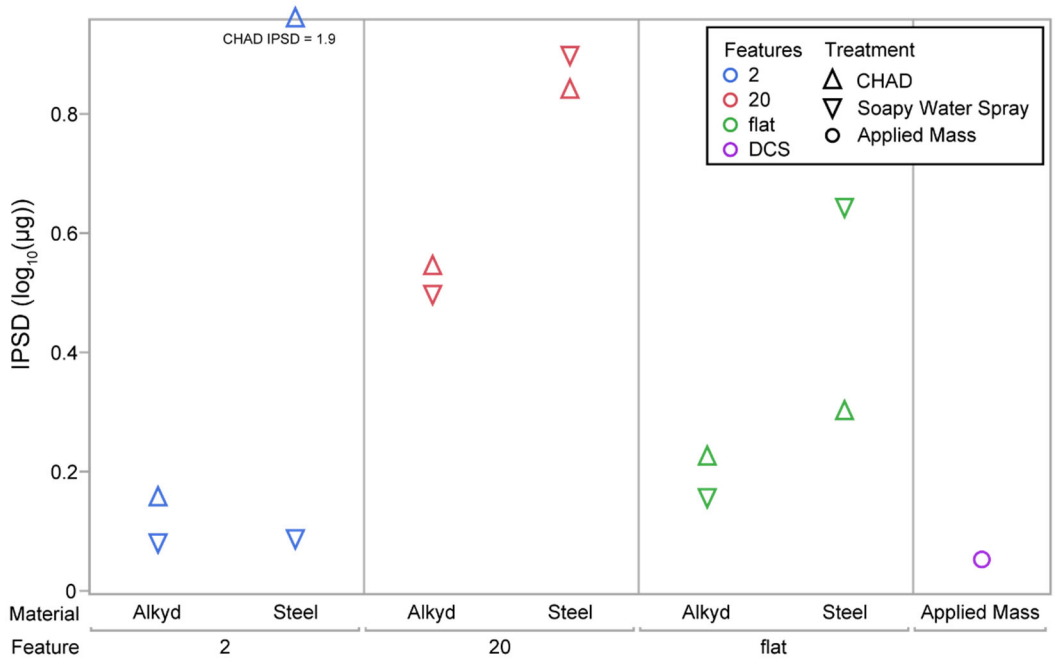


Figure 63. $IPSD_T$ outputs for each material-feature-treatment.

Across the tested conditions the variance contribution shows that the S_L (day-to-day) variance is typically less than the within day variance contribution. The variability of method output seems most correlated with specific material-feature-treatment combinations. Efficacy and variability are related to the agent distribution when the treatment is applied.[3] As treatments access the agent, especially entrained agent, details of the liquid flow are anticipated to influence the extent of removal, producing higher variability in some cases. It is likely that changing aspects of liquid treatment application process, such as sprayer pressure, distance, or angle could significantly influence efficacy and/or variability. Altering vaporous treatment processes such as air flow around the object or treatment duration may influence the reproducibility. In this perspective, the variability of the method response is a characteristic of the treatment steps, the technology, and the application processes used. The scale-up calculation (Section 6) propagates the laboratory-tested variability to the asset level to indicate the distribution of responses that may be observed. Alternately, specific efforts using this RA methodology could be conducted with the objective of reducing the variability to provide more reproducible hazard mitigation technologies and application processes.

The accuracy and precision of final methodology outputs such as M_{Asset} and MPD health-effect outputs is limited by the accuracy of <u>all</u> inputs including laboratory RA source terms *and the details and variability of operational contamination* (e.g., how many agent drops of what volume on what agent-material features). A more reproducible laboratory RA measurement *alone* may not improve the actual accuracy of the asset-level output. However, a more reproducible laboratory RA measurement would indicate a more reproducible treatment process. For example, focusing on the alkyd coating IPSD, the average value across treatments and features is 0.28 $\log_{10}(\mu g)$. For the application of scale-up calculations using the per drop normalization, a day-to-day variability of 0.28 $\log_{10}(\mu g)$, within a factor of antilog(0.28) or factor of 1.9×, may be sufficiently reproducible.

9. MIXED MATERIAL SHIM PANELS

Some asset will include regions of interfaces comprised of different materials and direct contact without an air gap, for example, a weather seal seam with an elastomer to coating interface. This section investigates if mixed material shim panels generated significantly different RA results than similar material shim panels. If a wetting material is present for a mixed material interface, it will dominate how the agent liquid spreads. For decontamination performance, it is anticipated that the gap width, and its influence on flow restrictions, is more influential than wetting characteristics. This could mean that shim panels may be much less influenced by the specific materials of the shim panel, and that not all material combinations need to be tested to represent these feature types on assets.

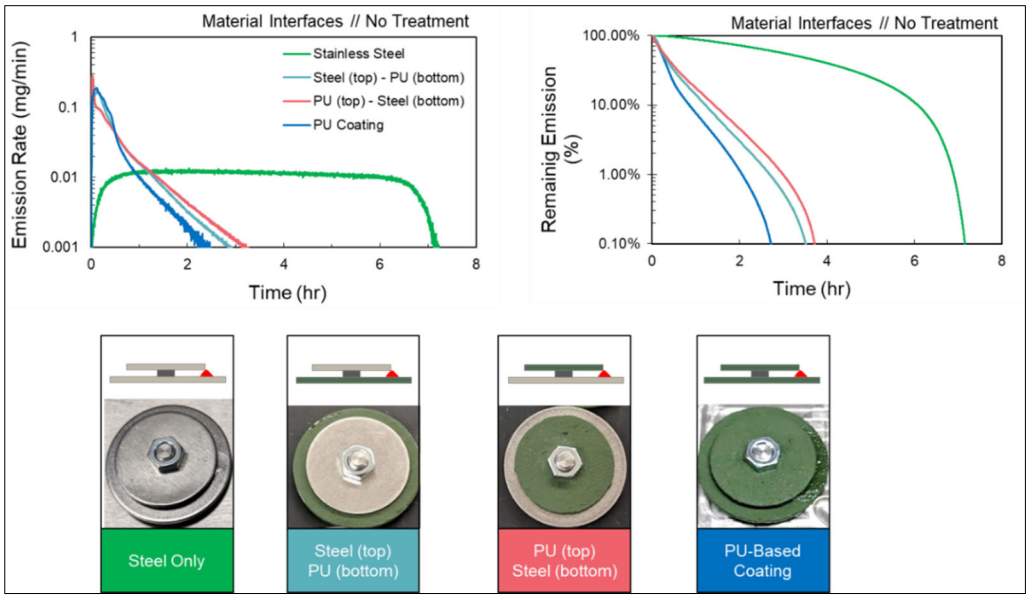


Figure 64. Influence of material interfaces on 2,5 lutidine simulant vapor emission, no mitigation treatment applied (from ref[3]).

9.1 Test Design

The sensitivity of shim panel results to combinations of top and bottom washers of different materials in a horizontal orientation was tested with the conditions indicated in Table 69. The test was designed for comparison to results from Section 7, where similar materials were used on the top and bottom washers. It was postulated that multiple material interfaces may need to be characterized if the LD to single material interfaces is statistically significant and greater than ± 0.4 LD, similar to the sensitivity analysis from Section 5. The data is used to identify if a minimum suggested set of materials can be broadly used to represent many materials.

Table 69. Test parameters to characterize effects of mixed material shim panels.

Parameter	Conditions	Comments
Materials (top washer material- bottom washer material)	Steel-silicone, Steel-PU coating, PU coating-silicone, Alkyd coating-silicone	Combinations of impermeable, elastomer, and spreading and non-spreading coatings
Agent	HD	-
Age Time	60 min	Capillary flow is rapid (<< 1 min) and an impermeable material was used.
Shim Thickness	0.002, 0.020 inch	include all shim sizes
Orientation	Horizontal	-
Droplet count ×volume (µL)	1 ×1 µL applied via M10 pipette	Single droplet application using a standard PDP
Temperature	21 °C	Ambient laboratory conditions ±3 °C, RH not controlled in hood environment
Treatment	1) Soapy water spray + Post-water rinse 2) CHAD (2 h, 76 °C, 95%RH)	-
Analysis Method	RA test	-
DCS	5 replicates	Single drop volume test use 5 replicates to confirm mass delivered
Replication	5	-

9.2 Results

The RA results for the mixed material and single-material shim panels with Soapy water spray are provided in Figure 65. The silicone-steel combination was tested twice, providing a total of ten replicates. Visual inspection of the data shows minimal differences from modifying the top material. However, the PU-Steel 20 mil shim combination exhibited significantly greater variability than the PU-PU shim panel. The LD comparing shim panels constructed of similar materials to different materials is provided in Figure 66. The stars in Figure 65 indicate the materials originally tested in Section 7, specifically, silicone was always tested with a steel top material (i.e., no silicone-silicone shim panels) and is used as the reference value for the LD calculations. None of the materials was greater than ± 0.4 LD and statistically significant.

Figure 65. Mixed material shim panel results for 1 µL drops of HD with soapy water spray. Top row provides the individual RA data points. The bottom row provides the mean and standard deviation of the log-transformed RA data. Star denotes the condition for similar materials.

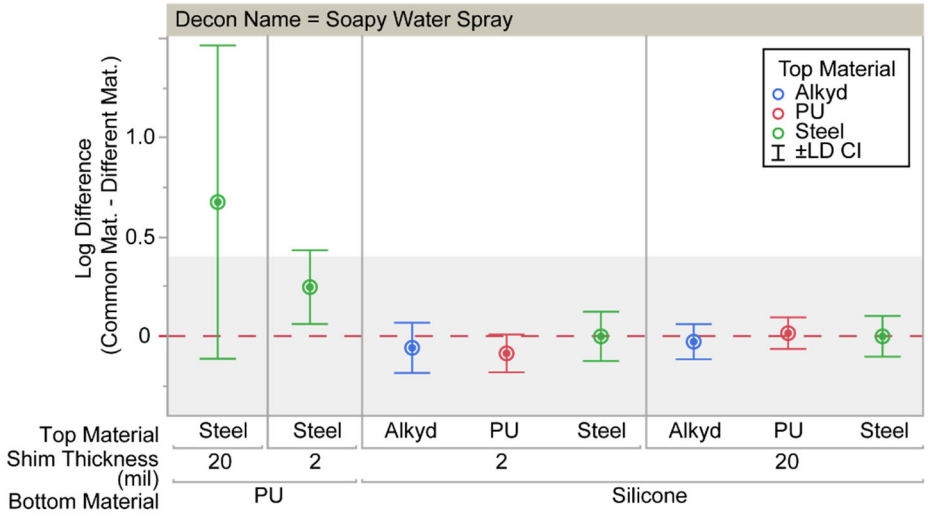


Figure 66. LD comparison of RA for common material (e.g., PU bottom-PU top) to mixed material (e.g., PU bottom-steel top) shim panels. The silicone shim panels always used steel for the top material which is used as the common material. The grey region illustrates the ± 0.4 LD difference threshold.

The RA results for the mixed material and single material shim panels with CHAD are provided in Figure 67. Visual inspection of the data shows minimal differences from modifying the top material. The LD comparing shim panels constructed of similar materials to different materials is provided in Figure 68. None of the materials was greater than ± 0.4 LD and statistically significant. For steel, the CI includes zero which indicates that the two conditions are not significantly different.

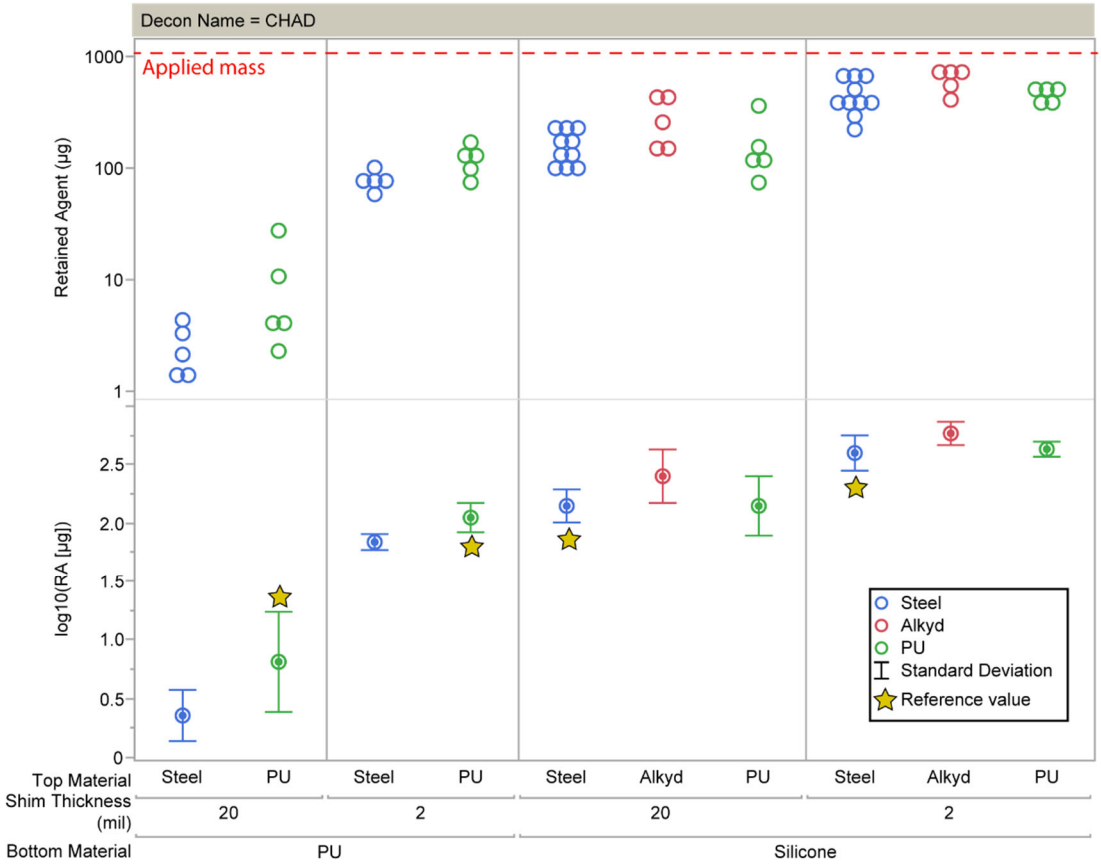


Figure 67. Mixed material shim panel results for 1 µL drops of HD with CHAD. Top row provides the individual RA data points, bottom row provides the mean and standard deviation of the log-transformed RA data.

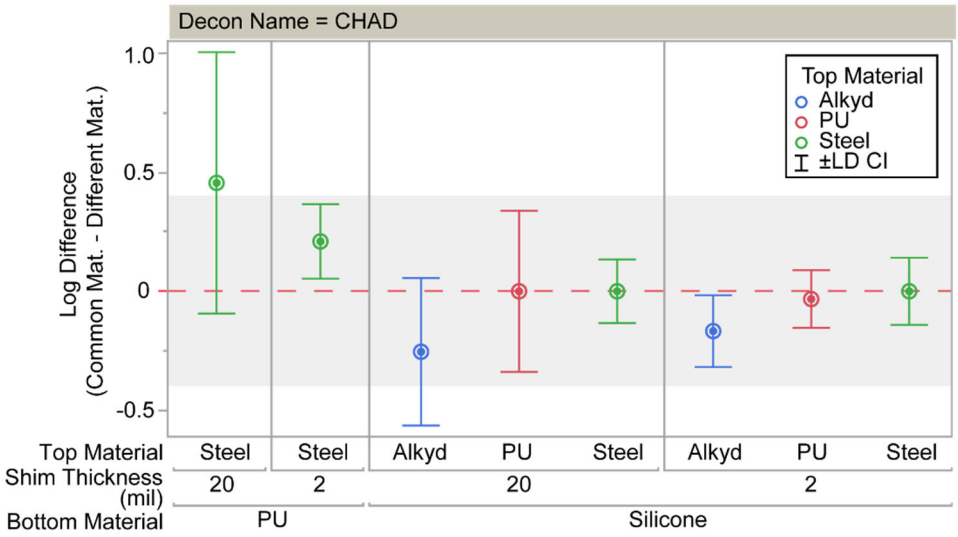


Figure 68. LD comparison of RA for common material (e.g., PU bottom-PU top) to different material (e.g., PU bottom-steel top) shim panels. The silicone shim panels always used steel for the top material which is used as the common material. The grey region illustrates the ± 0.4 LD difference threshold.

These results have demonstrated that shim panels with washers constructed of dissimilar materials did not provide significantly different RA mass than shim panels of similar materials. The different material condition would apply to asset features such as weather-stripping interfaces. Previous work indicated that the more wetting material likely dominated the agent response, and that the more wetting material should be used to represent the feature. In cases where wetting is not known or significantly different, using the higher RA/drop material is recommended.

10. ASSET TESTING RESULTS

Validation Goal 3 is to demonstrate the accuracy of using laboratory panel data to represent an asset with the RA-based asset level outputs. This section describes the testing of the 'asset' that will be used as a reference to determine the accuracy of the Output B calculations (RA-based asset level calculations, as illustrated in Figure A-1). Calculating the accuracy of Output B relative to the results from the tested asset will be the validation of the output calculation. The sample size for testing each drop configuration and asset is seven (7), which is the practical limit for testing items of this size in one session for the DEVCOM CBC laboratories. The test conditions are provided in Table 70.

Table 70. Test conditions for asset validation testing.

Parameter	Conditions	Comments
Assets	Multi-tool, Picatinny rail accessory	Representative materials from assets to be used in calculations including impermeable, wetting, and non-spreading
Agent	HD	
Age Time	60 min	Typical contamination duration.
Panel variations	N/A	N/A
Drop count ×volume (µL)	4 × 1 µL [total volume 4 µL], 5 × 0.2 µL [total volume 1 µL]	Each asset will receive only one drop volume
Contamination Temperature	21 °C	Standard ambient temperature
Treatment	1) Soapy water spray followed by water rinse (30 min residence) 2) CHAD treatment (2 h at 76 °C, 95%RH)	Details provided in the text
Analysis Method	RA test	Output includes RA for each panel, and DCS per test session.
DCS	5 replicates	5 replicates will be acquired for each drop volume used
Replication	7 (per asset, contamination configuration)	Replication based on throughput for testing these size assets in current laboratory configuration

10.1 Asset Contamination and Source Term Selection

The methodology focuses on testing panels, however the testing conducted here uses small assets for the purposes of method validation. Initial efforts to test small assets have been previously developed.[25] One consideration for selecting the assets used here is compatibility with laboratory containers used for RA extraction. The multi-tool is long and narrow and can fit in a centrifuge tube, the Picatinny rail accessory is small enough to fit in a 8 oz extraction jar typically used for 2 in panel extractions.

Data demonstrations have illustrated that drop volume and the type and configuration of materials to be contaminated can be highly influential on the observed results. Therefore, these tests involved contamination of the assets at specific locations with multiple drops of a single volume (i.e., variable drop volume on a single asset was not tested). Two contamination configurations were used involving 4 × 1 µL drops and 5 × 0.2 µL drops. For validation purposes, Goal 3 is to compare the agent retention response for the assets to a sum of

panel-based data based on the asset contamination conditions, including agent drop volume as well as material type and feature. Images of specific contamination locations are provided in Figure 69 and Figure 70. Descriptions of these contamination points are provided in the next section. Gaps in materials were measured using a Starrett feeler gauge set (model number 66, McMaster-Carr, part 2070A8).

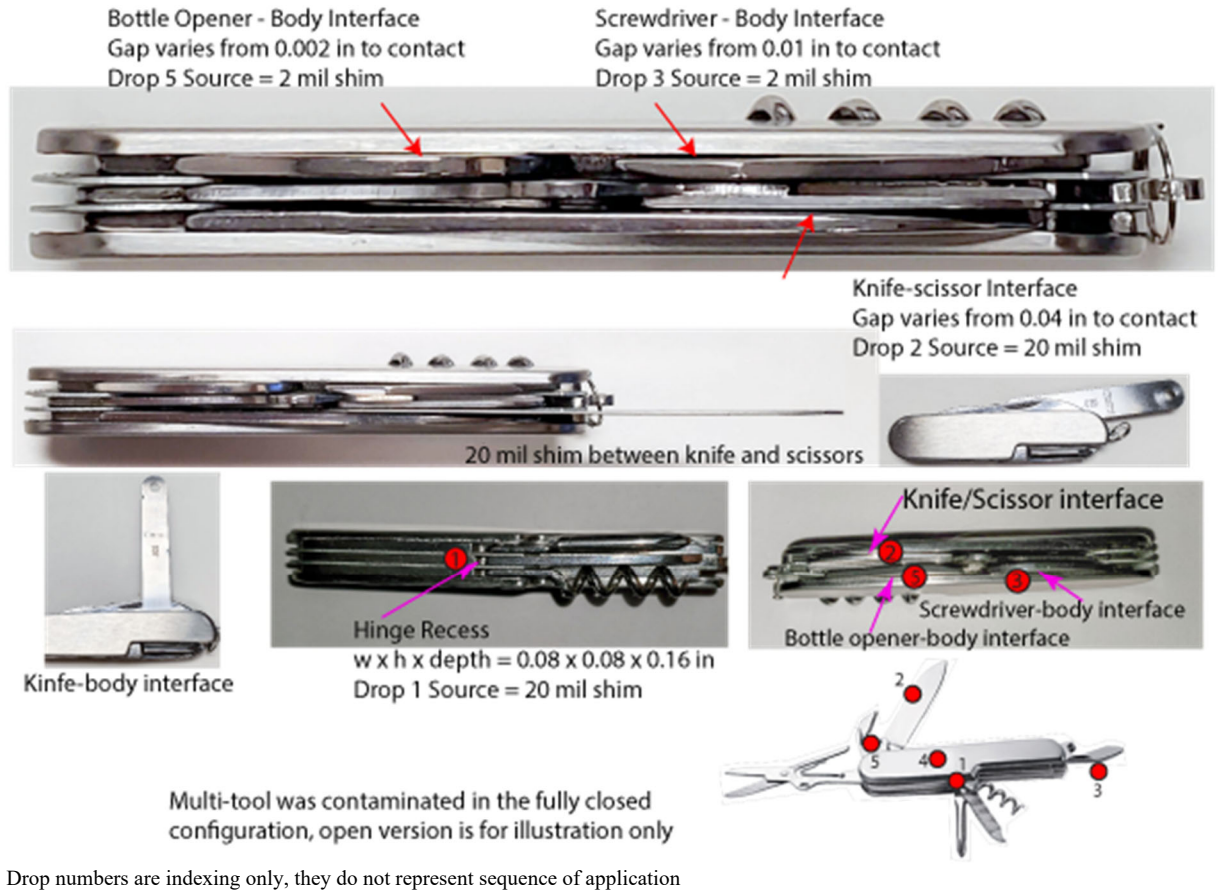


Figure 69. Illustration of locations for drop placement on the multi-tool.

For the multi-tool, each drop was assigned an index number to allocate the appropriate panel material-feature data source term (e.g., material-flat, 0.020 in shim, etc.). For the multi-tool these are identified in Table 71. In the case of the Picatinny rail accessory, each drop was assigned a specific panel configuration, as described in Table 72.

Table 71. Assignment of asset drops to panel source data for the multi-tool.

Asset Drop Index	Description	Panel Source Material	Panel Source Feature	Agent Delivery
1	Hinge recess	Steel	20 mil shim	used for 1 and 0.2 µL contamination
2	Knife-scissor interface	Steel	2 mil shim	used for 1 and 0.2 µL contamination
3	Screw driver-body interface	Steel	2 mil shim	used for 1 and 0.2 µL contamination
4	Grip side	Steel	Flat panel	used for 1 and 0.2 µL contamination
5	Bottle opener-body interface	Steel	2 mil shim	used only for 0.2 µL contamination

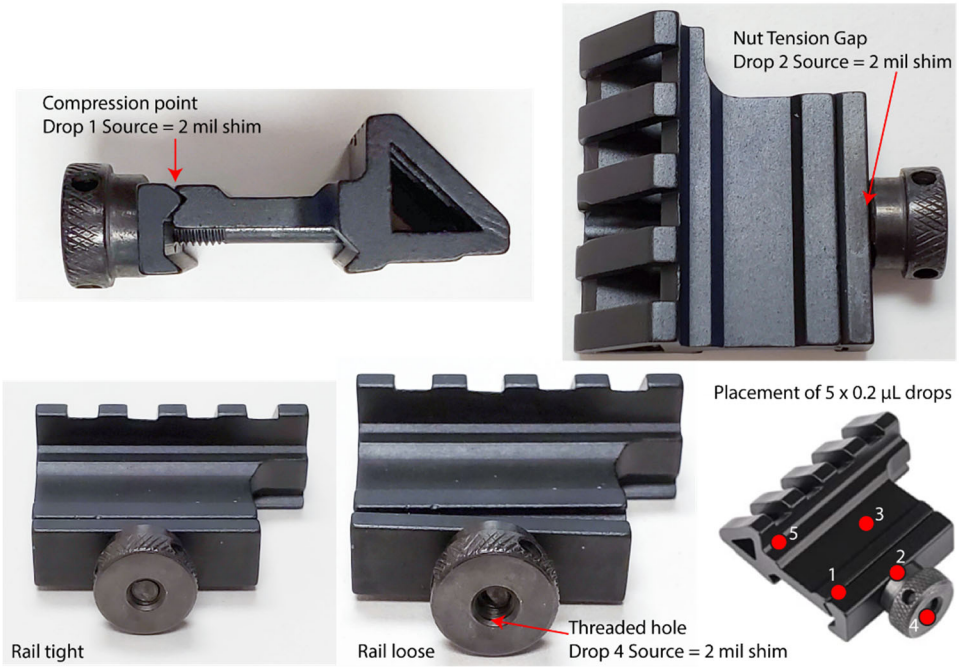


Figure 70. Illustration of locations for drop placement on the Picatinny rail accessory.

Table 72. Assignment of asset drops to panel source data for the Picatinny rail accessory.

Asset Drop Index	Description	Panel Source Material	Panel Source Feature	Agent Delivery
1	Rail gap	Steel	2 mil shim	used for 1 and 0.2 µL contamination
2	Tension knob-screw	Steel	2 mil shim	used for 1 and 0.2 µL contamination
3	Flat surface	Steel	Flat Panel	used for 1 and 0.2 µL contamination
4	Tension knob-cap	Steel	2 mil shim	used for 1 and 0.2 µL contamination
5	Corner	Steel	20 mil shim	used only for 0.2 µL contamination

10.2 Treatment Process

The use of small assets in laboratory testing requires modifications to how decontaminants are applied. In the case of CHAD treatments, the assets were placed in the enclosure. However, the spray application with soapy water required additional considerations. The Multi-tool was held in the vertical position by the key ring on one end of the device. The multi-tool was positioned at an angle to spray the cork screw and the spiked flat side sprayed on one side for 4 seconds from moving the spray applicator from the top to bottom of the multi-tool. The multi-tool was then rotated to spray directly into the knife interface for an additional 4 seconds progressing from top to bottom.

The Picatinny rail was placed in a horizontal position with the tension screw facing to the right. It was sprayed for 4 seconds from front to back with the sprayer at about 30

degrees from the surface normal. The rail was rotated clockwise 90 degrees and sprayed for an additional 4 seconds from front to back.

10.3 Results

The RA results for the assets and each treatment are provided in Figure 71, and Table 73. Both assets are comprised of metal, the multi-tool is made of steel (type unspecified), the Picatinny rail is a black anodized aluminum (type unspecified).

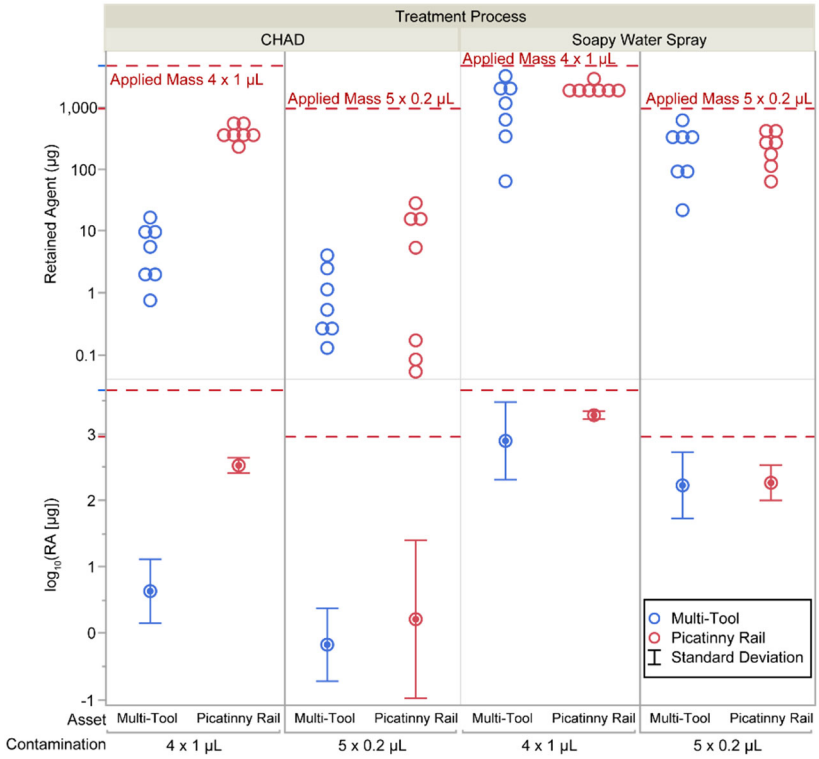


Figure 71. Asset RA results for CHAD and soapy water spray treatments. The top of each graph shows the individual data points, points are jittered in the x axis for better visualization. The lower graph shows the arithmetic mean and standard deviation of the $\log_{10}$ transformed RA.

The reproducibility of the asset results as expressed by CV of the RA data span 14–117% (Table 73). The Picatinny rail with CHAD for 5 × 0.2 µL drops produced data with 4 responses near 10 µg and 3 responses near 0.1 µg. This variability could be a result of the contamination process generating different agent distributions within the complex features of the asset, and/or the ability of the treatment process to access the agent. These data are used as the reference data to evaluate the ability of laboratory panel data to predict asset level responses.

Table 73. Asset RA Summary Statistics

Test ID	Asset Type	Decon Name	Agent Drop Vol (µL)	Agent Drop Count	n	RA (µg)			log_{10}(RA µg)		
						Mean*	Std Dev	CV %	Mean	Std Dev	CV_{log} %
CAP046	Applied Mass	N/A&	0.2	5	4	1015.21	29.33	2.89	3.01	0.01	1.5
			1	4	5	4579.99	90.85	1.98	3.66	0.01	1.5
	Multi-Tool	CHAD	0.2	5	7	1.28	1.49	116.31	-0.17	0.55	100.3
			1	4	7	6.63	5.72	86.31	0.63	0.48	83.7
		Soapy Water Spray	0.2	5	7	256.16	204.09	79.67	2.23	0.50	88.2
			1	4	7	1325.49	1103.49	83.25	2.90	0.58	108.2
CAP047	Applied Mass	N/A	0.2	5	5	1046.23	19.41	1.86	3.02	0.01	1.5
			1	4	5	4181.65	87.95	2.10	3.62	0.01	1.5
	Picatinny Rail	CHAD	0.2	5	7	9.34	10.97	117.5	0.21	1.19	500.7
			1	4	7	346.8	91.71	26.44	2.53	0.12	18.4
		Soapy Water Spray	0.2	5	7	211.26	103.03	48.77	2.27	0.27	42.8
			1	4	7	1935.36	281.49	14.54	3.28	0.06	9.1

*Presented mean value uses arithmetic mean, geometric mean value is calculated and presented in later sections

&Applied mass was determined from DCS rather than a positive control dose of an asset.

11. SCALE-UP CALCULATION ACCURACY ASSESSMENT

As part of validation Goal 3, the $M_{Asset,\ calculated}$ results generated by the scale-up calculation are compared to the $M_{Asset,\ measured}$ results from Section 10. The ratio of predicted / measured asset mass is used to assess the accuracy of the calculation, as described in Section 6.8. Geometric mean values for the calculated and measured M_{Asset} are used to calculate the ratio. R is calculated for eight (8) test conditions including the two assets (i.e., the multi-tool and Picatinny rail), two drop volumes (0.2 and 1 µL), and two treatment processes (soapy water spray and CHAD). Further, $M_{Asset,\ calculated}$ is evaluated for each of the three methods used to calculate the source term inputs for the MC methods as described in Section 6.4 (i.e., fit, indirect using 0.2 µL drops, and indirect using 1 µL drops). The calculation will be considered acceptable if R is within the range of 0.1–10. The asset level output calculations including material contribution calculations (Output B_1) and asset level LD (Output B_2) and are demonstrated for each case. These data will be used to demonstrate a health-based MPD (Output B_3) in Section 12.

11.1 Source Term Inputs

Both the multi-tool and the Picatinny rail are constructed of bare metals. Therefore, all source terms used in the calculation are based on results from steel panels (Table 71 and Table 72). While not all metal surfaces interact similarly with agent, treating each metal as stainless steel is a working approximation. The systematic error is expected to be comparable to other sources of error. Each of the calculation methods to determine µ and σ for the MC calculations may have different values based on the assumptions and specific data used. A comparison of these source terms is provided in Figure 72 for CHAD and in Figure 73 for soapy water spray treatments. The graphs display the results after antilog transformation ($\mu^* \pm \sigma^*$, using Eq. B-3 and Eq. B-4). The fit method uses the regression constructed from all 16 data points for the material-feature-treatment and provides the output for the indicated drop volume using Eq. 7. The indirect methods use the drop volume indicated (e.g., Indirect V=0.2 µL uses only the five replicates of 0.2 µL drop test results) to calculate the output at the desired drop volume using Eq. 9. The direct source term output provides the same output as the indirect method when the output drop volume and indirect drop volume (source) are the same (i.e., for an output of 0.2 µL the direct source term is the same as Indirect V=0.2 µL). As seen in the figures, the source term output of each method is slightly different based on the data used and assumptions of the calculations. The following sections characterize the use of these source terms to scale-up the laboratory data to represent the tested assets. Note that there are cases where the laboratory results were all below detection producing $\sigma = 0$, a lower bound threshold $\sigma = 0.05 \log_{10}(\mu g)$ was implemented in these cases.

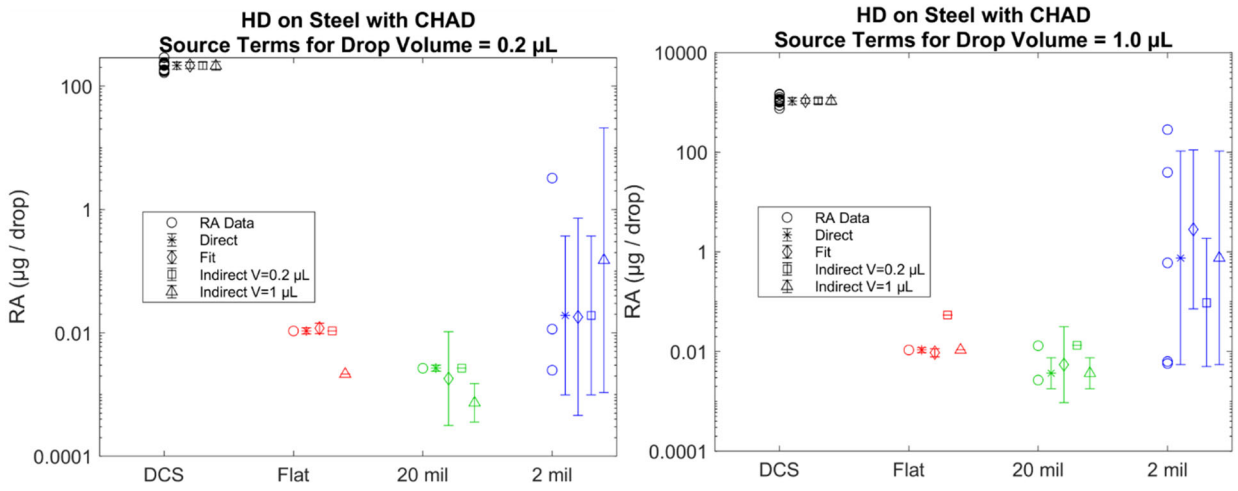


Figure 72. Comparison CHAD treatment µ* ± σ* values for each fit approach for 0.2 µL drops (left) and 1 µL drops (right).

Table 74. Source term input values for µ and σ for each fit type for HD on Steel with CHAD

Drop Vol (µL)	Feature Type	Fit (log_{10}(µg))		Indirect V= 0.2 µL (log_{10}(µg))		Indirect V= 1 µL (log_{10}(µg))	
		µ	σ	µ	σ	µ	σ
0.2	DCS	2.34	0.05	2.33*	0.07	2.33	0.07
	flat	-1.93	0.08	-2.67*	0.05	-2.67	0.05
	20	-2.74	0.76	-3.13*	0.31	-3.13	0.31
	2	-1.74	1.60	-0.82*	2.14	-0.82	2.14
1	DCS	3.03	0.01	3.03	0.06	3.03*	0.06
	flat	-2.03	0.06	-1.97	0.05	-1.27*	0.05
	20	-2.26	0.54	-2.44	0.31	-1.87*	0.05
	2	0.45	1.14	-0.12	2.14	-1.02*	1.29

*Direct fit source terms are equivalent to indirect source terms when the applied drop volume matches the drop volume used for the indirect calculation

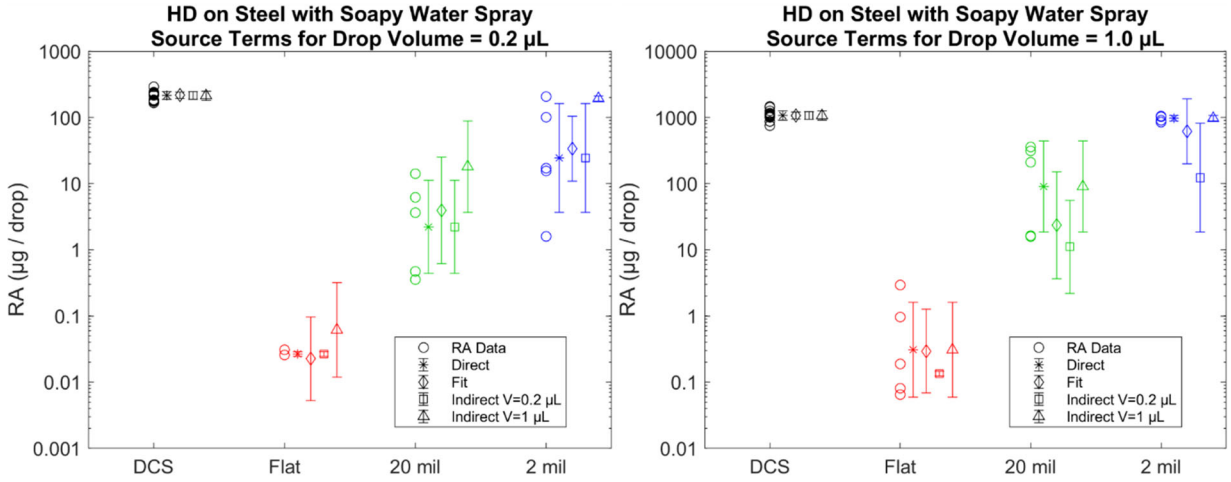


Figure 73. Comparison soapy water spray treatment µ* ± σ* values for each fit approach for 0.2 µL drops (left) and 1 µL drops (right).

Table 75. Source term input values for µ and σ for each fit type for HD on Steel with soapy water spray

Drop Vol (µL)	Feature Type	Fit (log_{10}(µg))		Indirect V= 0.2 µL (log_{10}(µg))		Indirect V= 1 µL (log_{10}(µg))	
		µ	σ	µ	σ	µ	σ
0.2	DCS	2.34	0.05	2.33*	0.07	2.33	0.07
	flat	-1.65	0.63	-1.57*	0.04	-1.21	0.72
	20	0.60	0.81	0.35*	0.70	1.26	0.69
	2	1.53	0.49	1.39*	0.82	2.29	0.04
1	DCS	3.03	0.05	3.03	0.06	3.03*	0.06
	flat	-0.53	0.63	-0.51	0.72	-0.88*	0.04
	20	1.37	0.81	1.96	0.69	1.04*	0.70
	2	2.79	0.49	2.99	0.04	2.09*	0.82

*Direct fit source terms are equivalent to indirect source terms when the applied drop volume matches the drop volume used for the indirect calculation

11.2 Asset Level Results

The results of the scale-up calculation process are presented here for the 0.2 and 1 µL drop volumes for the multi-tool with soapy water spray treatment process. The scale-up process for the other conditions tested, including for each combination of asset, drop volume, and treatment condition, are provided in Appendix H. The overall accuracy assessment is summarized across all conditions for each source term calculation technique in Section 11.3. The source term values used to conduct all calculations were provided in Section 11.1.

11.2.1 Multi-tool Asset with 0.2 µL Drops and Soapy Water Spray Treatment

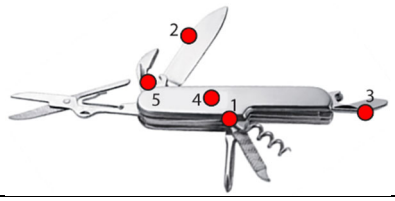
The scale-up calculation uses the sum of the per drop responses to represent the asset, as described by Eq. 4. The laboratory testing exhibited variability of the RA results. Each

drop will contribute a response defined by the measured RA distribution. The implementation of a MC algorithm (Section 6.2) enables randomly sampling from the range of potential RA responses for each drop (described by the source term inputs μ and σ). The script used to perform the MC random sampling is provided in Appendix F. The MC algorithm performs 10,000 iterations of Eq. 4, randomly sampling each drop to generate a distribution of $M_{Asset,Calculated}$ responses.

This MC process is performed for each of the source term calculation techniques (fit, indirect using 0.2 μL drop data, and indirect using 1 μL drop data) to generate an asset level output. The assumptions used for each technique alter the source term inputs, the specific values used for this asset are from Table 75. In this case the Indirect (V=0.2 μL) source term used the same drop volume panel data to predict the 0.2 μL drops placed on the asset. The Indirect (V=1.0 μL) source term used the 1 μL panel data to extrapolate the predicted output for a 0.2 μL drop. The fit source term utilized all replicates of all drop volumes of the panel data to estimate the response for 0.2 μL drops. The accuracy of each source term calculation technique is evaluated comparing the measured asset result to the calculated output.

The MC algorithm aggregates the per drop RA using Eq. 4, the list of material-features and drop volumes used for the calculation is provided in Table 76, based on the detailed description in Table 71. The contamination pattern uses five drops to deliver a total of 1 μL of agent to the asset.

Table 76. Description of drop count and drop volumes used to represent the contamination of the multi-tool asset with 0.2 μL drops

Treatment	Material-Feature	Number of Drops	Drop Vol (μL)	Asset
Soapy water spray	steel-flat	1	0.2	
	steel-20 mil	2	0.2	
	steel-2 mil	2	0.2	
	total	5	1	

The seven replicates of the tested asset (Asset RA, as measured in Section 10.3) and the MC output of the 10,000 iterations for each source term calculation technique are shown in Figure 74. To visualize 10,000 data points a swarm plot is used, where the width of the data represents the density of points, similar to a histogram. A statistical description of these data is calculated by $\log_{10}$ transforming the M_{Asset} value to produce the geometric mean, the PI, and upper TI, shown in Figure 74. The PI error bars for the Asset RA (calculated with Eq. 15) are large due to the lower replication (n=7) of the asset test data. The PI error bars for the fit techniques (calculated using Eq. 14) are smaller, based on the data distributions and the much larger sampling (n=10,000). These differences in sampling also change the relationship of the upper TI predicted, recall that the upper TI (calculated using Eq. 12) indicates that 95% of the sample results should be below the specified value (with 95% confidence). The R value below the PI error bars indicates the ratio of the calculated M_{Asset} to the measured M_{Asset} (as defined in Section 6.8), values closer to 1.0 indicate better fits. These results show that the fit method accurately predicted the M_{Asset} within a factor of R=0.70, the indirect fit using V=0.2 μL data within a factor of R=0.57, the indirect fit using V=1 μL data within a factor of R=4.37. All the fit methods produced results within the acceptance criterion range.

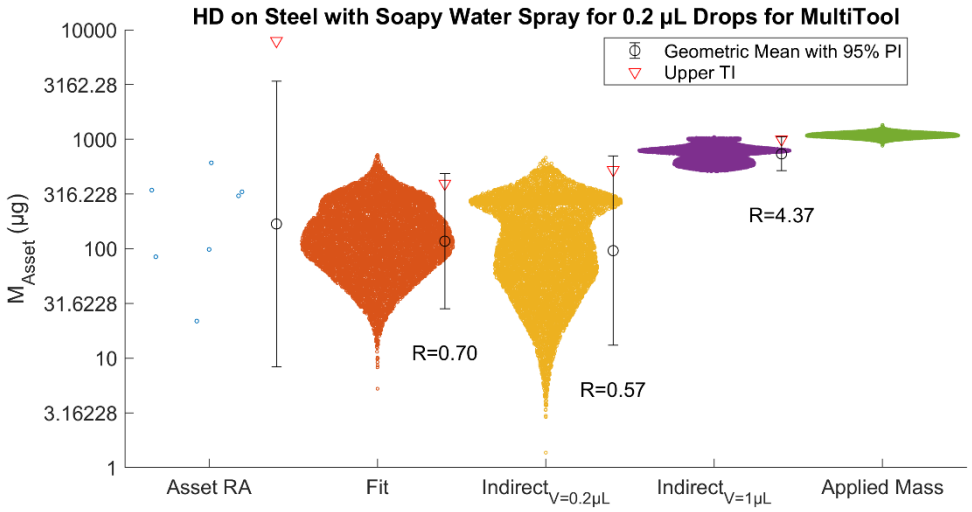


Figure 74. MC output visualization for the multi-tool asset with 0.2 µL HD drops treated with soapy water spray. The circle with error bars to the right of each swarm plot represent the geometric mean and 95% PI for the data.

The tabulated outputs of the results are provided in Table 77. The methodology output B_2 (Figure A-1) of asset level performance as quantified by LD from the applied mass is provided. The calculation of M_{Asset} enables quantifying LD at the asset level, rather than at the per material-feature level. The LD value includes a 95% CI on the difference, recall that a CI is a descriptor for the estimate of the LD and is dependent on sampling. The CI for MC output is much smaller because 10,000 samples were used, compared to the seven samples used to directly measure the Asset RA. For example, these data show that soapy water spray provided 0.81 ± 0.46 LD for the tested asset compared to the applied agent mass, and that the fit predicted 0.97 ± 0.01 LD.

Table 77. M_{Asset} output for the tested asset and calculation variations for the multi-tool asset with 0.2 µL HD drops treated with soapy water spray.

Type	Geometric Mean (µg)	Upper Bound TI (µg)	Upper Bound 95% PI (µg)	μ_{Output} ($\log_{10}(M_{Asset}$ µg))	σ_{Output} ($\log_{10}(M_{Asset}$ µg))	LD ± CI (relative to applied mass)
Asset RA	168.29	7929.55	3393.89	2.23	0.50	0.81 ± 0.46
Fit	117.25	393.25	485.22	2.07	0.31	0.97 ± 0.01
$Indirect_{V=0.2µL}$	96.03	524.46	704.28	1.98	0.44	1.05 ± 0.01
$Indirect_{V=1µL}$	735.79	996.79	1050.75	2.87	0.08	0.17 ± 0.00

The accuracy ratio results (R) comparing each analysis technique to the tested asset are provided in Table 78. For these data the fit regression provided the most accurate estimate of M_{Asset} with an $R_{Absolute}$ of 1.44. For this asset condition all source term techniques met the criterion of $R_{Absolute} < 10$ (R is within 0.1–10).

For context on how well the PI and TI for the calculation techniques represent tested asset, ratio values for the quantity are also provided. Note that the magnitude of the PI and

TI values are influenced by the number of samples used in the calculation (i.e., lower degrees of freedom), thus the Asset RA values are typically greater than those observed from the MC calculations. If the upper bounds are compared to health-based requirements, then some measure of the accuracy should be considered. Here, significant differences are seen in the predicted upper PI and TI, with an R value for the upper TI of 0.05 for the fit method versus 0.13 for Indirect using V=1 µL. No acceptance criteria were established for upper bound estimates.

Table 78. Accuracy ratio of the calculation variations for the multi-tool asset with 0.2 µL HD drops treated with soapy water spray.

Type	R: Geo Mean	$R_{Absolute}$: Geo Mean	R: Upper TI	R: Upper PI
Fit / Asset	**0.70**	1.44	0.05	0.14
$Indirect_{V=0.2\mu L}$ / Asset	**0.57**	1.75	0.07	0.21
$Indirect_{V=1\mu L}$ / Asset	**4.37**	4.37	0.13	0.31

Bold blue values are the accuracy ratio that should be within the validation criterion range of 0.1–10.

There were cases in the MC iterations where the random sampling of a given condition (especially for larger σ values) could produce an RA output that was greater than the applied mass. The MC algorithm checks for this condition and substitutes the unrealistically high value with the applied mass value. This process can create many occurrences with the same high values, which may show up as peaks in the swarm plot. The presence of these peaks could cause deviations from the expected log-normal distributions (e.g., skew and kurtosis effects) and related geometric mean and PI calculations. Additionally, the presence of near zero value σ for below detection results can also influence output distributions that may not be log normal. To build trust in the accuracy of using the geometric mean, a histogram of the results is provided in Figure 75, the red line shows a fitted normal distribution to the log-transformed results. Ideally, the red line should have similar shape and magnitude as the histogram. Deviations of the red line from the histogram indicate that the log-normal distribution may not be an accurate description of the data distribution, and quartile (i.e., non-parametric) analysis may be a better description of the results.

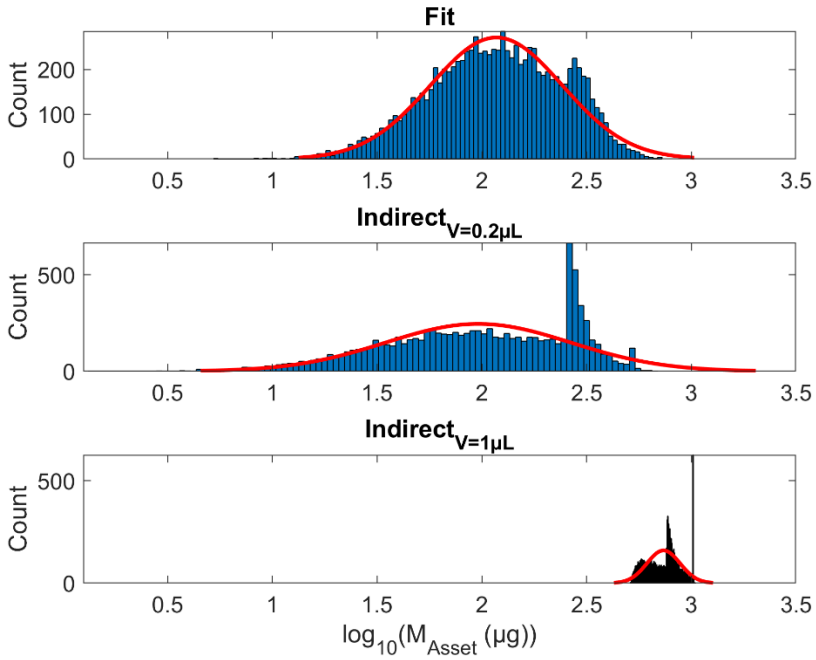


Figure 75. Histogram of the M_{Asset} output fit to a log-normal distribution (red line), for the multi-tool asset with 0.2 µL HD drops treated with soapy water spray

The geometric mean calculation assumes that the data are log-normal, with some deviations from normality seen in Figure 75 (attributed to the non-detect or high value source terms). The MATLAB function *quantile* is used to identify the magnitude of M_{Asset} for specific quantiles such indicating that 95% of the data are less than an indicated value. The quantile output requires more samples than the seven replicates collected for the Asset RA data, thus only the MC output is presented in the quantile output (Table 79).

Table 79. Quantile analysis of the M_{Asset} output for the multi-tool asset with 0.2 µL HD drops treated with CHAD.

Quantile	Fit Analysis (µg)	Indirect$_{V=0.2µL}$ (µg)	Indirect$_{V=1µL}$ (µg)
25%	70.963 (0.6x)	46.775 (0.4x)	630.507 (0.8x)
Median 50%	120.012 (1.0x)	103.988 (1.0x)	765.524 (1.0x)
75%	204.192 (1.7x)	256.276 (2.5x)	828.137 (1.1x)
95%	351.958 (2.9x)	361.151 (3.5x)	1014.490 (1.3x)

Values in parenthesis are the ratio of the value to the median.

The MC calculation tracks the source of agent mass, enabling the attribution of which material-features contributed to the total M_{Asset} output. This output is not possible to generate by testing assets, it is only possible using the MC per drop aggregation algorithm. The tested asset is simple and represented by only three material-feature combinations. The material contribution calculation (Section 6.7) was performed to quantify how each material-feature contributed to the total output. Figure 76 provides the relative contribution for each feature type (i.e., Output B_1 from Figure A-1). Each iteration of the MC algorithm generates different masses for each drop and the total mass, resulting in a different relative contribution for each iteration. A swarm plot is used to indicate the frequency with which a given feature is a significant contributor. The number in parenthesis along the x-axis indicates the median relative

contribution value for the material-feature combination. For example, the steel 2 mil feature was typically responsible for 86% of the M_{Asset} mass, and the steel 20 mil feature was typically responsible for 13.6% of the M_{Asset} mass, and the steel-flat response provided an insignificant (0.02%) contribution to the M_{Asset} mass. A similar analysis can be done without normalization to the M_{Asset} mass to indicate, for example, that typically 87.8 µg of the 117 µg ($M_{Asset,\ fit\ geometric\ mean}$) retained by the asset was attributed to the steel-2 mil feature.

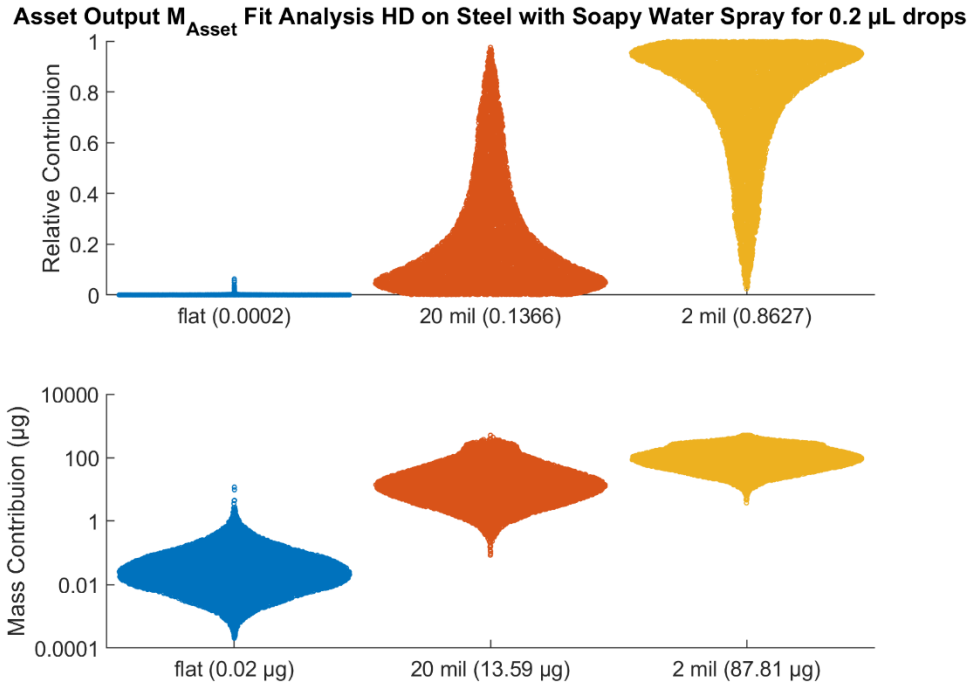


Figure 76. Relative contribution and mass contribution graphs for each feature for the multi-tool asset with 0.2 µL HD drops treated with soapy water spray, values in parenthesis are the median response for the material-feature indicated.

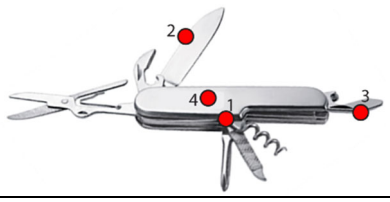
The contribution calculations enable material attribution, here showing that the 2 mil shim panel was by far the major source of RA. These data could be used to identify and prioritize the agent sources on an asset that may require further treatment (e.g., perform more treatment processes on small features), or be used to identify and justify different material choices on assets. The simple materials used for assets tested here make this analysis somewhat trivial, however, when extended to more complex assets, this type of analysis may provide significant insight. This relative contribution analysis also indicates that the accuracy of the M_{Asset} output is mostly influenced by the accuracy of the calculation technique for the 2 mil shim as it is the dominant source. For example, poor accuracy of the flat panel feature would have negligible contribution to the M_{Asset} output.

These data have demonstrated the ability of the analysis techniques to scale-up laboratory panel data to represent a small asset contaminated with 0.2 µL drops and treated with soapy water spray. In this case, all calculation techniques met the criterion of $R_{Absolute} < 10$.

11.2.2 Multi-tool Asset with 1 µL Drops and Soapy Water Spray Treatment

The same sequence of analysis is conducted for each of the asset-drop volume-treatment combinations. The list of material-features and drop volumes used for the calculation is provided in Table 76, based on the detailed description in Table 71. The 1 µL drop contamination uses four drops to apply a total of 4 µL of agent to the asset.

Table 80. Description of drop count and drop volumes used to represent the contamination of the multi-tool asset with 1 µL drops

Treatment	Material-Feature	Number of Drops	Drop Vol (µL)	Asset
Soapy water spray	steel-flat	1	1	
	steel-20 mil	2	1	
	steel-2 mil	1	1	
	total	4	4	

The seven replicates of the tested asset (Asset RA, as measured in Section 10.3) and the MC output of the 10,000 iterations for each source term calculation technique are shown in Figure 77. These results show that the fit method accurately predicted the M_{Asset} within a factor of R=0.91, the indirect fit using V=0.2 µL data within a factor of R=0.06, the indirect fit using V=1 µL data within a factor of R=1.74. The accuracy of the indirect fit using V=0.2 µL did not meet the acceptance criterion, note that this is the case where the 0.2 µL drop data is extrapolated to the 1 µL condition. The other fit methods produced results within the acceptance criterion range.

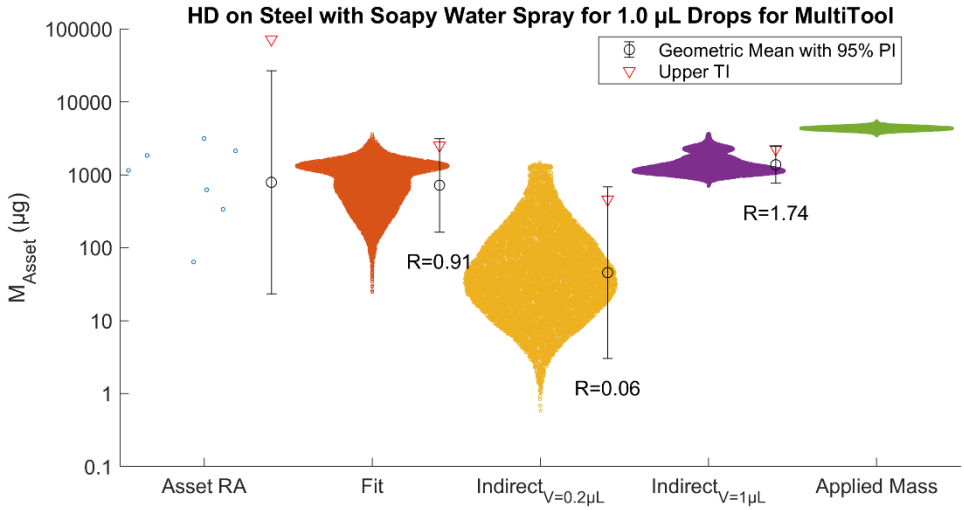


Figure 77. MC output visualization for the multi-tool asset with 1 µL HD drops treated with soapy water spray. The circle with error bars to the right of each swarm plot represent the geometric mean and 95% PI for the data.

The tabulated outputs of the results are provided in Table 81. The methodology output B_2 (Figure A-1) of asset level performance as quantified by LD from the applied mass is

provided. These data show that soapy water spray provided 0.74 ± 0.54 LD for the tested asset compared to the applied agent mass, and that the fit predicted 0.78 ± 0.01 LD.

Table 81. M_{Asset} output for the tested asset and calculation variations for the multi-tool asset with 1 µL HD drops treated with soapy water spray

Type	Geometric Mean (µg)	Upper Bound TI (µg)	Upper Bound 95% PI (µg)	μ_{Output} ($\log_{10}(M_{Asset}$ µg))	σ_{Output} ($\log_{10}(M_{Asset}$ µg))	LD ± CI (relative to applied mass)
Asset RA	787.81	71915.97	26607.87	2.90	0.58	0.74 ± 0.54
Fit	718.83	2527.87	3144.78	2.86	0.33	0.78 ± 0.01
$Indirect_{V=0.2µL}$	45.51	459.43	686.40	1.66	0.60	1.98 ± 0.01
$Indirect_{V=1µL}$	1372.82	2245.14	2445.35	3.14	0.13	0.50 ± 0.00

The accuracy ratio results (R) comparing each analysis technique to the tested asset are provided in Table 82. For these data the fit regression provided the most accurate estimate of M_{Asset} with an $R_{Absolute}$ of 1.10. For this asset condition, the accuracy of the indirect fit using V=0.2 µL did not meet the acceptance criterion; the $R_{Absolute}$ value indicates that this calculation technique predicted a value 17.3× less than the tested asset RA. The other fit methods produced results within the acceptance criterion range of $R_{Absolute} < 10$ (R is within 0.1–10).

For context on how well the PI and TI for the calculation techniques represent tested asset, ratio values for the quantity are also provided. If the upper bounds are compared to health-based requirements, then some measure of the accuracy should be considered. Here, the calculations are providing estimates much smaller than indicated from the test data, likely due to the differences in degrees of freedom in the calculation. No criteria were established for upper bound estimates.

Table 82. Accuracy ratio of the calculation variations for the multi-tool asset with 1 µL HD drops treated with soapy water spray.

Type	R: Geo Mean	$R_{Absolute}$: Geo Mean	R: Upper TI	R: Upper PI
Fit / Asset	**0.91**	1.10	0.04	0.12
$Indirect_{V=0.2µL}$ / Asset	**0.06**	17.31	0.01	0.03
$Indirect_{V=1µL}$ / Asset	**1.74**	1.74	0.03	0.09

Bold blue values are the accuracy ratio that should be within the validation criterion range of 0.1–10.
Red values indicate results are outside the acceptance criterion.

To build trust in the accuracy of using the geometric mean, a histogram of the results are provided in Figure 78, the red line shows a fitted normal distribution to the log-transformed results. Ideally, the red line should have similar shape and magnitude as the histogram. Deviations of the red line from the histogram indicate that the log-normal distribution may not be an accurate description of the data, and quartile (i.e., non-parametric) analysis may be a better description of the results.

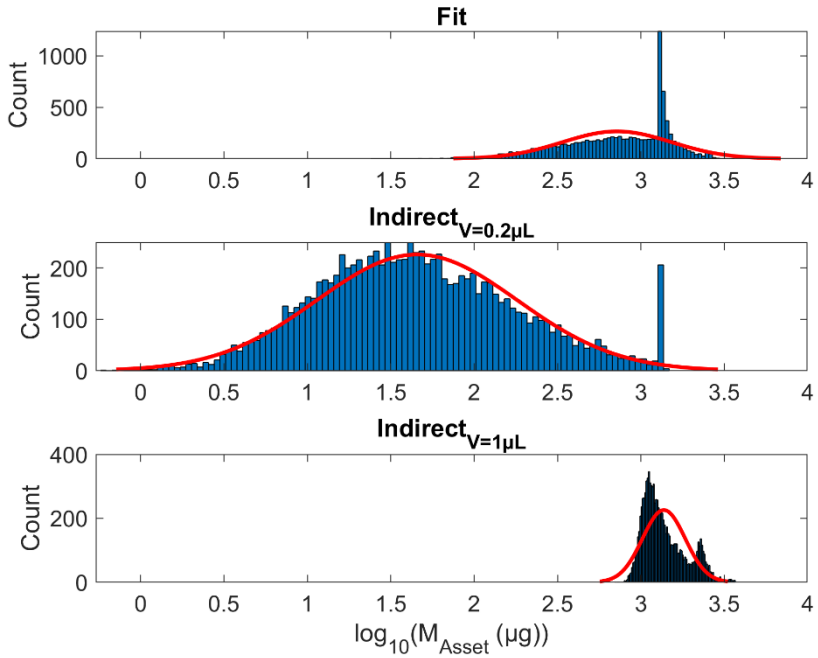


Figure 78. Histogram of the M_{Asset} output fit to a log-normal distribution (red line), for the multi-tool asset with 0.2 µL HD drops treated with soapy water spray.

The geometric mean calculation assumes that the data are log-normal, with some deviations from normality seen in Figure 75 (attributed to the non-detect or high value source terms). The MATLAB function *quantile* is used to identify the magnitude of M_{Asset} for specific quantiles such indicating that 95% of the data are less than an indicated value. The quantile output requires more samples than the seven replicates collected for the Asset RA data, thus only the MC output is presented in the quantile output (Table 79).

Table 83. Quantile analysis of the M_{Asset} output for the multi-tool asset with 0.2 µL HD drops treated with CHAD.

Quantile	Fit Analysis (µg)	Indirect$_{V=0.2µL}$ (µg)	Indirect$_{V=1µL}$ (µg)
25%	432.981 (0.5x)	16.927 (0.4x)	1098.451 (0.9x)
Median 50%	847.120 (1.0x)	41.687 (1.0x)	1267.776 (1.0x)
75%	1309.820 (1.5x)	112.970 (2.7x)	1634.370 (1.3x)
95%	1788.946 (2.1x)	562.443 (13.5x)	2405.021 (1.9x)

Values in parenthesis are the ratio of the value to the median.

The MC calculation tracks the source of agent mass, enabling the attribution of which material-features contributed to the total M_{Asset} output. Figure 76 provides the relative contribution for each feature type. For example, the steel 2 mil feature was typically responsible for 86.3% of the M_{Asset} mass, and the steel 20 mil feature was typically responsible for 13.5% of the M_{Asset} mass, and the steel-flat response provided an insignificant (0.04%) contribution to the M_{Asset} mass. A similar analysis can be done without normalization to the M_{Asset} mass to indicate, for example, that typically 622 µg of the 787 µg ($M_{Asset,\ fit\ geometric\ mean}$) retained by the asset was attributed to the steel-2mil feature.

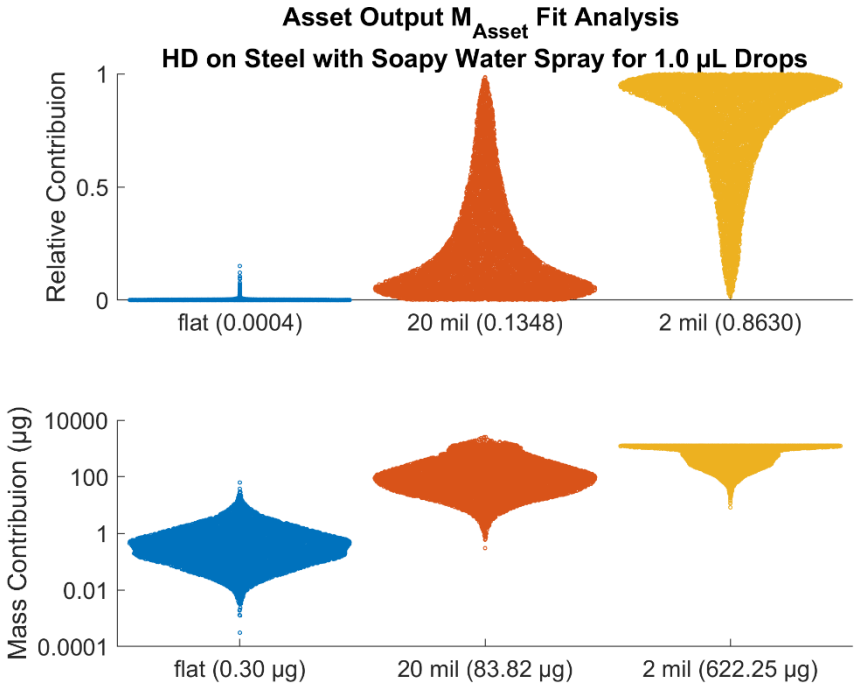


Figure 79. Relative contribution and mass contribution graphs for each feature for the multi-tool asset with 1 µL HD drops treated with soapy water spray, values in parenthesis are the median response for the material-feature indicated.

The contribution calculations enable material attribution, here showing that the 2 mil shim panel was by far the major source of exposure. This relative contribution analysis also indicates that the accuracy of the of the M_{Asset} output is mostly influenced by the accuracy of the calculation technique for the 2 mil shim as it is the dominant source. For example, poor accuracy of the flat panel feature would have negligible contribution to the M_{Asset} output.

These data have demonstrated the ability of the analysis techniques to scale-up laboratory panel data to represent a small asset contaminated with 1 µL drops and treated with soapy water spray. In this case, the extrapolation of the 0.2 µL data to represent 1 µL drops did not provide the desired scale-up accuracy. However, the other calculation techniques met the criterion of $R_{Absolute} < 10$.

11.3 Accuracy Summary for M_{Asset} Results

The calculation results presented in Section 11.2 and Appendix H are summarized here. The validation objective was to evaluate the ability of the scale-up calculation to use panel data to predict the responses for tested assets. The ratio, *R*, of the predicted to measured values were to be within the range of 0.1–10 (i.e., within a factor of 10 high or low).

The use of 0.2 and 1 µL agent drop volume contamination of the asset results in the application of a total of (5 × 2 µL) 1 µL or (4 × 1 µL) 4 µL of agent, a four-fold difference in contamination density on the asset. These data also show how per drop normalization enables the estimation of asset retention for different asset-level contamination densities.

As a first comparison, the geometric mean and 95% PI for the asset RA and calculation techniques are provided in Figure 80. In most cases, the PI for the tested asset spans

more than an order of magnitude. Ideally, if the laboratory panel data produces a similar mean and standard deviation per drop as observed on the test asset, then the M_{Asset} mean value and PI should be similar. This is generally observed for the soapy water spray treatment. In the Asset analysis for the CHAD treatment, it was observed that the relative contributions of the 2 mil shim dominated the response providing a median 87% of the total asset mass (Figure H-6). The laboratory data for steel with the 2 mil shim exhibited an exceptionally large variability (σ = 1.29 $\log_{10}(\mu g)$ for 0.2 µL drops and σ = 2.14 $\log_{10}(\mu g)$ for 1 µL drops, from Table 53). As a result, the predicted PI span many orders of magnitude for the CHAD treatment. This was a result of the variability observed for the CHAD panel data. Note that the CHAD treatment process used a reduced treatment time to ensure measurable values, the variability for longer treatment durations may be different. In contrast, the soapy water spray asset RA tended to have smaller PI, and the predicted mass for this treatment process was also much smaller. For soapy water spray, the 2 mil shim results tended to represent a median contribution of 86% to the asset response (e.g., Figure 79), with much smaller variability compared to CHAD treatment (σ = 0.82 $\log_{10}(\mu g)$ for 0.2 µL drops and σ = 0.04 $\log_{10}(\mu g)$ for 1 µL drops, from Table 40). The soapy water spray is more reproducible (i.e., smaller variability), but shows much less efficacy (reduction in M_{Asset}).

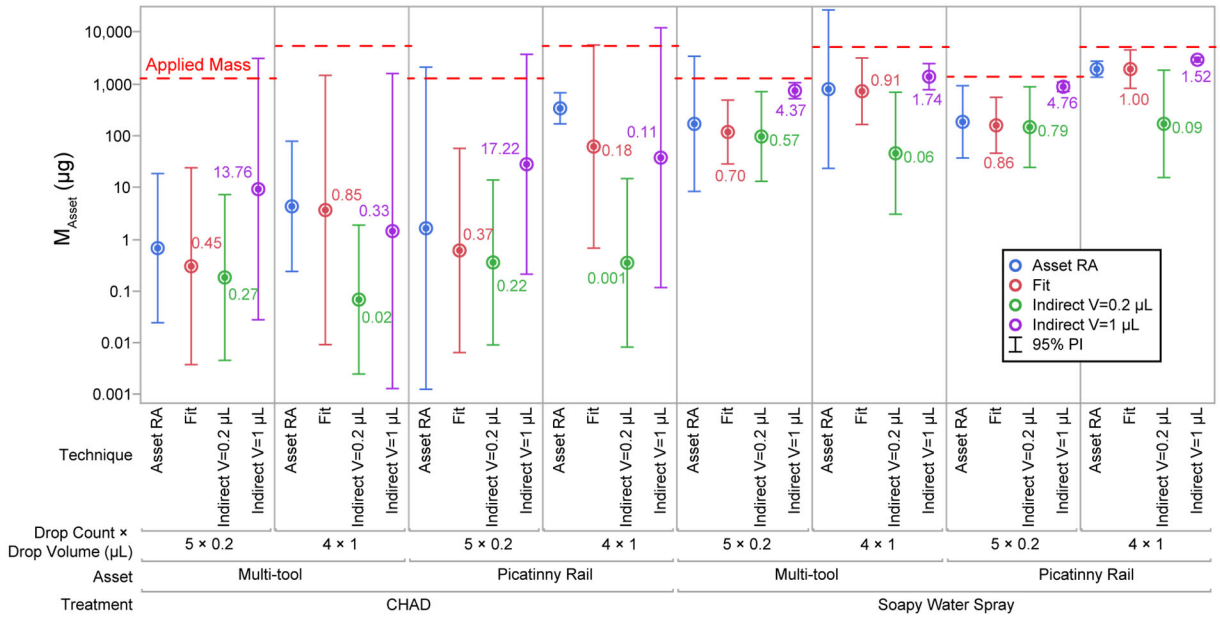


Figure 80. Summary output of M_{Asset} calculations for CHAD and soapy water spray treatments, circle shows the geometric mean, error bars correspond to the 95% PI, the numerical value is *R* (predicted / measured).

The R values results for each analysis technique are presented in Figure 81. For the fit technique, all M_{Asset} R values are within the validation criterion of 0.1–10. The text values in the graph indicate the $R_{Absolute}$ value. For example, for the multi-tool contaminated with 5 × 0.2 µL drops and the CHAD treatment process, the fit technique predicted a M_{Asset} value 2.24× less than the measured value. In *most* cases, the *R* values for indirect techniques are within the validation criterion of 0.1–10. Generally, for the indirect technique the accuracy of the M_{Asset} calculation is good when the drop volume used to generate the source term equals the drop

volume used on the asset (i.e., direct fitting or interpolated), and less accurate when the asset drop volume was extrapolated. For example, for the indirect V=0.2 µL fit, 0.2 µL panel data is used, the square data points for that fit type are extrapolated to 1 µL drops and tend to be outside of the validation criterion (i.e., more than 10× less than the measured response). When the indirect V=1 µL technique is used, the 0.2 µL results are extrapolated, and tend to produce values greater than the measured asset, and exceeding the validation criterion by more than 10× greater than the measured response.

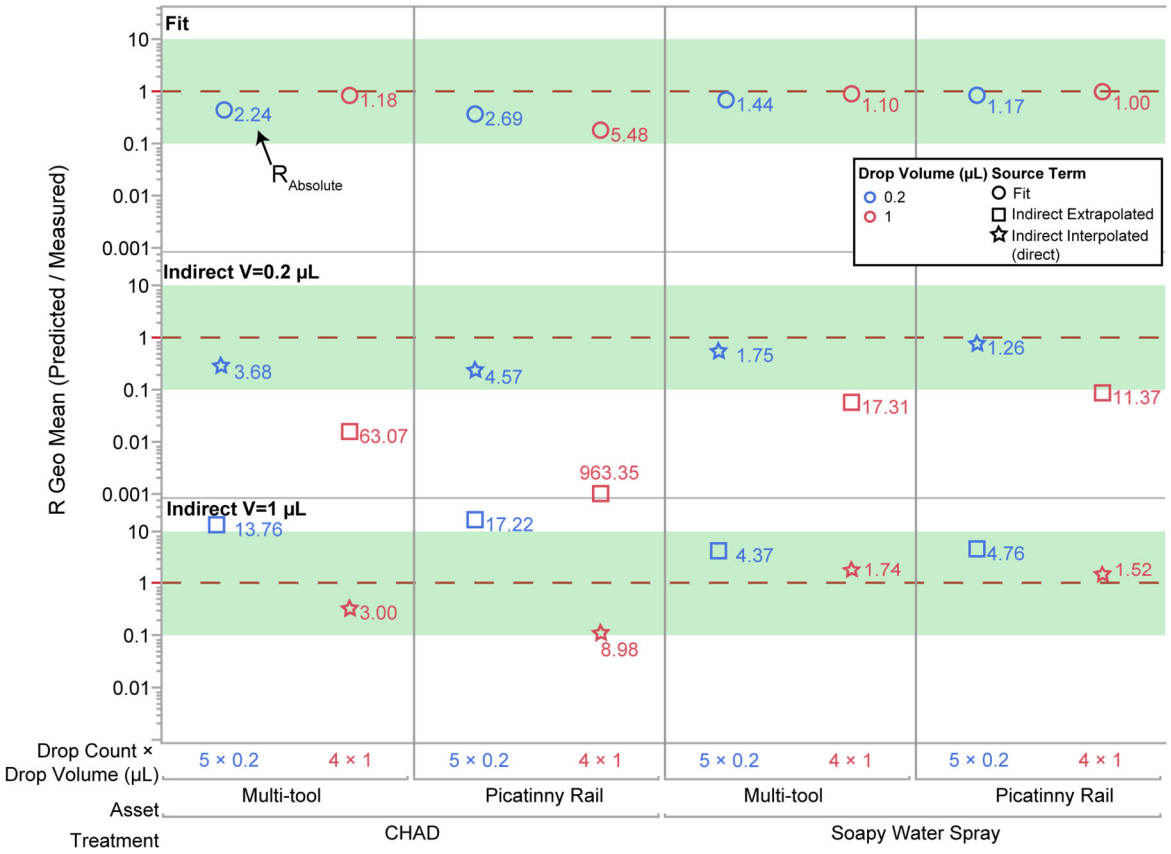


Figure 81. Trends for the ratio R for geometric mean (predicted / measured) with each source term calculation technique. The text values indicate the $R_{Absolute}$ magnitude. Green shading indicates the validation criterion of 0.1–10.

Summary statistics of the R values are provided in Table 84. The ratio R captures if the prediction over or underestimated the measured value. However, if the source term calculation had instances of over or underestimating the measured value the summary statistics would tend towards a value of 1. The $R_{Absolute}$ is calculated as R when R is greater than 1, and 1/R otherwise. The absolute ratio is always greater than 1 and the summary statistics describe the overall accuracy of the source term calculation; lower values (i.e., 1.0) of $R_{Absolute}$ indicate better accuracy. The $R_{Absolute}$ shows that the fit analysis provides the best accuracy ($R_{Absolute}$ closer to 1.0). For the fit analysis, the calculated geometric mean for M_{Asset} is within a $R_{Absolute}$ factor of 1.7× (lower than measured because the R value is less than 1). For the tested conditions, the maximum $R_{Absolute}$ was 5.48. Indirect technique exhibited less accuracy with geometric mean

values for $R_{Absolute}$ on the order of 12× (underpredicting for 0.2 µL drops) and 4.94× overpredicting for 1 µL drops.

Table 84. Summary statistics for R values for M_{Asset} across the analysis techniques

Technique	R M_{Asset}			$R_{Absolute}$ M_{Asset}		
	Geo Mean	Min	Max	Geo Mean	Min	Max
Fit	0.59	0.18	1.00	1.71	1.00	5.48
Indirect V=0.2 µL	0.08	0.001	0.79	12.05	1.26	963.35
Indirect V=1 µL	2.17	0.11	17.22	4.94	1.52	17.22

Red text indicates values not meeting the validation criteria

The prediction accuracy for upper bound estimates is provided in Table 85. There was no criterion for the accuracy of upper bound intervals. Additionally, the upper bound limits for the tested asset RA may be inflated based on the smaller sample size (n=7). Overall, the fit technique provided results within a geometric mean of 10, with some cases producing values as high as 547 factor difference for the TI. The indirect technique with V=1 µL drops produced lower values than the V=0.2 µL drop condition.

Table 85. Summary statistics for $R_{Absolute}$ values for upper bound intervals for each calculation technique.

Type	$R_{Absolute}$ Upper TI			$R_{Absolute}$ Upper PI		
	Geo Mean	Min	Max	Geo Mean	Min	Max
Fit	9.4	1.3	547.7	5.8	1.3	37.0
Indirect V=0.2 µL	37.5	2.1	1958.0	11.0	1.0	150.4
Indirect V=1 µL	6.0	1.1	32.0	6.9	1.2	166.3

The overall trends for accuracy indicate that the fit technique provides significantly better accuracy for predicting the geometric mean of an asset compared to the use of the indirect technique. The indirect technique assumes that efficacy is constant for all drop volumes (i.e., the slope, *m* is 1), and the decreased accuracy for extrapolated conditions indicates that drop volume effects can be significant. The improved accuracy for indirect technique using the 1 µL drops, versus the 0.2 µL drops, may be a result of the drop volumes used. First the 1 µL drop volume is a midpoint for the range of drops selected, where similar ranges of extrapolation to larger and smaller drops may minimize error. A more in-depth analysis of the effect of slope magnitude on indirect technique accuracy is presented in Appendix G.

The indirect technique may provide less accuracy for asset-level predictions, and is close to the desired R of 0.1–10. If the test objective is to enable scale-up to assets, variable drop volume testing will provide better accuracy. When using previousdata or testing resources only allow for fixed agent drop volume testing, the indirect technique may be the only option. In this case, the analysis could recognize the decrease in accuracy and assume an 'uncertainty factor' of 10–100 (i.e., assume the result is a factor of 10–100 lower than actual) to provide safe-

sided estimates for outputs. As a program matures, if necessary, the variable agent drop volume results could be acquired to provide improved accuracy.

The assets tested here are constructed of impermeable materials, and five or less drops of agent were applied. The objective of this work was to specifically control the contamination of the asset to perform this accuracy calculation. These configurations were selected to enable testing at the laboratory scale for this first-time analysis. Further, the contribution analysis indicated that the steel 2 mil shim results were responsible for more than 80% of the total asset mass. As a result, the accuracy of the steel 2 mil shim condition is the primary factor that influences the accuracy of the scale-up results. Future efforts would benefit from including other materials, especially material conditions with more reproducible results to confirm the accuracy of the scale-up calculations.

These data and results demonstrate validation goal 3. The fit technique was shown, on average, to predict the geometric mean of the asset within a factor of 1.7×, with a maximum error of underpredicting the asset mass by a factor of 5.5×. An indirect technique that assumes that efficacy is independent of the agent drop volume (i.e., *m* of Eq. 5 is 1.0) and relies on the extrapolation to different agent drop volumes, was found to be less accurate (e.g., within a factor of 5× of the tested asset RA using 1 µL drops, or 12× for 0.2 µL drops). It was observed that for the assets tested, the 2 mil shim results were the dominant contribution to the total asset mass (e.g., Figure 76). As a result, these accuracy assessments are most sensitive to the ability for each calculation technique to represent the 2 mil shim panel data.

11.4 Accuracy of M_{Asset} for Other Conditions

The analysis characterizing the accuracy of M_{Asset} conducted here was focused on HD and for impermeable materials. Previous studies using similar treatment processes have demonstrated that the magnitude and variability of measurements are a function of agent-material combinations,[6,7] and that the methodology enables characterization of these factors regardless of the agent or material used. A fundamental assumption of the test methodology is that a representative agent distribution was generated, and that the treatment process would be conducted similarly for the laboratory panel and the full-scale asset. If this is the case, the RA response measured by the test methodology captures the salient agent-material-feature interactions (e.g., spreading, sorbing, evaporation) and how the treatment process accesses and alters the resulting agent distribution through removal or reaction.

The testing conducted here used the highly controlled contamination of small assets and demonstrated the accuracy of scaling laboratory data. The exact location and volume for each drop was controlled and the source term for each agent-material-feature was directly tested. In practice, contamination will be random, asset geometries will be more complex, more materials than are practical to test may be present. The measurement and aggregation of sources is straightforward, the random contamination of full-scale assets and assignment of laboratory generated source terms to specific asset features is more complex and presents the greatest potential influence on the accuracy of M_{Asset} values. For example, in Appendix C, an approach is developed to approximate the contamination probability that is used to determine what features may be contaminated for a given contamination vignette. Some features present substantially

different source term magnitudes; the exact drop distribution (what features are contaminated) could significantly influence the M_{Asset} value.

The Monte Carlo methods demonstrated in this report are ideally suited to perform repeated random contamination of assets and characterize the resulting distribution of potential responses. Initial results (not shown) indicate that assets receiving a few drops may present responses spanning orders of magnitude upon repeated random contamination. This is thought to be representative of the range of response that could be observed, *based on the specific features contaminated for each iteration.* Whereas assets receiving thousands of drops produce variation within a factor of two because the specific features contaminated are more uniformly contaminated. The accuracy of the M_{Asset} value relies on the description of how the asset is contaminated. The distribution of results could indicate if all assets were contaminated below requirements or if 90% of assets would be below requirements (10% of decontaminated assets did not meet the requirement). Further, many contamination vignettes could be explored to understand the situations that generate the greatest exposure risks. From this, the ability to define a contamination probability for various features that aligns with the contamination vignette of interest is paramount in considering the accuracy of the calculation output. Further, this illustrates how the health-based outputs are highly sensitive to the details of contamination.

11.5 Considerations for Future Test Designs

These results illustrate that there are multiple approaches to using the laboratory data. The end use of the results may drive the needs of data collection. If the objective is focused on producing asset-level health-based outputs for variable operational contamination conditions, then variable agent drop volume test data analyzed with the fit regression will provide the best accuracy. If the objective is screening multiple technologies or experimental factors, more efficient constant agent drop volume testing and indirect fit could be used to provide an initial approximation for scale-up results. Variable drop volume testing could utilize multiple sampling schemes and tested drop volumes shown in Table 86.

Table 86. Contamination Test Designs

Test Design	Contamination	Replication	Scale-up Accuracy	Limitations
Contamination Density	Apply a specific starting challenge (g agent / m^2 material)	a set of replicates is typically acquired for each starting challenge of interest.	Contamination density scaling approaches using contaminated area could be used.	Data can be analyzed using extrapolation approach
Extrapolation	per drop normalization, one or more drops of a specific volume is applied to the material	Similar to legacy, however the number of drops does not have to be adjusted to achieve a specific starting challenge	less accurate, especially as the operational drop volumes diverge from the tested agent drop volume	Assumes no drop volume effects on efficacy
Fit	per drop normalization, one or more drops of a specific volume is applied to the material, *a range of drop volumes are individually tested*	Replication is distributed across agent drop volumes, more replication provides more degrees of freedom in the analysis and more accurate estimates of mean and standard deviation outputs	more accurate	Requires more test samples

Testing using fixed contamination density has typically used five replicates for constant agent drop volume testing. For example, a full factorial investigation of treatment process–agent–material combinations is required with each condition acquiring five identical test replicates. In the case of variable agent drop volume, each drop volume contributes to the degrees of freedom in the output. The objectives of the methodology assessment produced a sampling plan with 16 replicates per treatment-agent-material-feature combination, however, other sampling plans can be used for future efforts. Table 87 provides examples of alternate sampling plans that could support the indirect or multivolume fit techniques. It should be recognized that variable agent drop volume testing does require slightly more laboratory planning and organization to manage (i.e., additional DCS replicates and test execution to ensure the correct agent drop volume is applied to the specified sample).

Table 87. Sampling plan options for future testing for each agent-material-feature-treatment combination

Agent Drop Volume (µL)	Replication					
	Methodology Assessment	Fixed Contamination Density	Extrapolation from V = 1 µL	Fit, replication low	Fit, replication medium	Fit, replication high
0.2	5	-	-	2	3	2
0.6	2	-	-	-	-	2
1.0	5	5	5	2	3	2
2.0	2	-	-	-	-	2
4.0	2	-	-	2	3	2
Total samples	16	5	5	6	9	10

A universal specification for how much data is required to provide sufficient accuracy and statistical confidence in the results is non-trivial to define. The data presented in this methodology assessment have shown that the variability of the method output is specific to the treatment process and agent-material-feature under test. Conditions with more variability would require more replication for the same confidence. Across many test programs executed over the last 10 years at DEVCOM CBC, a typical data variability for flat panel results (RA standard deviation) seems to be on the order of 0.2–0.3 $\log_{10}(\mu g)$. It is recommended that a statistical analysis is conducted to optimize test guidance on settings and replication to use for testing.

12. MAXIMUM POTENTIAL DOSE DEMONSTRATION

A new output capability in the methodology involves the use of the M_{Asset} output to approximate a maximum potential dose. In this case the total RA associated with an asset is assumed to produce an exposure and serve as an upper bound estimate. If this MPD output is below the relevant health effect requirement, then any detailed exposure assessment associated with this asset would be equal to or less than the MPD. This type of analysis can be performed on directly tested assets, or on the scale-up calculation output of M_{Asset} results.

12.1 Percutaneous Exposure

Percutaneous (i.e., through the skin) contact is one route of entry to the human body that is relevant for chemical warfare agent exposures. In the context of MPD it is assumed that all of the asset RA mass contributes to the exposure dose. The multi-tool will be used to demonstrate the MPD calculations. For this situation, it will be assumed that the multi-tool is held in the palm of a hand.

The data acquired for this program used the chemical warfare agent HD, which is a local effect agent. The exposure dose that generates an acute response is a localized skin surface concentration, often specified as μg cm^{-2} (agent mass per exposed skin area). For this demonstration a *conceptual* toxicity level of 10 μg cm^{-2} will be used as a pass/fail criterion. Notionally, if the exposure dosage exceeds this level, some percentage of the population would exhibit a negative health effect, such as erythema, that would inhibit their ability to complete their mission. The methodology for the evaluation is independent of the specific toxicity value used.

This MPD exposure will consider that the total agent mass retained by the multi-tool becomes distributed on one hand. The skin surface area for various anatomical body regions is available from other resources.[26,27] For the average American male, the palmar surface area of the hand is between 76–107 cm^2, the smaller value of 76 cm^2 will be used here to produce an upper bound skin surface concentration. The localized skin concentration exposure dosage is calculated as

$$D_{local} = \frac{M_{Asset}}{A_{skin}} \qquad \text{Eq. 24}$$

Where

D_{local} is the localized skin concentration of agent (μg cm^{-2})

M_{Asset} is the mass of agent associated with asset (μg)

A_{skin} is the area of skin interacting with the asset (cm^2)

It is necessary to assume that the agent becomes distributed on skin area used. The discrete drops of agent on the asset may produce highly localized skin surface concentrations, but it is not practical to map all potential drops to this level of detail, especially for other more complex assets. Further, extended handling of the object is likely to ‘smear’ and distribute the agent over time.

The predicted M_{Asset} output from Section 11.2 for the fit technique (e.g., results from Table 73) are used to calculate D_{local}, with the results provided in Table 88. Here the upper TI is used (defined in Section 6.6), recalling that this level provides 95% confidence that 95% of decontaminated assets should be below this value. These data, when compared to the conceptual toxicity level of 10 µg cm^{-2} would indicate that the MPD associated with the use of CHAD (with a 2 h treatment duration) on a multi-tool contaminated as indicated, produced results well below the health effect value. For soapy water spray treatment, the 5 × 0.2 µL contamination is also below health effect levels. However, the 4 × 1 µL agent drop volume condition is very close to the health effect level, and the upper TI is three times the health effect limit.

Table 88. Percutaneous skin concentration output for HD contamination and treatment of Multi-tool assets.

Asset	Contamination	Treatment	M_{Asset} (µg)		D_{local} (µg cm^{-2})	
			Geo Mean	Upper 95% TI	Geo Mean	Upper 95% TI
Multi-tool	5 × 0.2 µL	CHAD	0.3	12.5	0.004	0.16
		Soapy water spray	117.2	393.3	1.5	5.2
	4 × 1 µL	CHAD	3.6	598.4	0.048	7.9
		Soapy water spray	718.8	2527.9	9.5	33.3

Contact area assumed as 76 cm^2 as a lower bound for the palmar hand area.
Values in red text are above the conceptual toxicity level of 10 µg cm^{-2}.

Nerve agents such as GD or VX, are systemic agents. The relevant exposure metric is total dose in mass units, rather than a localized skin concentration. Nerve agent data was not collected here, however, for the sake of demonstration, we reuse the HD data as though it was VX nerve agent. For this demonstration, we will use a *conceptual* health effect exposure dose of 25 µg to represent the toxicity value for the desired health effect level. Recall, the methodology for the comparison of results to the health effect level is independent of the specific toxicity value used. Because nerve agents are systemic, and the MPD assumption is that all asset mass produces an exposure, the M_{Asset} value is compared directly to the toxicity value. The results in Table 89 show that all cases of soapy water spray are above the toxicity value; the technology did not meet the requirement specifications.

Table 89. Percutaneous skin concentration output for *emulated* VX results.

Asset	Contamination	Treatment	M_{Asset} (µg)	
			Geo Mean	Upper 95% TI
Multi-tool	5 × 0.2 µL	CHAD	0.3	12.5
		Soapy water spray	117.2	393.3
	4 × 1 µL	CHAD	3.6	598.4
		Soapy water spray	718.8	2527.9

HD data is reused as *emulated* VX data for this conceptual demonstration.
Values in red text are above the conceptual toxicity level of 25 µg.

For CHAD the 4 × 1 µL contamination condition resulted in more than 5% of the multi-tools producing results significantly above toxicity value. The 5 × 0.2 µL contamination case was successfully decontaminated below health effect levels. However, a value of 12.5 is rather close to the requirement of 25 µg. If MC results were used to predict this value and the evaluator wanted to consider an uncertainty factor of 10,[§§§] the evaluation could be adjusted to consider if the Upper 95% PI was 10 times less than the requirement. This type of adjustment would provide a safety margin should there be biases in the calculation or specification of the vignette.

These data have illustrated how the scale-up calculation can use RA derived M_{Asset} value to evaluate a MPD percutaneous exposure and compare the results to relevant toxicity values.

12.2 Vapor Exposure Dose

The M_{Asset} output can be used to estimate an upper bound steady-state vapor source term. The source term is then used to calculate an environmental concentration that can be used to determine a vapor exposure dosage for comparison to health-based requirements.

VE MPD assumes that all RA associated with an asset is emitted over the exposure vignette time duration at a *constant emission rate*. Experimental results show that vapor emission tends to rapidly decay over time, however, the rate of decay is not known without testing. The constant (i.e., steady state) emission rate based on B_0 should provide a safe-sided emission rate; typically less than 100% of the RA is emitted over 24 h time periods. For emission rates that have non-linear decay, the majority of emission tends to occur shortly after treatment, however assets may not be reissued to personnel for some time before the exposure may actually occur (Figure 82). Toxicological response is most accurately represented using the ten Berge toxic load model[28]

$$k = C^{TLE}t \qquad \text{Eq. 25}$$

§§§ Uncertainty factors are commonly used in exposure assessments. The value of 10 is provided here as a conceptual value, other values could be used. Uncertainty factors have not been previously used in hazard mitigation technology evaluations. If used, an uncertainty factor should selected with a documented rationale.

Where

k	is the effective dosage (mg min m^{-3})
C	is the steady-state environmental concentration over the exposure duration (mg m^{-3})
TLE	is the toxic load exponent
t	is the exposure duration (min)

The TLE is an agent specific parameter that should be provided with the toxicity values, note that the unit convention for k does not carry the TLE exponents. The ten Berge equation is cited as the DoD-accepted method to evaluate vapor exposure in FM 3-11.9, and is used by the Army Public Health Center (APHC) to perform the time extrapolations to determine exposure concentration (EC) levels for various exposure durations.[28-30] For some chemicals the dose rate (i.e., vapor concentration) influences the dosage (i.e., quantity) of chemical that induces a health-based response. The TLE accounts for the effects of dose rate. For agents with a TLE greater than 1.0 (i.e., most nerve agents), the steady state assumption may underestimate health effects if personnel interact with the assets during the high emission rate period (typically short durations during the first few hours after treatment). It is assumed that personnel would be conducting personal decontamination during these periods. The steady-state emission rate will overestimate the actual emission rate at longer time durations. Metrics such as the total potential vapor emitted mass (VEM) will be greater than or equal to the experimentally determined values, similar trends should be observed for exposure doses since the MPD assumes all agent results in exposure.

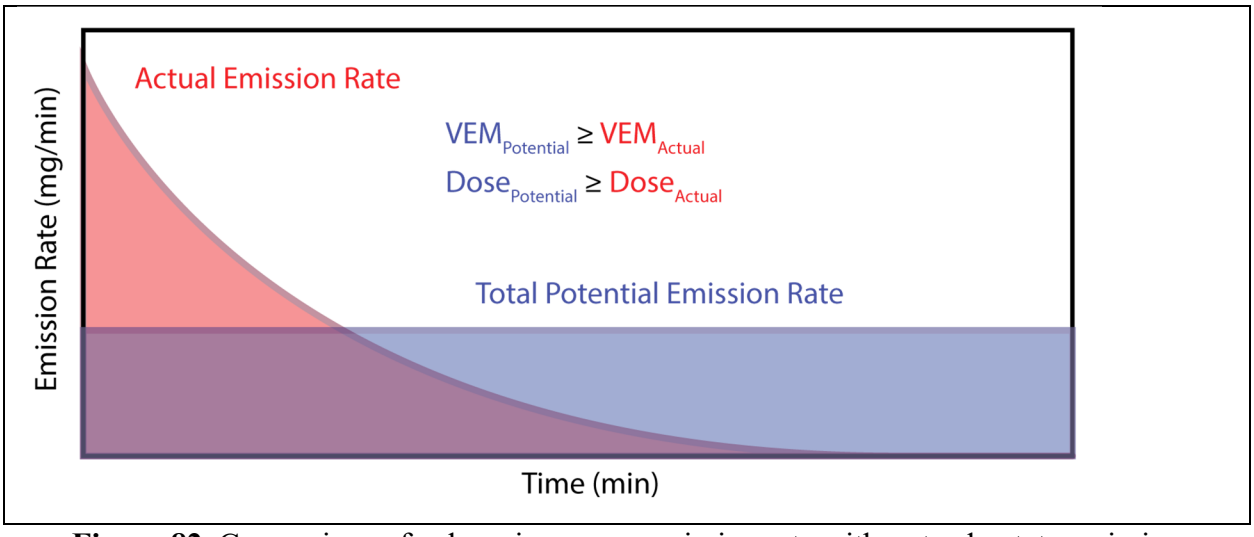


Figure 82. Comparison of a decaying vapor emission rate with a steady-state emission approximation.

As the example vignette, two soldiers will each have the multi-tool in a vehicle cabin for 8 hours. The vehicle interior volume is estimated to be 3 m^3, and using a ventilation estimate of 0.3 m^3 min^{-1} from a civilian passenger sedan with windows up, ventilation off, driving at 60 miles per hour.[31] Note that equivalent ventilation information (i.e., the air change

rate) is required for the any vignette of interest. For this exposure assessment, it is assumed that the two warfighters are in the vehicle for the full 8 hours. The empty volume of the vehicle is used here, the volume of air that is displaced by the two personnel is not accounted for. Further, it is assumed that the agent vapor released from the asset is promptly and completely mixed through the interior volume (i.e., the environment is well mixed.).

The mass balance equation specifies the rate of change of the environmental vapor concentration, *C(t)*, as a function of the object emission rate, *E(t)*, the volume of enclosed free air, *V*, and the fresh air flow rate, *Q*.[32]

$$\frac{dC}{dt} = \frac{E(t)}{V} - C(t)\frac{Q}{V} \qquad \text{Eq. 26}$$

Where

E(t)	Is the object emission rate (mg min^{-1})
V	enclosed free air volume (m^3)
C(t)	vapor concentration (mg m^{-3})
Q	fresh air flow (ventilation) rate through the enclosed volume (m^3 min^{-1})

If the source term, *E(t)*, is steady-state (as assumed by the MPD calculation), then *C(t)* will be asymptotically approach 99% of the steady-state concentration within 6.9 air changes.[33] Assuming instantaneous steady-state concentration, which produces a small overestimate of the resulting vapor dosage, these steady-state values allow the differential equation to be reduced to an algebraic expression for *C*

$$C = \frac{E}{Q} \qquad \text{Eq. 27}$$

Where

E	Is the total steady-state asset emission rate (mg min^{-1})
C	Is the stead-state environmental vapor concentration (mg m^{-3})

Where the environmental vapor concentration, *C*, can be used to calculate an exposure dosage using Eq. 25. For HD, the TLE is 1.0. For the exposure assessment here, the total MPD steady-state vapor emission rate of the multi-tools (from the scenario) is

$$E = \frac{a\, M_{Asset}}{t} \qquad \text{Eq. 28}$$

Where

a	is the number of assets in the environment (2 multi-tools)

t is the exposure duration in minutes (8 h is 480 min)

The relevant calculations and outputs are provided in Table 90. For this demonstration, a *conceptual* vapor toxicity value for HD of 11.5 mg min m^{-3} (equivalent to the 8 h vapor concentration of 0.024 mg/m^3) is used as the requirement value not to exceed. Similar calculations could be performed for outdoor environments using the appropriate transport and dispersion modeling tools.

Table 90. MPD vapor exposure assessment for two multi-tool assets in an enclosed vehicle for 8 h.

Contamination	Treatment	M_{Asset} (µg)		E (mg min^{-1})		C (mg m^3)		k (mg min m^{-3})	
		Geo Mean	Upper 95% TI	Geo Mean	Upper 95% TI	Geo Mean	Upper 95% TI	Geo Mean	Upper 95% TI
5 × 0.2 µL	CHAD	0.3	12.5	6.25E-07	2.60E-05	2.08E-06	8.67E-05	0.0010	0.042
	Soapy water spray	117.2	393.3	2.44E-04	8.19E-04	8.14E-04	2.73E-03	0.39	1.3
4 × 1 µL	CHAD	3.6	598.4	7.60E-06	1.25E-03	2.53E-05	4.16E-03	0.012	2.0
	Soapy water spray	718.8	2527.9	1.50E-03	5.27E-03	4.99E-03	1.76E-02	2.4	8.4

Values in red text are above the conceptual toxicity level of 11.5 mg min m^{-3}.

Based on the results presented in Table 90. The MPD vapor exposure indicated that all responses were less than the specified toxicity value, *for this vignette*. However, in the case of 4 × 1 µL contamination treated with soapy water spray, the upper TI is very close to the health-effect level. While the 4 × 1 µL contamination condition produced an upper TI of 8.4 mg min m^{-3}, which is less than the specified toxicity value, it is within a factor of 2 which may not provide an appropriate margin of safety. Similar to the percutaneous MPD example, if MC results were used to predict this value and the evaluator wanted to consider an uncertainty factor of 10 (for example), the evaluation could be adjusted to consider if the Upper 95% TI was 10 times less than the requirement.

12.3 Health Effects Outputs Are Systems Level Interactions

The reason decontamination is performed is to enable operations in a reduced protective posture without generating negative health effects. For this reason, many hazard mitigation technology key performance parameters are specified using health-based requirements. However, *the hazard mitigation technology being evaluated is not the only factor that influences if health effects would occur*. Health effects are generated by personnel interacting with post-decontaminated assets. The exposure is a result of multiple material, technology, and operational factors. These factors include material effects, such as how much agent a given material retains or how well the decontaminant removes agent from the material and how this relates to the resulting vapor or contact transfer source terms. Other influential factors include the operational inputs, such as how assets are contaminated, how long the

decontamination process lasts, when the assets are reissued, when MOPP reduction occurs, and how personnel use the decontaminated assets.

The hazard-mitigation technology can only reduce source terms based on when and how it is operationally implemented. It has often been assumed that the decontamination technology is the principal factor for evaluation of post-decontamination hazards, however, the use of these tools and the exposure-assessment process help to show that *all these factors are influential toward mitigating health effects*. The resultant health effects (or mitigation of health effects) that are associated with implementing a hazard-mitigation technology can only be evaluated by *combining the material effects with operational inputs using the exposure-assessment process*.

An implication of the systems aspect of exposure assessment outputs is that the *health effect results are only as accurate as the combination of the laboratory data AND accuracy of the operational context provided* (e.g., how many agent drops of what volume on what agent-material features, what is the actual contamination duration, etc.). The range of potential operational considerations is large, which influences the range of health effects that could be observed for a given situation. The scale-up calculations presented here enable a characterization of these operational variations. Further, the use of RA data and the MPD calculations enable the exploration of many operational situations using the least laboratory resources and calculation burden. Regarding the methodology assessment, this provides context towards the acceptability of the asset-level accuracy ratio being within the range of 0.1–10. The ability to describe all operational context within a factor of 10 (high or low) is unlikely, and thus the asset-level output is likely not the resolution limiting contribution to the desired health-based outputs.

The range of operational variability does not make this an intractable problem, rather the analysis approach should account for this variability. The implementation of Monte Carlo calculations as part of the scale-up calculation enables the exploration of random contamination and operational factors to indicate the range of potential asset-level operational responses.

12.4 Summary

The MPD calculation provides a simple upper bound estimate for vapor and contact transfer exposure assessments using RA data. The MPD calculation assumes that all asset agent contributes to the exposure, which may be a significant overestimate. The MPD is provided as an initial tool to evaluate how a technology may perform in operational context before the more expensive vapor and contact testing and analysis is performed. Additionally, the calculations associated with MPD are significantly easier and require far fewer detailed inputs than full exposure assessments. If a given vignette passes the MPD evaluation, then no exposure assessment (focusing on similar assets and conditions in a similar contamination vignette) can exceed this upper bound.

These data have demonstrated how laboratory panel data can be used to approximate the response of an asset with per drop normalization and scale-up calculations. The results can be used in an exposure assessment to compare to health-based requirements and evaluate the operational impact.

13. CONCLUSIONS

A methodology assessment was performed to evaluate the reproducibility and IPSD of the retained agent (RA) method on a range of materials in different orientations and configured as shim panels for the purpose of hazard mitigation technology characterization. This report covered the full process from laboratory measurement of the data elements through data normalization, scale-up, and a worst-case exposure assessment estimate that can be compared to health-based vapor and contact requirements to assess hazard mitigation technologies. The accuracy of the scale-up calculations was evaluated by testing two assets and comparing the observed results to the calculated scale-up results using flat and shim panel RA responses. Overall, the methods were found to perform within the established acceptance criteria.

13.1 New Capabilities

This increment to chemical hazard mitigation technology test methodology presents revolutionary changes to the material geometries tested, data analysis approaches, and scale-up calculations. One of the significant elements of this methodology is the use of a shim panel that generates entrained agent distributions. This feature is used to generically represent any feature on assets that could generate entrained agent. It was observed that the efficacy of decontamination technologies could be decreased by as much as 1900× for entrained agent compared to flat panels. Accounting for this change in efficacy is paramount for estimating the efficacy that would be observed on full scale assets in an operational context.

It has been demonstrated that starting challenge alone insufficiently specifies a contamination condition for an accurate and conclusive measurement of performance for hazard mitigation technologies.[11] Efficacy results can strongly depend on the drop volumes used to generate a staring challenge on materials. This occurs principally because the interaction area between liquid droplets and surfaces, which is primarily responsible for RA, varies with droplet volume and is sensitive to the liquid-material surface interactions. This implies that if the laboratory tested agent drop volume does not match the operational drop volumes, the scale-up calculations may not be accurate. A new data analysis approach that accounts for variable liquid drop volume and count was developed and implemented. Normalization of the RA mass per drop as a function of the drop volume using an empirical least-squares regression reveals the prevailing trends in the assessment of decontamination efficacy that are otherwise not resolvable. This normalization approach captures the effect of drop volume and variable contamination density, and is the foundation for the scale-up of laboratory test conditions to operational context. One impact of this approach is that *data analyzed on a per drop basis can be used to calculate the asset level response for any contamination density*. This also allows for panels used in testing to be miniaturized, which can significantly increase test throughput.

The new scale-up calculation accounts for how each agent drop contributes to the total RA mass of the asset; the whole is equal to the sum of the RA per drop. The laboratory generated RA per drop provides a *source term* that is aggregated to represent an asset. This requires that laboratory testing produces the RA source terms on a per drop basis for each material-feature combination found on an asset with which agent interacts, specifically, the mean and standard deviation of RA on a per drop basis. This data use provides a shift in perspective in the objectives of laboratory RA data from strictly characterizing *efficacy* as defined by log reduction to efficacy in terms of mitigation of health effects at the asset level.

13.2 Verification

The RA data is the primary data element produced by the test methodology. The methodology assessment considered verification and validation aspects of the RA measurement. The verification analysis included extraction efficiency (EE) and positive control samples to isolate and demonstrate the ability of the methodology to reproducibly extract and contaminate items. These demonstrations established that the materials are reproducibly contaminated and analyzed to enable the characterization of the affect (i.e., the RA mean and standard deviation) of the treatment process. Because these steps include all processing except for the hazard mitigation technology treatment process, it is inferred that variability in RA data greater than observed in the isolated control tests can be attributed to variability of the treatment process for a given agent-material-feature combination. The verification data demonstrated that the agent can be delivered, extracted, and quantified with an acceptable high degree of repeatability (e.g., average CV of 8%) for most agents and materials. These results demonstrated Validation goals 1.1 and 1.2.

13.3 RA Data Element Variability

Validation goal 1.3 was to demonstrate the single day repeatability of different treatment procedures (i.e., the CV_{Log}). There was no threshold criterion established for CV_{Log} as part of the methodology assessment. The variability observed in an RA measurement after a treatment process will stem from both the ability to reproducibly generate an agent distribution in or on the material as well as the influence of the treatment process. For example, if the spray duration, pressure, or angle were altered for the soapy water spray, a different CV_{Log} may be observed. It is thought that the variability of RA results after a treatment process primarily reflects the variability of the treatment process being evaluated. The test method (treatment and analysis procedures) provides *a characterization of the effect of the treatment process* (i.e., mean value *and the variability* of the RA measurement). It is assumed that the variability of RA data observed in panel data represents the variability that would be observed when decontamination is conducted at the asset scale.

Testing using two decontamination technologies was conducted to characterize RA-based source terms, and quantify the reproducibility and intermediate precision standard deviation (IPSD) as a measure of repeatability (Section 7). All testing was performed with the agent HD on horizontal orientation materials including steel, two coatings, and an elastomer silicone. Two treatments, soapy water spray and chemical hot air decontamination (CHAD) were evaluated.

The data were identified to be highly skewed, and the use of log transformation was used. The CV_{log} was characterized for each material-feature-treatment combination and identified trends related to shim size, with flat panels having the least variability (e.g., CV_{log} of 4–25% for coatings or silicone), with steel presenting larger CV_{log} values up to 150%. Shim panel results had higher RA values and sometimes larger CV_{log} values from 6–100% for coatings or silicone. In the case of the steel 2 mil shim with CHAD (CV_{log} of 19000%) it seems the vaporous technology either completely removed agent from the feature, or did not access the agent in all cases. In contrast, with soapy water spray the steel 2 mil shim was much more reproducible (CV_{log} of 6.2% for the 1 µL drop) but minimal efficacy was observed (i.e., most of the agent remained in the feature). These types of observations are a demonstration of how the

variability of the RA output of the methodology may be different across agent-material-feature-treatment combinations.

A subset of the conditions used for the reproducibility conditions were repeated over five test days to characterize the ability to generate the same answer from day-to-day. The method was evaluated by determining the repeatability and intermediate precision standard deviation (IPSD) for each sample type (DCS, and RA). DCS control sample IPSD levels demonstrate the ability of the contamination and measurement aspects of the method to produce similar results from day-to-day; the IPSD is 7.9%. The larger IPSD values observed for panels (from 12–233%, excluding the 2 mil steel with CHAD treatment of 7600%) illustrate the variability of the treatment process. The IPSD is a characteristic of the specific agent-material-feature combinations, the treatment steps, the technology, and the application processes used.

13.4 Sensitivity Analysis

The calculation of an asset-level output requires that corresponding laboratory data is available to represent the asset. However, acquiring data for all materials in all orientations for all features (e.g., flat and shim panel) may present significant test burden and *may not be necessary*. A few material-feature combinations may be responsible for the vast majority of retained agent on an asset.[3] However, it is not known *a priori* which material-feature combinations may be the significant contributors. The identification of a high priority material-feature is a combination of source term magnitude, abundance of the material-feature on the asset of interest, and probability of contamination. A test was performed to demonstrate when orientation data or decontaminant spray variations may be *necessary* to enable accurate asset-level outputs (Section 5). The test design included two agents (HD, VX), two panel orientations (horizontal, vertical), both flat and shim panels, two spray angles relative to horizontal (30°, 90°) and two spray variations. An initial threshold of features with greater than 0.4 LD as being sufficiently significant to warrant collection in future test efforts. For example, if the horizontal and vertical orientations produce RA data that is more than ± 0.4 LD (RA different by a factor of antilog(0.4) or 2.5×), then both conditions could be tested, otherwise one condition can be tested and used to represent both orientations.

Overall, no significant differences were observed between orientations for the 2 mil shim case; *orientation had no effect on 2 mil shim RA results for this particular spray treatment*. For the 20 mil shim there were cases where the LD was more than ± 0.4 between orientations. The two cases with statistically significant results were HD on the Alkyd coating with an LD of 0.7 and VX on steel with an LD of –1.3. Sometimes the vertical orientation retained more agent (VX on steel), and other times horizontal orientation retained more agent (HD on Alkyd). For flat panel the HD on steel case was the only condition where there was a statistically significant difference greater than ± 0.4 LD between orientations. The LD of –2.3 (a factor of 200×) is a significant difference, where the geometric mean RA for HD in a vertical orientation was 24.6 µg/drop and horizontal orientation was 0.11 µg/drop. This demonstrates a case where orientation had a significant effect on the RA. Overall, it seems that orientation may have an influence on flat panel testing, and a much smaller influence on shim panel results.

Some asset will include regions of interfaces comprised of different materials and direct contact without an air gap, for example, a weather seal seam with an elastomer to coating interface. Additional testing (Section 9) was conducted evaluating multi-material shim panels to evaluate if the measured RA is significantly different than shim panels constructed of similar

materials. If a wetting material is present for a mixed material interface, it will dominate how the agent liquid spreads Figure 64.[3,4] For decontamination performance, it is anticipated that the gap width, and its influence on flow restrictions, is more influential than wetting characteristics. This could mean that shim panels may be much less influenced by the specific materials of the shim panel, and that not all material combinations need to be tested to represent these feature types on assets.

The results demonstrated that shim panels with washers constructed of dissimilar materials did not provide significantly different source terms than shim panels of similar materials. Based on the test results, it does not seem that shim panels of different materials need to be specifically tested. In cases where wetting is not known or significantly different, using the higher RA/drop material is recommended.

For future studies, the value of ± 0.4 LD was an initial estimate for a source term to be sufficiently different to justify the resources of collecting the data versus using some form of data substitution (e.g., use horizontal orientation test results for all orientations). The contributions of a given source term (i.e., material-feature) to the asset are a combination of the source term magnitude, abundance of the material-feature on the asset of interest, and probability of contamination. Additional studies could identify the LD magnitude (and other parameters) that would identify that collecting a given condition significantly influences asset level output. For example, the methodology assessment included testing two shim sizes, 2 and 20 mil, because initial data demonstrated a *statistically significant difference* in 'small' and 'moderate' size features. However, the test execution cost for collecting data for both size shims should be considered to identify if both sizes are *needed* to provide *sufficiently accurate* asset-level outputs.

13.5 Per Drop Normalization

Validation goal 2 was to demonstrate per drop normalization calculations. Three techniques were used to calculate per drop normalized RA source terms (Section 7). RA data was collected for panels where agent drop volumes ranging from 0.2–4 µL were applied. Each of the source term calculation methods may produce different values based on the assumptions and specific data used. The fit method uses an empirical regression using a non-linear relationship with drop volume constructed using all tested agent drop volumes for the material-feature-treatment. The indirect methods assume a linear relationship of RA with the agent drop volume and use the data from a single agent drop volume to extrapolate results to all other agent drop volumes.

The root mean square error (RMSE) of the fit can be thought of as the *standard deviation of the fit*, similar to the standard deviation of a specifically tested agent drop volume. The RMSE of the fit quantifies both the σ (random error) and the fitting error. If a regression fit is reasonable, then RMSE is an acceptable estimate of σ. Overall, as shown in Section 7.6, the fit method tended to produce the lower RMSE value (i.e., better fit), with indirect fit using 1 µL data providing the next best fit, and indirect fit using 0.2 µL data providing the largest RMSE.

The use of 0.2 and 1 µL agent drop volume contamination of the asset results in the application of a total of (5 × 2 µL) 1 µL or (4 × 1 µL) 4 µL of agent, a four-fold difference in contamination density on the asset. These data also show that the per drop normalization enables the estimation of asset retention for different asset-level contamination densities.

13.6 Scale-up Calculation

The intent of laboratory-based testing for chemical warfare agent hazard mitigation technologies is to characterize how discrete drops of liquid agents interact with materials to determine whether the technology reduces the quantity of agent retained by the material. It is expected that results from laboratory testing indicate how a technology would perform in an operational environment on actual assets. The ability to produce output in this context required new approaches for data analysis. The new scale-up calculation accounts for how each agent drop contributes to the total retained agent mass of the asset; the whole is equal to the sum of the RA per drop. It was identified that the RA data are highly skewed and best described with a log-normal distribution, asset-level output uncertainty (i.e., variability) cannot be calculated with standard propagation of uncertainty approaches using log-normal data. Repeated random sampling Monte Carlo (MC) techniques are better suited to provide the range of response that could be observed at the asset level. Further, this type of MC technique is also ideally suited to enable the random contamination (i.e., random drop volumes interacting with random components of an asset) anticipated for full-scale asset contamination.

Validation goal 3 was to demonstrate the ability of the scale-up calculation to represent an asset. Two types of small assets were contaminated, treated, and extracted to provide a measured M_{Asset} result. In this case, the *contamination was specifically and carefully controlled* to enable the assessment of calculation accuracy between the tested assets and the laboratory scale-up result. The ratio, R, of predicted / measured asset mass is used to assess the accuracy of the calculation. The calculation was considered acceptable if R is within the range of 0.1–10 (i.e., within a factor of 10 high or low).

MC techniques used 10,000 iterations of repeated random sampling to perform the source term aggregation calculation. The MC calculation was conducted using the each of the source term calculation techniques (i.e., fit, indirect using 1 µL drops, and indirect using 0.2 µL drops). For the fit analysis, the calculated geometric mean for M_{Asset} was within factor of 1.7× (under predicting). This was significantly better agreement than anticipated, and within the acceptance criterion.

The indirect technique exhibited less accuracy with geometric mean values within a factor of 12× (underpredicting using the 0.2 µL drop data) and 4.94× overpredicting using the 1 µL drop data. The decrease in accuracy indicates that, for the tested treatment processes using steel, that the effect of agent drop volume on efficacy should be characterized to enable accurate asset-level scale-up results.

The indirect technique may provide less accuracy for asset-level predictions, and is close to the desired R of 0.1–10. Where possible variable drop volume testing is advisable. When using contamination density data or testing resources only allow for fixed agent drop volume testing, the indirect technique may be the only option. In this case, the analysis could recognize the decrease in accuracy and assume an ‘uncertainty factor’ of 10–100 (i.e., assume the result is a factor of 10–100 lower than actual) to provide safe-sided estimates for outputs.

13.7 Maximum Potential Dose Health Effect Output

A new output capability in the methodology involves the use of the M_{asset} output to approximate a maximum potential dose (MPD). RA outputs have typically been used for laboratory-based analysis only. It was previously not clear how a total mass measurement could

be translated for use in vapor or contact exposures. Previous studies have shown that RA reflects the total agent mass that is available for personnel to experience a dose from vapor emission or contact transfer.[8] In this methodology, the principle of RA as an upper bound (i.e., worst case) estimate of available agent mass is used to approximate vapor and contact sources from the asset scale. The total asset mass is assumed to be emitted as a vapor over a specified duration (i.e., steady state vapor emission) or that that all mass produces contact transfer exposure. For most cases, only a fraction of the total mass will be emitted as a vapor or transferred via contact. In the case that all mass is emitted, the MPD provides the estimate for this worst-case situation.

The MPD is provided as an initial tool to evaluate how a technology may perform in operational context before the more expensive vapor and contact testing is performed. Additionally, the calculations associated with MPD are significantly easier and require far fewer detailed inputs than full exposure assessments. These data have demonstrated how laboratory panel data can be used to approximate the response of an asset with per drop normalization and scale-up calculations. The results can be used in an exposure assessment to compare to health-based requirements and evaluate the operational impact, using RA data. A demonstration of the calculations and comparison to conceptual health-based requirements was provided.

13.8 Methodology Implementation

These data demonstrate that the methodology can be utilized to reproducibly execute panel testing. It was demonstrated that the shim panel features enable the characterization of entrained agent effects, which substantially influence the measured source terms used to represent assets. The scale-up calculation was demonstrated to provide accurate representations of asset level RA which can be translated to operational health-effect levels. From these results, the methodology is recommended to be implemented for use in Test and Evaluation of chemical hazard mitigation technologies.

References

1. Boyne, D.A.; Ruth, J.L.; Piesen, J.C.; Mantooth, B.A.; Eikenberg, J.H.; Smallwood, S.Q. Effect of Thickener on Measurement of Simulant Retention and Decontamination Performance for Materials; CCDC CBC-TR-1580; Combat Capabilities Development Command Chemical Biological Center: Aberdeen Proving Ground, MD, 2019. UNCLASSIFIED Report (AD1073141), Public Release (AD1073141).
2. Varady, M.J.; Riley, P.C.; Mantooth, B.A.; Schenning, A.M.; Fouse, J.C.; Pearl, T.P. Evaporation and Degradation of A Sessile Droplet of VX on an Impermeable Surface; ECBC-TR-1478; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2017. UNCLASSIFIED Report (AD1039376), Public Release
3. Hawbaker, N.A.; Mantooth, B.A.; Pearl, T.P. Effect of Complex Features, Surfaces, and Interfaces on Post-Decontamination Vapor Emission from Contaminated Materials DEVCOM CBC-TR-1734; U.S. Army Combat Capabilities Development Command Chemical Biological Center: Aberdeen Proving Ground, MD, 2022. Public Release Report (AD1157423)

4. Mantooth, B.A.; Willis, M.P.; Myers, J.P.; Lalain, T.A. Development and Demonstration of the Complex Test Panel Methodology; ECBC-TR-994; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2012. UNCLASSIFIED Report, Distribution C (ADB404306).
5. Willis, M.P.; Mantooth, B.A.; Lalain, T.A.; Fouse, J.C.; Riley, P.C. Development of the Complex Coupon Test Panel Methodology: A Physics-Based Dynamic Contact Angle Evaluation; ECBC-TR-1002; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2012. UNCLASSIFIED Report (ADB391877).
6. Davies, J.P.; Mantooth, B.A. Chemical Agent Resistance Method Multi-Laboratory Validation; ECBC-TR-1449; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2017. UNCLASSIFIED Report, Distribution C (AD1039332).
7. Mantooth, B.A.; Lalain, T.A.; Pusey, S.M.; Willis, M.P.; Davies, J. Development of the Chemical Contaminant and Decontaminant Test Methodology Source Document: Test Material Treatment, Remaining Contaminant, Contact Test, and Method Validation; ECBC-TR-991; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2013. UNCLASSIFIED Report (ADB392004)
8. Mantooth, B.A.; Stevenson, S.M.; Escarsega, J.; Pearl, T.P.; Ruth, J.L. Development of a Chemical Agent Resistance Method For Chemical Agent Resistant Coatings; CBC-TR-1608; U.S. Army CCDC Chemical Biological Center: Aberdeen Proving Ground, MD, 2020. UNCLASSIFIED Report, Distribution C
9. Mantooth, B.A.; Myers, J.P.; Sheahy, M.L.; Pearl, T.P.; Chesebrough, M.J.; Ruth, J.L.; Piesen, J.C. Effect of Chemical Purity on Measurement of Agent Resistance and Decontamination Performance for Materials; ECBC-TR-1537; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2018. UNCLASSIFIED Report, Public Release (AD1059274).
10. Smallwood, S.Q.; Mantooth, B.A.; Sheahy, M.L.; Pearl, T.P.; Chesebrough, M.J.; Ruth, J.L.; Piesen, J.C. Effect of Using Stabilized VX on Measurement of Agent Retention and Decontamination Performance for Materials; ECBC-TR-1559; U.S. Army Combat Capabilities Development Command Chemical Biological Center: Aberdeen Proving Ground, MD, 2019. UNCLASSIFIED Public Release Report (AD1069284)
11. Mantooth, B.A.; Pearl, T.P.; Stevenson, S.M.; Smallwood, S.Q.; Eikenberg, J.H.; Sheahy, M.L.; Gehring, D.G.; Chesebrough, M.J.; Ruth, J.L. Normalization of Contamination Conditions for Measurement of Agent Retention and Decontamination Performance on Materials; CCDC CBC-TR-1767; DEVCOM Chemical Biological Center: Aberdeen Proving Ground, MD, 2022. UNCLASSIFIED Report, Public Release (AD1170213).
12. Mantooth, B.A.; Willis, M.P.; Procell, L.; Davies, J. Transport and Reactivity of Decontaminants to Provide Hazard Mitigation of Chemical Warfare Agents from Materials; ECBC-TR-1383; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2016. UNCLASSIFIED Report, Public Release (AD1011071).
13. Mantooth, B.A.; Stevenson, S.M.; Varady, M.J.; Pearl, T.P.; Boyne, D.A.; Hulet, M.S.; Escarsega, J.; Levine, F. Influence of Variable Sample Preparation on the Chemical Resistance of Paint Coatings Including Free Films, Accelerated Cure, and Pigment Types;

ECBC-TR-1551; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2018. UNCLASSIFIED Report, Distribution C (AD1063217).

14. Shue, M.; Lalain, T.; Mantooth, B.; Humphreys, P.; Hall, M.; Smith, P.; Sheahy, M. Low-Level Analytical Methodology Updates to Support Decontaminant Performance Evaluations; ECBC-TR-883; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2011. UNCLASSIFIED Report, Public Release (ADA546021).
15. Lavagnini, I.; Magno, F.; Traldi, P. A Statistical Overview on Univariate Calibration, Inverse Regression, and Detection Limits: Application to Gas Chromatography/Mass Spectrometry Technique. Mass Spectrom. Rev. 2007, 26, 1-18.
16. Lalain, T.; Mantooth, B.; Shue, M.; Pusey, S.; Wylie, D. Chemical Contaminant and Decontaminant Test Methodology Source Document, Second Edition; ECBC-TR-980; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2012. UNCLASSIFIED Report, Public Release (ADA566601).
17. Limpert, E.; Stahel, W.A.; Abbt, M. Log-normal distributions across the sciences: Keys and clues. Bioscience 2001, 51 (5), 341-352.
18. Mantooth, B.A.; Pinto, N.A.; Varady, M.J.; Stevenson, S.M.; Hall, M.; Mendez, S.; Pearl, T.P.; Ulmes, K.M. Health Effects Test Guide: Implementation Demonstration And Case Studies; CBC-TR-1603; U.S. Army Combat Capabilities Development Command Chemical Biological Center: Aberdeen Proving Ground, MD, 2019. UNCLASSIFIED Report, Distribution C (AD1085115).
19. Splinter, K.; Sigler, G.; Kolsti, K. Tolerance Intervals Demystified; STAT COE-Report-09-2020; Air Force Institute of Technology, STAT Center of Excellence: 2950 Hobson Way – Wright-Patterson AFB, OH 45433, 2020. UNCLASSIFIED, Distribution Unlimited
20. Limpert, E.; Stahel, W.A. Problems with Using the Normal Distribution - and Ways to Improve Quality and Efficiency of Data Analysis. PLoS One 2011, 6 (7).
21. Aitchison, J.; Brown, J. The Log-normal Distribution. Cambridge University Press: Cambridge (UK), 1957.
22. Accuracy (Trueness and Precision) of Measurement Methods and Results Part 3: Intermediate Measures of the Precision of a Standard Measurement Method; 5725-3; ISO: 1994.
23. D'Onofrio, T.G.; Davies, J.P.; Steinbach, C.B.; Ruppert, C.J. Low-Volatility Agent Permeation (LVAP) Verification and Validation Report; ECBC-TR-1274; ECBC: APG, MD, 2015. UNCLASSIFIED Report Public Release
24. Horwitz, W.; Albert, R. The Horwitz Ratio (HorRat): A Useful Index of Method Performance with Respect to Precision. Journal of the AOAC International 2006, 89, 1095-1109.
25. Myers, J.P.; Morrissey, K.M.; Piesen, J.C.; Ruth, J.L. Decontamination of Personal Effects Using Humidified Chemical Hot Air Decontamination (CHAD); CBC-TR-1530; CCDC Chemical Biological Center: Aberdeen Proving Ground, MD, 2018. Public Release, Distribution Unlimited (AD1093265).

26. EPA Exposure Factors Handbook (Final Report); EPA/600/P-95/002F a-c; National Center for Environmental Assessment, Office of Research and Development, U.S. Environmental Protection Agency: Washington, DC, 1997.
27. Ivancic, W.A.; Nishioka, M.G.; Barnes, R.H.; Hubal, E.C.; Morara, M.; Bortnick, S.M. Development and Evaluation of a Quantitative Video-fluorescence Imaging System and Fluorescent Tracer for Measuring Transfer of Pesticide Residues from Surfaces to Hands with Repeated Contacts. The Annals of Occupational Hygiene 2004, 48 (6), 519-532.
28. ten Berge, W.F.; Zwart, A.; Appelman, L.M. Concentration-Time Mortality Response Relationship of Irritant And Systemically Acting Vapours and Gases. J. Hazard. Mater. 1986, 13 (3), 301-309.
29. Acute Toxicity Estimation and Operational Risk Management of Chemical Warfare Agent Exposures; USACHPPM 47-EM-5863-04; U.S. Army Center for Health Promotion and Preventive Medicine: Aberdeen Proving Ground, MD, May 2004. UNCLASSIFIED Report, Public Release (ADA426988).
30. Health-Based Chemical Vapor Concentration Levels for Future Systems Acquisition and Development; USACHPPM 64-FF-07Z2-07; U.S. Army Center for Health Promotion and Preventive Medicine: Aberdeen Proving Ground, MD, February 2008. UNCLASSIFIED Report
31. Ott, W.; Klepeis, N.; Switzer, P. Air change rates of motor vehicles and in-vehicle pollutant concentrations from secondhand smoke. Journal of Exposure Science and Environmental Epidemiology 2008, 18 (3), 312-325.
32. Mantooth, B.; Lalain, T.; Willis, M.; Myers, J. Development of the Chemical Contaminant and Decontaminant Source Document Vapor Emission Test Methodology; ECBC-TR-941; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2011. UNCLASSIFIED Report (ADB386183)
33. Standard Guide for Small-Scale Environmental Chamber Determinations of Organic Emissions from Indoor Materials/Products; D 5116-06; ASTM International: West Conshohocken, PA, 2006.

ACRONYMS AND ABBREVIATIONS

θ	equilibrium contact angle
CASARM	chemical agent standard analytical reference material
CI	confidence interval
CT	contact transfer
CV	coefficient of variation
CWA	chemical warfare agent
DCS	dose confirmation samples
GC	gas chromatography
HD	bis(2-chloroethyl) sulfide
LC	liquid chromatography
LD	log difference
MC	Monte Carlo
MPD	Maximum Potential Dose
M&S	modeling and simulation
M/W	methanol/water
PU	polyurethane
PI	prediction interval
RA	retained agent
RMSE	root mean square error
SE	standard error
TI	tolerance interval
VCSC	vapor composite system calculation
VE	vapor emission
VX	*O*-ethyl-*S*-(2-diisopropylaminoethyl) methyl phosphonothiolatephosphonothioate

Blank

Appendix A: Methodology Development Background

A.1 Definition of Tiered Laboratory-based Method Outputs for Addressing Operational Conditions

With the expansion to incorporate different panel types in this updated method comes modifications to various high-level aspects associated with execution of hazard mitigation technology test and evaluation. Most importantly, there is a need to define how data generated by the methodology in the laboratory are analyzed for operational relevancy. This end-use perspective is required to ensure the methodology and calculations are providing the most accurate and reproducible results, using the simplest and least resource intensive approach possible.

The updated method covers the procedures for testing and evaluating the efficacy of chemical agent decontaminants, Figure A-1 illustrates the methodology. The *treatment procedure* includes the contamination and application of hazard mitigation technology(s) to test materials. For each test panel*, one of three laboratory *analysis methods* may be performed including RA, vapor emission (VE), or contact transfer (CT). The analysis methods produce the measured data elements of the methodology. These data elements are used in *calculations* that produce various *outputs* depending on the type of data that is collected. For RA, either material-level performance, i.e., source reduction, can be determined, or scale-up outputs associated with asset-level retention can be generated. For VE and CT, these data are used in scale-up calculations for vapor or percutaneous exposure assessments. The details and examples of these outputs are covered elsewhere.[1]

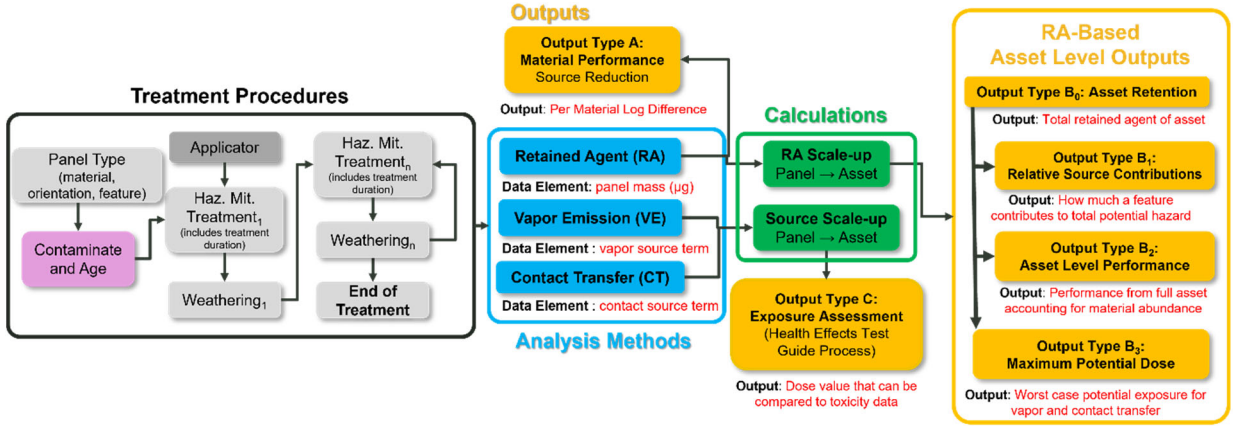


Figure A-1. Inputs/settings for the updated method structure, including treatment procedures, analysis methods, as well as calculations and outputs.

* The term, panel, refers to any test article, with simplified geometry, meant to represent the surface of an asset. Panels can be flat, shim, horizontal, vertical, or composite, and can range in size provided they conform to available test infrastructure. This method focuses on the testing of panels, as opposed to assets (which can be full-scale or scaled down equipment items).

This methodology assessment documents the ability to produce type A and B outputs, both of which involve the RA analysis method for flat (both horizontal and vertical) panels and shim panels. The three analysis methods measure related information regarding the agent remaining on a panel after a treatment procedure. The RA analysis method quantifies the total quantity of agent retained by a test item and the VE and CT analysis methods measure time-resolved rates for release of the RA by the material. The RA represents the total quantity of agent that may be emitted as a vapor or transferred via contact and can be used as an upper bound estimate for total potential exposures, the maximum potential exposure that could occur from an asset. The various analysis methods require significantly different levels of effort, both in data collection and analysis. The RA test can be conducted with higher throughput and *significantly* less effort. It is estimated that the RA analysis method can be performed with about 10% of the resources (time and cost) compared to equivalent testing and analysis for collecting VE and CT data. This methodology assessment effort focuses exclusively on the RA analysis method.

The methodology is comprised of several layers. The core of the methodology is the set of actions performed in the laboratory that comprise the *treatment procedures* (i.e., the contamination and decontamination of a panel) and *analysis methods* that produce specific data elements (i.e., RA source terms, vapor emission source terms, contact transfer source terms). Data elements are used in various calculations to analyze efficacy at the material or asset levels. Based on the objective of the test, data from different materials and test conditions may be required. For example, to represent one asset would require identifying the materials of construction and what complex features would need to be tested to enable the desired calculations. The ability to represent a different asset may require data for other materials. The methodology components to generate data elements do not change, but program specific needs will identify the different data conditions required to produce the desired outputs. A test guide is implemented in the methodology to facilitate the identification of the data needs to enable specific outputs.

The ability to represent asset-level output requires that data for the materials of construction are available. However, it is not practical (or necessary) to test all the materials of construction for all assets of interest. Additionally, previous results have shown that the agent drop volume influences efficacy. Data was acquired in this validation effort to determine if the accuracy of asset level calculation is influenced by inclusion of variable drop volume conditions. This does not alter the treatment procedures or analysis method, only the guidance on what data to acquire to provide acceptable accuracy for the desired outputs using the least testing resources necessary. As part of the test strategy the materials and salient operational contamination conditions to capture in a test are program specific and would need to be identified.

A.1.1 Methodology Calculations and Outputs

There are two analysis methods that can be used to support three calculated output types of the methodology, A, B, and C, as indicated in Table A-1. These output types range from assessing how different material surfaces, including entrained geometries, may alter technology efficacy to translating how different material configurations contribute to total agent retention and full exposure assessments using VE and CT source terms. The methodology is designed to enable these questions to be answered using laboratory data to address operational contexts. Output C is a topic of the Health Effect Test Guide (HETG),[1] and beyond scope of this effort. It is described here to indicate the type of output that requires VE/CT source term data.

Table A-1. Output types for decontamination methodologies.

Analysis Methods	Output Type	Output Name	Example Question(s)	Example Output/Answer
Retained Agent (RA)	A	Material Performance	What is the log reduction for a specific surface (material, orientation, and feature) for a given agent under specific test conditions? Did alternate test conditions (i.e., technology, duty cycle, etc.) provide better performance? [technology optimization or comparison]	Log reduction (compared to a reference condition such as initial challenge) equals X Technology A performed better than technology B by a factor of X Duty cycle A showed a factor of X better performance than duty cycle B
	B	B_0: Asset Level Retained Agent	How much agent is associated with an asset after a treatment process	Calculation basis used for other asset level evaluations (Output types B_1, B_2, and B_3)
		B_1: Relative Source Contributions	What materials contributed the most to the total potential source from an asset? What materials/features are highly influential and should be the focus of testing/optimization?	Used to prioritize materials for testing Under the tested conditions, 75% of the total asset source term is attributable to material A (high influence on hazard)
		B_2: Asset Performance	By how much was the total source reduced from the full asset as a result of hazard mitigation actions?	Use of technology under the tested condition resulted in a 3 log reduction for the full-scale asset compared to the reference condition (e.g., weathering alone). Answers similar to output A but at the asset level
		B_3: Maximum Potential Dose	What is the maximum potential dosage that could result from personnel interacting with this asset?	If the maximum dosage is less than relevant toxicity values, all detailed exposure assessments will be below the toxicity value or, the maximum dosage is above toxicity values, detailed exposure assessment is needed to provide a more accurate assessment
Vapor Emission (VE) and/or Contact Transfer (CT)	C	Exposure Assessment (HETG)	Would health effects be observed for vignette X after using the technology at test condition Y? If reissue time of the asset was delayed for 2 hours, would health effects be observed?	Exposure assessments conducted using VE or CT source terms to answer: For vignette A, the vapor dose to personnel would be X (mg min m^{-3}), which is over the requirement for no greater than 25% of personnel exhibiting mild health effects

A fundamental assumption in evaluation of hazard mitigation technologies in the laboratory is that the results observed on panels can be related to full-scale assets in operational context. However, a comprehensive procedure for this type of data use has been lacking that allows for health-based requirements to be properly addressed in hazard mitigation technology evaluations. This updated methodology leverages past efforts from the HETG,[1] along with work on the design of the shim panel,[2] to arrive at a means for translating laboratory scale data to an operational context. The process requires accounting for the abundance of materials, including their probability of being contaminated and the magnitude of agent retention after

decontamination. It was found that the ability to effectively translate results for flat panels and complex features is facilitated using a per drop normalization,[2,3] rather than starting challenge based on surface averaged contamination density used in the previous approaches (e.g., HETG). The per drop normalization is the foundation of the approach to scale data from laboratory conditions to operational context and is the basis for the RA-based asset level calculations, *regardless of panel type*. Appendix D of the methodology (separate document) covers how data acquired in a per drop context is translated to full scale assets.

The new RA-based asset performance outputs (type B) are new and significant advancements. The calculations enable the evaluation of how individual material efficacy relates to the operational context by providing a material abundance weighted assessment of a hazard mitigation technology. The updated methodology includes simple calculations, based on the per drop scaling in Appendix D of the methodology, to *approximate* full-scale asset performance. Output B_0, which quantifies the total agent associated with an asset, provides the basis for other type B RA-based outputs. Output B_1, indicates relative source contributions, which material/features(s)/feature(s) most significantly contribute to the asset retention, and can be used to identify high priority areas to improve technology performance (e.g., efficacy for constrained geometries). Output B_2 quantifies the extent of agent removal from an asset, either compared to the initial contamination, to other technologies, or to alternate treatment process conditions. Whereas Output A provides material-feature level information, Output B_2 determines asset level performance that accounts for the abundance of asset materials and features. Output B_3 approximates the worst case for possible health effects by assuming all of the agent associated with the asset results in an exposure dose. While this is an overestimate, it captures the upper-bound maximum potential dose (MPD) that could ever result from personnel interacting with the asset. Performing a full exposure assessment (Output C) requires *significant* operational detail, and non-RA vapor emission or contact transfer data. Output B_3 enables a first approximation of operational exposure using far fewer data and analysis resources.

The ability to determine when personnel can reduce protective posture without observing health effects requires a *systems level analysis* that accounts for the operational vignette (Output C). Details from the vignette must be specified for this output, such as, asset contamination conditions, timing of the decontamination treatments, specific personnel interactions with the decontaminated assets, and hazard mitigation technology efficacy (degree of agent source term reduction) for the asset. The action or effect of the hazard mitigation technology is only one of many interacting factors that determine if health effects would be observed for personnel resulting from the use of decontaminated materials or assets.

Output C is very resource intensive with regards to the type and quantity of data required as well as the methods required for executing the analysis. Therefore, there is a need to provide a much lower burden approximation for exposure dose that can be used for competitive prototyping or process optimization in early program development. It is estimated that an Output C analysis requires roughly 20× more effort (i.e., cost and labor) to conduct than an Output B_3 MPD analysis.

The purpose of a hazard mitigation technology is to reduce the total agent from an asset, as quantified using different Output B types (Figure A-2). The exposure assessment process (Output C) integrates the specific details of how personnel use decontaminated assets. Depending on the operational details, only a fraction of the MPD will result in an actual exposure. Slight changes in how personnel interact with decontaminated assets can have orders

of magnitude effects on exposure. For example, altering TTP involving the use of personal protective equipment (PPE) or timing of decontamination could have significant influence on the actual exposure. Example factors addressed by each output are illustrated in Figure A-3. This is the first time this kind of operational information can be obtained from laboratory testing.

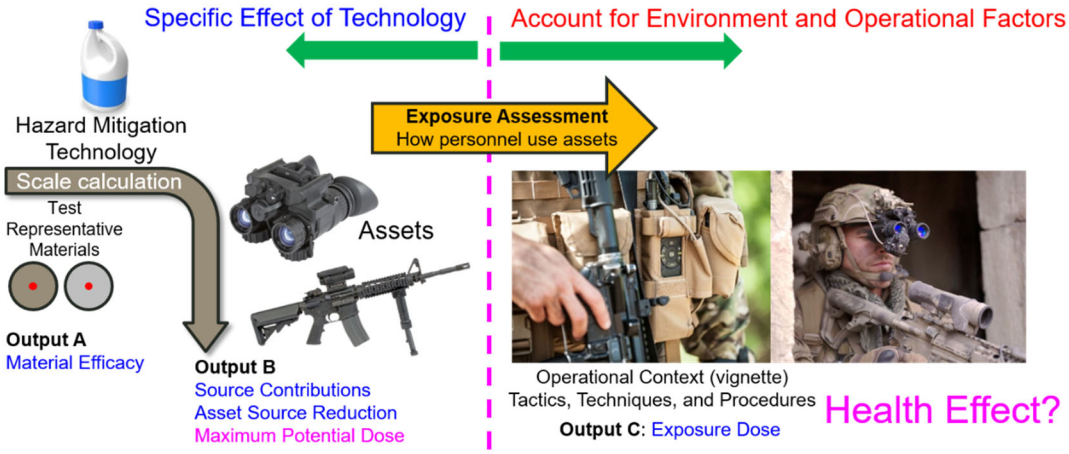


Figure A-2. Differences between Outputs A, B, and Output C. Outputs A and B focus on how the hazard mitigation technology affects agent on the asset, Output C accounts for how personnel interact with the asset after decontamination to determine if health effects would be observed.

☑ Factor accounted for by output
☒ Factor not accounted for by output

Specific Effect of Technology
Accounting for Environment and Operational Factors

	Factor	Material Performance (Output A)	RA-Based Asset Performance (Output B_x)	Exposure Assessment (Output C)
Technology Factors	Decontaminant performance per agent-material-feature	☑	☑	☑
	Variations in treatment process (e.g., timing, multiple treatments, application method, if tested)	☑	☑	☑
	Agent droplet volume effects	☑	☑	☑
Operational Factors	Contamination duration	☑	☑	☑
	Droplet surface distribution	☒	☑	☑
	Post-decon vignette (micro-activity)	☒	☒	☑
	CONOPS	☒	☒	☑
	Alter TTP (e.g., asset reissue timing)	☒	☒	☑
	Alter use of PPE	☒	☒	☑
	Relative effort (complexity, cost & time)	1	2	20
	Analysis method	RA	RA	VE/CT
	Output	Log difference (source reduction)	Maximum potential dose (Asset level source reduction/mitigation)	Exposure Dose (per vignette)

Figure A-3. Factors accounted for by the different method outputs, this validation effort focuses on Outputs A and B.

MPD provides an upper bound for the actual exposure dose from an asset, which may be a significant overestimate (e.g., 1–10,000×); use of the MPD for technology optimization may result in significant overprotection. However, any exposure dose from a given asset can never exceed the MPD. The details of how personnel use the asset are not required. This calculation works best for small items that are carried by personnel for long durations. As assets become large, like vehicles, where personnel do not interact with the full asset nor interact for long durations, the MPD approach may not be appropriate. Also note that the MPD analysis typically focuses on a single asset, if personnel are reissued multiple decontaminated assets, the potential exposure is aggregate. A demonstration of the MPD calculations was provided in Section 12.

A.2 Role of Shim Panels in Methodology Update

Shim panels are a new material feature for the methodology. In most respects, adding a shim panel is similar to the addition of a new flat panel material type. The shim panel is designed to utilize the current flat panel test infrastructure.

A.2.1 Approximating Asset Features

Inspection of most assets reveals key materials of construction, common material interfaces (e.g., seams, weather sealing), and fasteners such as screws, nuts, and pins. Most complex features exhibit some form of restricted geometry that is emulated by the shim panel.[2] There is guidance regarding military asset construction that advises how to minimize structures that lead to agent entrainment, but these measures are not always implemented on the types of assets that may be decontaminated.[4] Previous efforts characterized the difference in vapor emission from complex features versus flat panels on personal hand held electronic assets contaminated with agent (Figure A-4).[5] It was recognized that it is not practical to test all permutations of potential complex features of an asset or set of assets, especially with the variety found on vehicles (Figure A-5). It was determined that most all of these features can be simplified into a gap geometry that can be represented using the shim panel.[2,6,7] Further, two approximate size regimes exist where the size of the gap influences agent retention and decontaminant accessibility (Table A-2). Therefore, it is assumed that the results of the shim panel can be used to approximately represent the efficacy on a per drop basis of all complex features.

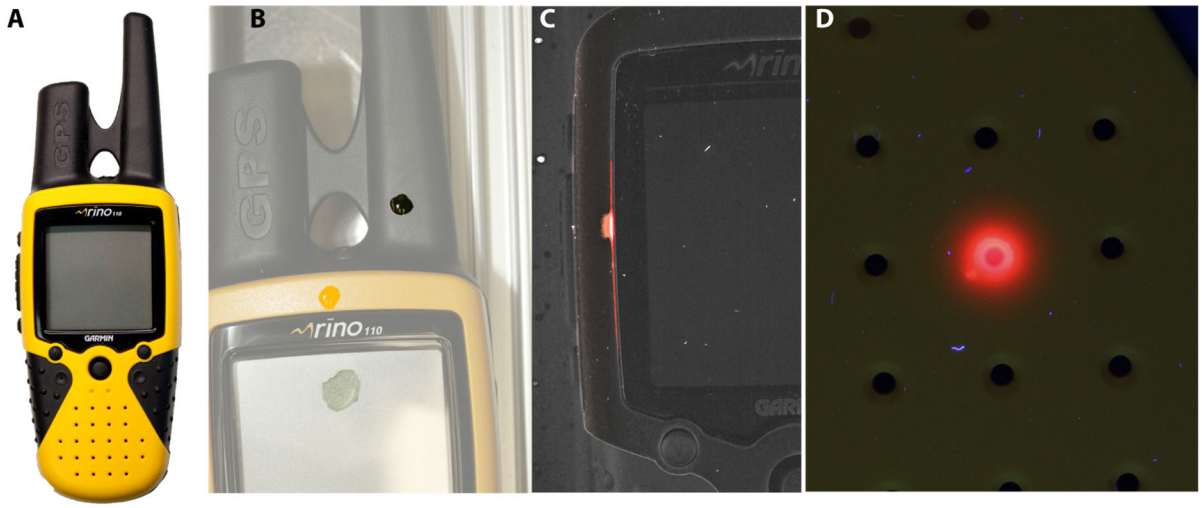


Figure A-4. Examples of flat and complex features on a commercial GPS radio (A), flat materials contaminated with 1 µL of VX (B), a 1 µL fluorescently labeled VX drop (to enable visualization) on the screen-body interface (C), and a speaker hole with 1 µL of fluorescently labeled VX (D).[5]

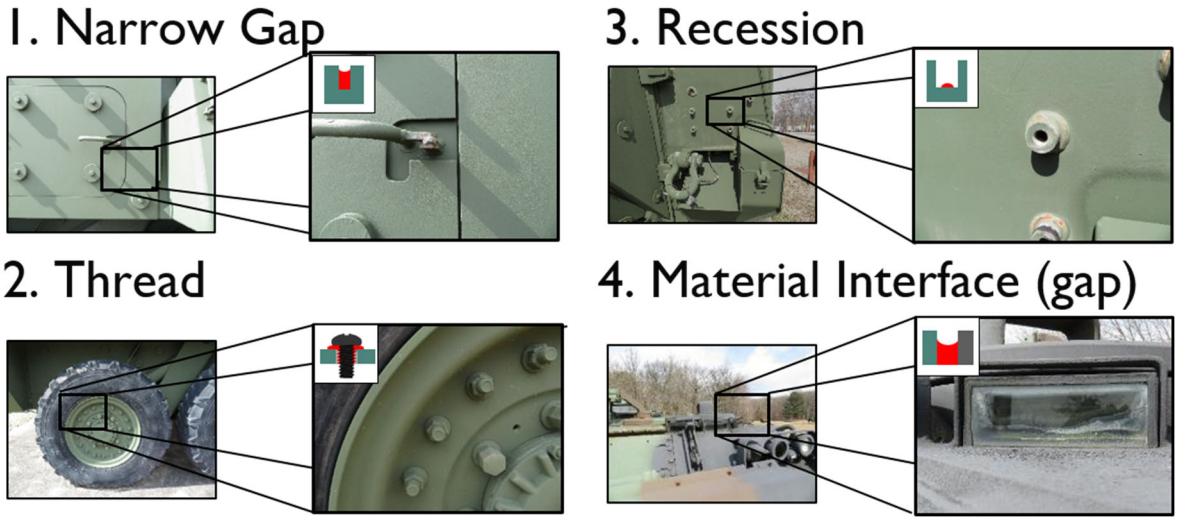


Figure A-5. Examples of constrained geometry features on a vehicle that are represented by a shim panel.

Table A-2. Effects of shim panel feature sizes on agent distributions and decontaminant access.

Complex Feature Size	Agent Distribution	Decontaminant Accessibility	Ability to Decontaminate
Small (<0.005 in)	Likely to draw liquid into feature if wetting	Highly restrictive to fluid (liquid or vapor) flow	May be highly challenging compared to flat panel
Moderate (<0.010-0.020 in)	May draw liquid into feature if wetting	Restrictive to fluid flow	Challenging compared to flat panel
Large (>>0.020 in)	Minimal effects	Minimal restrictions to fluid flow	Similar to flat panel

The purpose of a shim panel is to generate a representative agent distribution in a constrained geometry and characterize the ability of a decontaminant technology to access and remove the agent from the resulting distribution. The intent of the method is to *generically represent* entrainment for a wide range of features that are found on operational assets, rather than emulate entrainment for specific asset features. The data generated by the methodology is intended to represent a full-scale asset by treating the asset as the abundance weighted sum of individual materials and accounting for how many drops may interact with each material and configuration.[3]

A.3 Shim Panel Design

A.3.1 Overview

This new capability enables the characterization of how decontaminants and their application process access and remove agent in ways that were never possible with flat panels. These data show that many technologies are not accessing entrained agent. This is one of the important capabilities of shim panel testing. If entrained agent in small gaps is responsible for most potential exposures, these data will help to identify the factors (e.g., application technique, spray angle, decontaminant liquid properties) to optimize for improvements in decontaminant performance.

There are more possible orientations, gap sizes, and materials than can be realistically considered for testing in most technology evaluation programs. The shim panel is designed to capture the effect of contaminant drop interactions with small air gaps, representative of aspects of typical asset features, which can result in different degrees of restricted access for decontaminants to the contaminant.[2,6,7] The descriptions in this section cover developmental steps for the shim panel hardware and test execution as well as the resultant recommendations for implementation in the updated method.

The details of how liquid decontaminants are applied or sprayed may present some variables such as spray angle and spray velocity that are influential. Alternatively, wipe application may be very different, and factors for vaporous decontaminants may be sensitive to air velocity relative to the shim gap geometry. The purpose of the method is to characterize a decontaminant using the technology-specific prescribed application, the method does not constrain the techniques or factors used in testing.

A.3.2 Shim Panel Design

A diagram illustrating the shim panel assembly is shown in Figure A-6. There are 5 components that comprise the shim panel including two washers of two different radii (1.25 in. diameter for the bottom, 0.75 in. for the top washer), a variable thickness shim to separate the two washers, and then a screw and a nut to hold the assembly together. The result is a shim thickness-dependent air gap between the washers that can serve as a restricted geometry feature. The dimensions of the component materials were chosen to permit the widest range of testing, including compatibility with panel size restrictions for current vapor emission microchambers. The shim panel design enables the top or bottom washer to be any material that can be cut to size. If a flexible material is considered (e.g., weather seal), it could be placed on a stainless steel washer as a rigid support with a more rigid material for the top washer. The variable thickness shim washer and the hardware to hold the shim panel together are made of stainless steel. The screw type and length has been chosen so that the entire shim panel assembly, including the screw head as shown in Figure A-6, is < 0.4 inch, to accommodate the vapor emission microchamber cavity height (shim panel height not as critical for RA analysis methods). The screw is a united national fine (UNF) thread, 10-32, to allow for sufficient adjustment of torque when assembling the panel, with a diameter to match the holes in the shims, and the top and bottom washers.

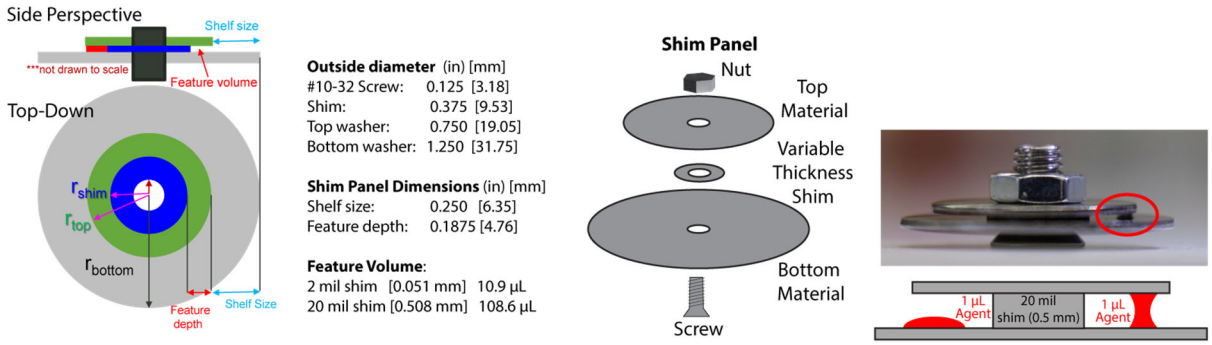


Figure A-6. Generalized schematic of the shim panel and definition of key dimensions.

There is a wide range of feature volumes and depths that could be found on assets (Figure A-5), and because the intent is to generically represent rather than directly replicate them, the shim panel feature characteristics were based on factors that enable testing. The shim panel washer and shim diameters were selected to provide a feature volume that is larger than most drops that may be applied, and a feature depth that could enable the agent to become entrained up to some distance from the interface (Figure A-6). The shelf size is set to be wide enough to deposit a contaminant drop and apply decontaminant, such that the liquids would interact with the gap opening. Additionally, shim panel design was informed by safety considerations associated with handling the panel during chemical warfare agent work in a chemical fume hood.

The components of the shim panel were selected to be ‘off-the-shelf’ as much as possible. Table A-3 lists the parts used for the development and validation testing. Similar parts from any supplier or manufacturer are acceptable. The as-received parts were sonicated in

acetone for 10 min to remove any dirt and oil. The acetone solutions were typically turbid and grey/brown after sonication indicating non-trivial quantities of 'soiling' for the as-received parts.

Table A-3. Part descriptions for the shim panel.

Shim Part	Description	Material	McMaster-Carr Part #	Cost/each ($)
Bottom Washer	Oversized Washer for Number 10 Screw, 0.219" ID, 1.250" OD, 0.043"-0.057" Thick	18-8 Stainless Steel	90313A105	0.8152
Top Washer	Oversized Washer for Number 10 Screw, 0.203" ID, 0.750" OD, 0.033"-0.047" Thick	18-8 Stainless Steel	90313A400	0.0754
Bottom Washer	Oversized Washer for Number 10 Screw, 0.219" ID, 1.25" OD, 0.042"-0.051" thick	Zinc plated steel for coating application	91090A104	0.132
Top Washer	Oversized Washer for Number 10 Screw, 0.203" ID, 0.75" OD, 0.043"-0.051" thick	Zinc plated steel for coating application	91090A126	0.190
Nut	Narrow Hex Nut, 10-32 Thread Size, 5/16" Wide	18-8 Stainless Steel	90730A411	0.0645
Screw	Phillips Flat Head Screw, 82 Degree Countersink, 10-32 Thread, 3/8" Long, Undercut	Passivated 18-8 Steel	91099A355	0.0759
Shim	Ring Shim, 0.02" Thick (20 mil*), 1/4" ID	18-8 Stainless Steel	98126A568	0.412
Shim	Ring Shim, 0.002" Thick (2 mil), 1/4" ID	18-8 Stainless Steel	98126A161	0.831
Bottom washer	Steel Washer, Oversized, Number 10 Screw Size, 0.203" ID, 1.25" OD	Black Oxide (similar to weapons finish)	90377A192	2.016
Top washer	Washer, Oversized, Number 10 Screw Size, 0.203" ID, 0.75" OD	Black Oxide (similar to weapons finish)	90313A400	0.2732

*Note: 1 mil = 0.001 in; mil is hardware and machining vernacular.

For shim panel assembly, a torque wrench was used to apply a reproducible low torque (3 in.-lbs.) to the nut (screw was held in place with a screwdriver). The lower torque value was selected to provide sufficient compression without deforming the washers and altering the shim spacing. This would be most applicable in the cases of non-metal washers.

Testing includes the use of painted washers. The washers were painted on one side and the edges. On inspection of vehicle images (Figure A-5), multiple cases of gaps between painted surfaces exist. The washers are arranged so that the painted sides face each other as shown in Figure 5. In this case, the shim panel presents a coating-coating interface with a variable thickness air gap.

A.3.3 Spacers for Soft Materials

Shim panels that include soft elastomers such as silicone creates a few challenges. First the silicone is not sufficiently rigid to handle the compression associated with shim panel assembly It was observed that when force was applied to tighten the nut, the shim compressed into the elastomer and did not produce the desired gap. A rigid spacer, the same thickness as the material was acquired and a backing steel washer is used to support the elastomer as shown in Figure A-7. The silicone panel was punched so the rigid spacer fit inside the base material and supports the shim and top washer.

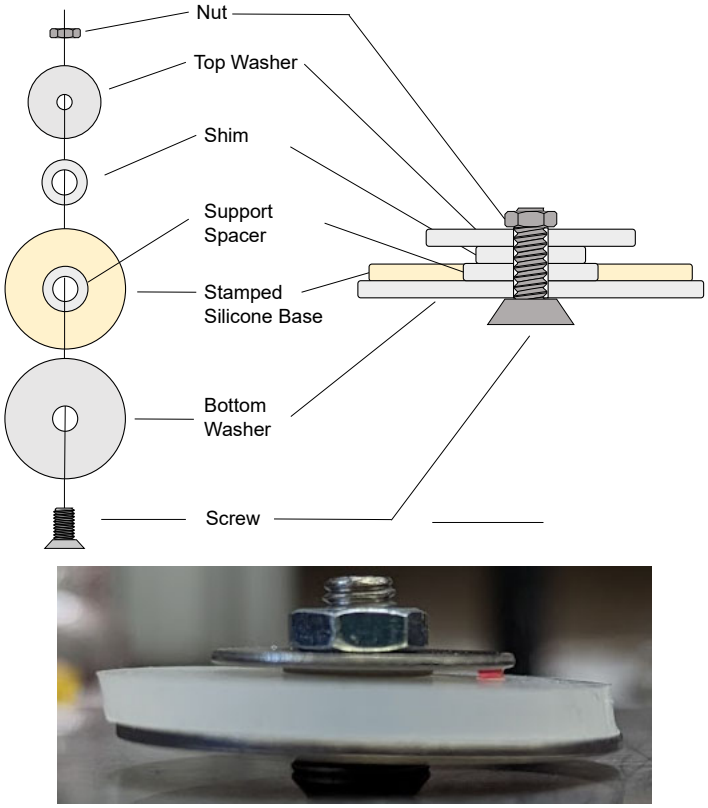


Figure A-7. Support spacers and base for elastomer shim panels.

A.3.4 Shim Panel Gap Spacing

As shown in Figure A-8, developmental evaluations for the shim panels considered 0.001, 0.002, 0.005, 0.010, 0.020 inch spacer thicknesses. The RA analysis method responses can be sorted into two ranges or types, due to ‘small’ and ‘moderate’ gap sizes. Gaps that are 0.001, 0.002, and 0.005 inch (1, 2, and 5 mil) can be categorized as small gaps, which produce results that are different than for moderate gaps, 0.01 and 0.02 inch (10 and 20 mil) (Figure A-8). In both cases, RA responses for shim panels with small and moderate gaps are different than for flat panels. From this, shims of 2 and 20 mil thickness were selected to represent small and moderate size gaps. This reduces testing to two levels, rather than evaluating all potential thicknesses. Further, within the assumptions of the shim panel to generically represent asset features, it is not critical to match explicit gap sizes on an asset, rather identify the preponderance of small versus moderate size gaps. Sections 5 and 7.7 describe the differences in responses observed for the 2 and 20 mil shim thicknesses.

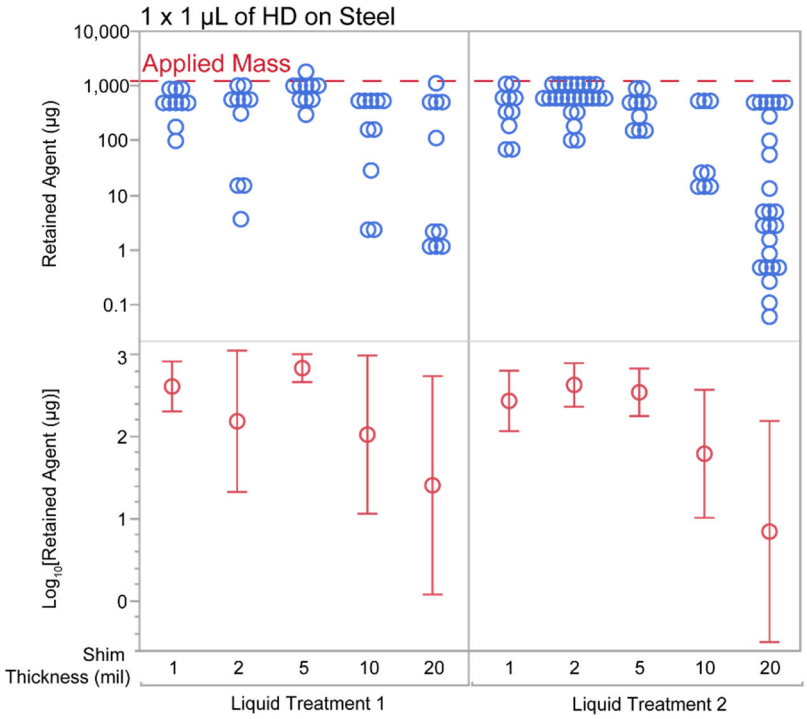


Figure A-8. Effect of shim thickness on RA response for 1 × 1 µL drops of HD and two different liquid treatment processes.

There was no critical requirement established for the tolerance of the gap spacing. The spacings were measured using a Starrett feeler gauge set (model number 66, McMaster-Carr, part 2070A8). Measurements were conducted for four (4) locations on each of 20 shim panels (Figure A-9). For metal materials, including paints applied to steel washers, the shim spacing was with ± 10% of the target gap. The silicone panel uses a rigid spacer to prevent compression of the shim into the elastomer (Figure A-7), therefore, any difference in thickness between the rigid spacer and the material influences the measured gap spacing. This is particularly pronounced for the 2 mil shim spacer for silicone, see Figure A-9. However, since shim panels with 2 mil and 5 mil spacer provided similar RA results for steel (Figure A-8), a range of small gap sizes should still be acceptable for silicone and exact thickness is not critical.

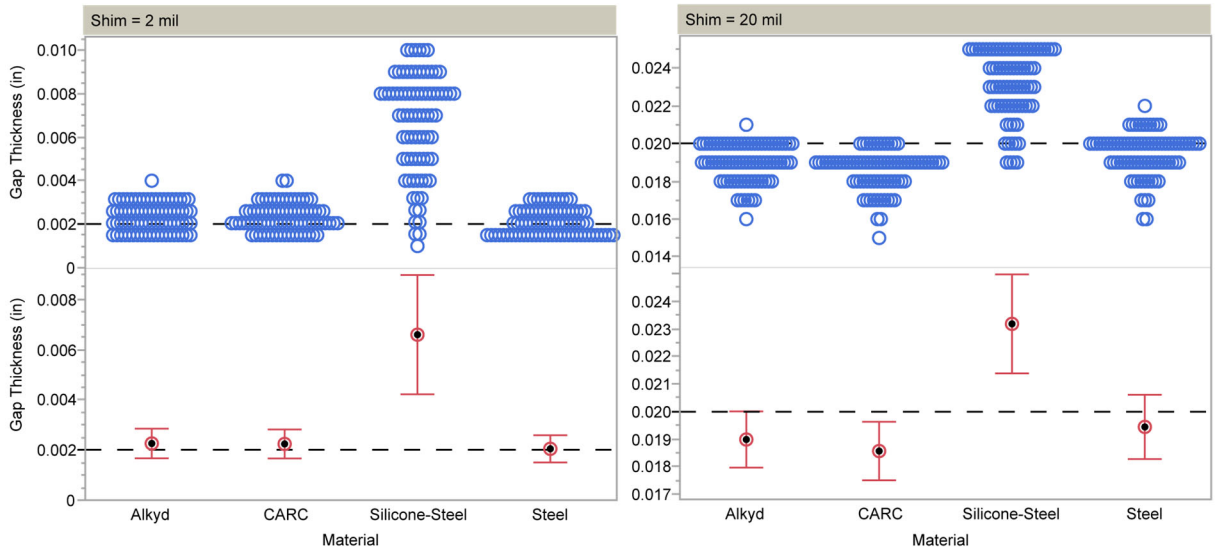


Figure A-9. Gap spacing measured with a feeler gauge across 4 locations on 20 shim panels for panels made of Alkyd, PU, silicone, and steel materials using 2 and 20 mil shims. Top graph shows the individual data points, bottom graph provides the mean and standard deviation. Dashed line shows the expected gap thickness.

Table A-4. Statistical summary of shim thickness gap measurements

Material	Shim Size (inch)	Thickness (inch)	
		Mean	Std. dev
Alkyd	0.002	0.0023	0.0006
	0.020	0.0189	0.0010
CARC	0.002	0.0022	0.0006
	0.020	0.0186	0.0011
Silicone	0.002	0.0066	0.0024
	0.020	0.0232	0.0018
Steel	0.002	0.0021	0.0005
	0.020	0.0194	0.0012

A.3.5 Temperature Effects on Shim Panels

This methodology assessment considered the reproducibility of results and calculation accuracy for ambient laboratory temperature and humidity, but the method can be used for a range of environmental conditions. The ability for the test infrastructure to reach and maintain variable desired environmental conditions is specific to the test infrastructure used. The shim panel should function as a representative material in any environmental condition. Temperature may influence thermal expansion of the shim washer and the resulting air-gap of the shim panel. For the materials used here, the 18-8 steel shim has a thermal expansion coefficient of 17×10^{-6} K^{-1}, therefore for a temperature change of ± 20 ºC the shim would change

thickness by ± 0.0000007 inches for the 0.002 inch shim or ± 0.000007 inches for the 0.020 inch shim; thickness would change by 0.03%. These differences in air-gap thickness of the shim panel are insignificant. For the materials that will be used in the target temperature range, the gap widths will not appreciably change. The variance in the shim thickness and material-washer deformations are probably more influential than the effect of thermal expansion.

Temperature and humidity could influence physical properties of the agent or decontaminant, and the rates of various transport and reactions processes that inform agent distributions (e.g., sorption, evaporation, reaction). The effects of temperature on these processes expected to influence RA measurements significantly more than any physical variations of shim panels due to temperature changes.

A.3.6 Shim Panel Handling During Testing

Development of the shim panel design took into account how handling of the assemblies would occur during agent testing, especially considering the constraints associated with agent operations (e.g., type of gloves). To move the shim panels within the hood for different test steps, operators gripped the protruding screw threads with forceps or tongs as shown in Figure A-10. Panels could also be placed on an extruded aluminum bar with notches to prevent sliding during treatment or processing. Care is taken to minimize splashing created by flowing water over the screw and nut.

Figure A-10. (A) Contamination with an M10 pipette of shim panels placed on an extruded aluminum support bar, (B) spraying the panels while on a support bar with a back splash to catch overspray, (C) post treatment water rinse showing an operator holding the panel by the protruding screw threads.

Some of the testing conducted in this work considered vertical orientations. The beveled screw used to compress the washers includes an angled surface that can be hung on a notch to suspend the panel. A ‘V’-shaped notch was cut into a thin rigid material, for which the beveled screw rests. For this work, the soapy water spray was low pressure and this configuration provided sufficient stability to hold the panels. If more aggressive treatments were to be used, a tighter ‘U’-shaped notch may hold the material with more stability. This style of holder enables the panel to be contaminated and treated without any ‘fixture’ hardware interacting with the contaminated region of the panel or influencing fluid flow.

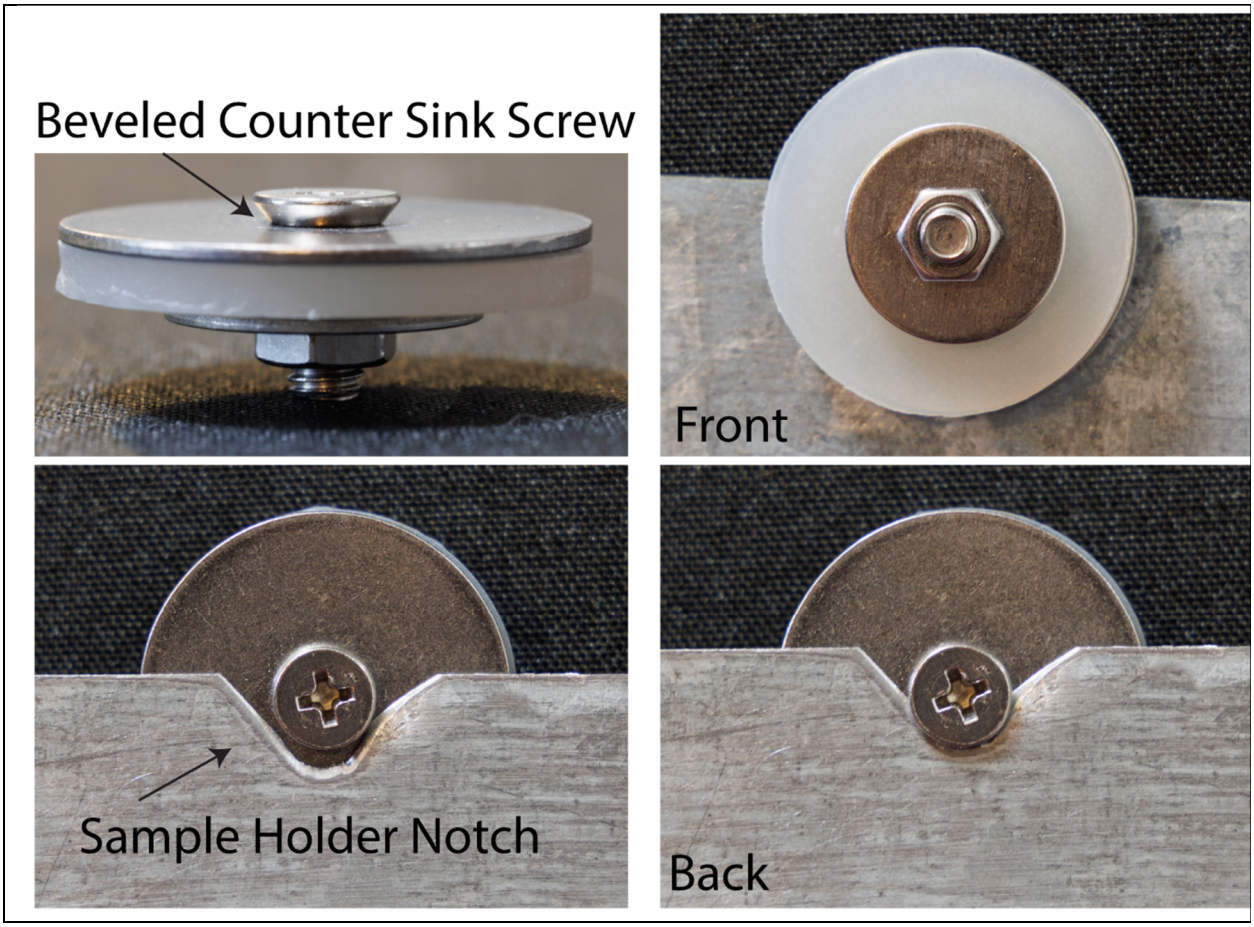


Figure A-11. Vertical orientation holder for shim panels.

A.3.7 Application of Contaminant to Shim Panels

The test procedure involves delivering a specific volume of the agent to the shim panel by directly depositing agent to the shim interface (Figure A-12) via pipette, syringe, or other dispensing device. It is assumed that the agent delivered to a shim panel is entrained in the feature. The actual distribution of agent in the feature (e.g., depth into the feature, spreading within the gap, or spreading out of the gap region) is not measured and is not typically accessible to visually characterize. Some idealized visualization techniques are demonstrated in Appendix F. For this method it is not required that the agent liquid make contact with or bridge both shim surfaces (i.e., top, and bottom washer). The shim panel enables the characterization of how the constrained geometry influences efficacy for a treatment process whether capillary action (i.e., wetting driven flow of the agent) occurred or not.

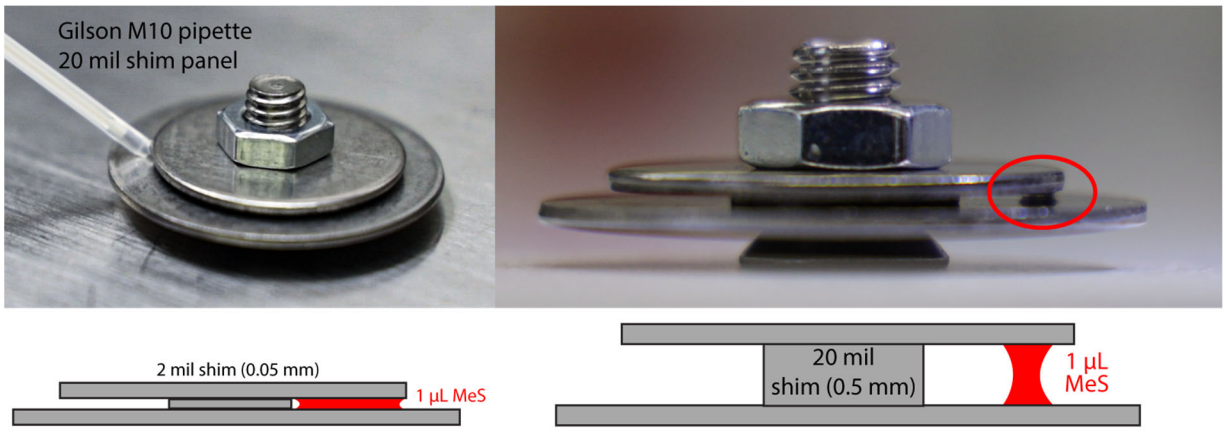


Figure A-12. Photographs of delivery of a contaminant to the shim panel using a pipette. Dimensions listed in the side view schematics correspond to thicknesses of the shims.

Traditionally, flat panel testing has used a repeater pipette to enable the reproducible delivery of multiple drops to material surface. However, the repeater pipette uses an impulse to eject drops from the pipette tip, rather than a more controlled touch off. Shim panel contamination focuses on delivering the contaminant into the gap of the panel. Operators were not able to reproducibly deliver drops in the shim gap using a repeater pipette due to the variance of the direction of drop ejection from the tool. As a result, a single action positive displacement pipette (Gilson M10 or P2 pipette, or syringe) was used to 'touch off' the liquid agent to the feature. Section 4 characterized the ability of pipettes and syringes to deliver the desired quantity of agent to the panel.

It is anticipated that the effect of the entrained agent will vary based on how the drop interacts with the gap (Figure A-13). Drop volumes down to 0.2 µL have been delivered to panels using a syringe pump. Data demonstrations show that the 0.020 inch gap resulted in a significant increase in performance of decontaminants for smaller drops. It is possible that this difference in performance may be related to the drop geometry in the gap feature (i.e., a sessile drop or a bridging drop). Depending on the volume of the drop and the liquid-material contact angle (θ), the sessile drop height could be tall enough to contact the top surface and form a

bridging drop. The materials of the shim panel are typically opaque, and it is not trivial to visualize if a bridging drop is formed (See also Figure G-3). The bridging drop has more liquid-material interfacial area and a different liquid-air interfacial area, and may result in capillary flow of the liquid into the gap. These factors may influence the ability of the decontaminant to remove the entrained agent.

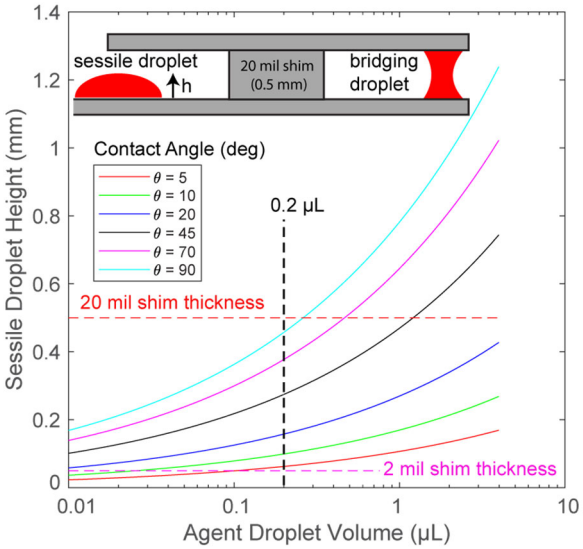


Figure A-13. Sessile drop height relative to the shim gap size for different liquid-surface contact angles.

To control the placement of the drops, the positive displacement tools are placed in direct contact with the shim panel at the gap and the liquid is 'gently injected' into the gap. For the PU coating with 0.002 inch shim, the drop seems to wet and bridge the top and bottom washers and spread along the perimeter of the interface. For the 0.020 inch shim on the PU coating, the drop wets the bottom washer and spreads underneath the top washer in a process known as hemiwicking.[8] For the steel shim panels, the drop was drawn into the gap almost instantaneously.

A.4 Previous Method Validation Efforts

There have been multiple past programs for the establishment of test methodologies associated with hazard mitigation testing of materials. These programs have included sensitivity and repeatability and reproducibility (R&R) studies. Where possible, these studies are leveraged to provide retrospective validation (validation of a method based on previous data, as indicated in ISO 5725) and methodology best practices and concepts. An overview summary is provided in Table A-5.

Table A-5. Previous reports providing validation relevant content.

Topic	Content	Applicable Data
Complex panel effects and contributions[2,6,7]	Sensitivity analysis for feature types, complex and vertical	Complex panel test rationale, data demonstration, focused on VX data, and vertical surfaces. Shim panel demonstration with simulant
Panel Test development[9,10]	Single laboratory study on sensitivity and R&R of RA testing, template for R&R approach	R&R analysis for treatment process (RA data), 2 temperatures, 2 materials, 3 agents (chapter 15)
Chemical Agent Resistance Methodology Development[11,12]	Coatings testing using a specific treatment process and RA analysis method. Relationship of RA, VE, and CT data.	Multi-laboratory study on R&R for a simulant, and HD, GD, VX on two coatings materials (RA data). Only multi-laboratory study known in hazard mitigation area involving the 4 test laboratories that commonly do agent testing.
Sensitivity analysis reports[13-16]	Multiple test settings may influence results, these document the magnitude of the effect	Sensitivity analysis of agent purity, agent stabilizer, agent thickener, dirty surfaces, vertical surfaces, decon applicators, subtle variations in coatings
Contamination parameter effects: drop volume, drop count, starting challenge[3]	Specification of contamination density	Drop volume effects and per drop normalization
Effects of decontamination parameters[17,18]	Physics based modeling of the contamination and decontamination process	Context on how timing may influence test results

As an example,[10,12] previous efforts have used ISO 5725 to determine the repeatability and intermediate precision standard deviation (IPSD) for a water rinse process on several materials.[10] ISO 5725 defines the IPSD of a method as a measure of precision performed for measurements that are made over long time intervals, within the same laboratory, but using different operator-equipment-calibration combinations. The IPSD value is expected to fall somewhere between the standard deviation values for repeatability and reproducibility. In general, a low IPSD indicates a stable method that measures or controls the factors that influence the measured result. The suite of methods provided in ISO 5725[19-21] provides guidance on how to determine IPSD for different measurement techniques and methodologies.

The results of the previous study indicated that while the method and measurement tools performed reproducibly (e.g., DCS were < 10% IPSD), the material being measured generated a wide range of values (e.g., up to 60% IPSD). If the material used for testing is representative of materials that will be used in the field, the range of values may also be observed in the field. Therefore, the IPSD value is also an indication of the range of responses that could be observed in full scale use. The approach of the validation study used the DCS to demonstrate the IPSD of the measurement system and the panel RA data to determine the IPSD of the material and treatment process.

It was generally found that the precision varies with agent-material-treatment combinations. Testing indicated that the variability and reproducibility are related to the agent distribution on the material when the treatment process is conducted. The effect of a treatment process on bulk liquid agent on the panels tends to be highly variable and sensitive to material roughness and treatment application fluid mechanics.

The validation of a new chemical agent resistance method (CARM) for coatings was conducted using a multi-laboratory validation effort.[11] An example output from this four laboratory evaluation is provided in Table A-6. This study included analysis of operator variation across multiple labs, and many other analyses to validate the treatment and analysis procedures. The CARM was based off of the hazard mitigation test methodology[9] where panels are contaminated, a short soapy water immersion treatment process is conducted, and the panel is analyzed with the RA method. Further analysis demonstrated that panel heterogeneity was responsible for the larger reproducibility values; the materials tested should be similar to those found on assets and therefore should reflect the behavior that may be observed at the asset level. Where possible, these data should be utilized to establish the capabilities of this methodology as they share almost identical procedures and analysis.

Table A-6. CARM ISO 5725 Precision Estimates for a multi-day, multi-laboratory validation study on PU coatings.[11]

Agent	Age Time (minutes)	RA Grand Mean (µg)	Repeatability Standard Deviation, s_r (%)	Reproducibility Standard Deviation, s_R (%)	Repeatability Limit "r" (%)	Reproducibility Limit "R" (%)
HD	5	107	35.7	41.7	100.0	116.8
	60	637	74.7	89.0	209.2	249.2
	360	1,269	15.0	44.8	42.0	125.4
MeS	5	539	44.9	50.0	125.7	140.0
	60	784	27.6	45.6	77.3	127.7
	360	483	23.1	57.9	64.7	162.1
VX	5	128	35.2	39.1	98.6	109.5
	60	467	27.3	32.5	76.4	91.0
	360	673	20.7	25.4	58.0	71.1

References

1. Mantooth, B.A.; Pinto, N.A.; Varady, M.J.; Stevenson, S.M.; Hall, M.; Mendez, S.; Pearl, T.P.; Ulmes, K.M. *Health Effects Test Guide: Implementation Demonstration And Case Studies*; CBC-TR-1603; U.S. Army Combat Capabilities Development Command Chemical Biological Center: Aberdeen Proving Ground, MD, 2019. UNCLASSIFIED Report, Distribution C (AD1085115).
2. Hawbaker, N.A.; Mantooth, B.A.; Pearl, T.P. *Effect of Complex Features, Surfaces, and Interfaces on Post-Decontamination Vapor Emission from Contaminated Materials* DEVCOM CBC-TR-1734; U.S. Army Combat Capabilities Development Command Chemical Biological Center: Aberdeen Proving Ground, MD, 2022. Public Release Report (AD1157423)
3. Mantooth, B.A.; Pearl, T.P.; Stevenson, S.M.; Smallwood, S.Q.; Eikenberg, J.H.; Sheahy, M.L.; Gehring, D.G.; Chesebrough, M.J.; Ruth, J.L. *Normalization of Contamination*

Conditions for Measurement of Agent Retention and Decontamination Performance on Materials; CCDC CBC-TR-1767; DEVCOM Chemical Biological Center: Aberdeen Proving Ground, MD, 2022. UNCLASSIFIED Report, Public Release (AD1170213).

4. *MIL-STD-3056 Design criteria for chemical, biological, and radiological system contamination survivability*; MIL-STD-3056; Department of Defense: 2016. (UNCLASSIFIED Distribution C FOUO).
5. Mantooth, B.; Willis, M.; Lalain, T. *Development of the Vapor Composite System Calculation*; ECBC-TR-925; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2012. UNCLASSIFIED Report (ADB378714).
6. Mantooth, B.A.; Willis, M.P.; Myers, J.P.; Lalain, T.A. *Development and Demonstration of the Complex Test Panel Methodology*; ECBC-TR-994; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2012. UNCLASSIFIED Report, Distribution C (ADB404306).
7. Willis, M.P.; Mantooth, B.A.; Lalain, T.A.; Fouse, J.C.; Riley, P.C. *Development of the Complex Coupon Test Panel Methodology: A Physics-Based Dynamic Contact Angle Evaluation*; ECBC-TR-1002; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2012. UNCLASSIFIED Report (ADB391877).
8. Varady, M.J.; Mantooth, B.A. Unsaturated Hemiwicking Dynamics on Surfaces with Irregular Roughness. *J. Colloid Interface Sci.* 2021, 604, 104-112.
9. Lalain, T.; Mantooth, B.; Shue, M.; Pusey, S.; Wylie, D. *Chemical Contaminant and Decontaminant Test Methodology Source Document, Second Edition*; ECBC-TR-980; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2012. UNCLASSIFIED Report, Public Release (ADA566601).
10. Mantooth, B.A.; Lalain, T.A.; Pusey, S.M.; Willis, M.P.; Davies, J. *Development of the Chemical Contaminant and Decontaminant Test Methodology Source Document: Test Material Treatment, Remaining Contaminant, Contact Test, and Method Validation*; ECBC-TR-991; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2013. UNCLASSIFIED Report (ADB392004)
11. Davies, J.P.; Mantooth, B.A. *Chemical Agent Resistance Method Multi-Laboratory Validation*; ECBC-TR-1449; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2017. UNCLASSIFIED Report, Distribution C (AD1039332).
12. Mantooth, B.A.; Stevenson, S.M.; Escarsega, J.; Pearl, T.P.; Ruth, J.L. *Development of a Chemical Agent Resistance Method For Chemical Agent Resistant Coatings*; CBC-TR-1608; U.S. Army CCDC Chemical Biological Center: Aberdeen Proving Ground, MD, 2020. UNCLASSIFIED Report, Distribution C
13. Boyne, D.A.; Ruth, J.L.; Piesen, J.C.; Mantooth, B.A.; Eikenberg, J.H.; Smallwood, S.Q. *Effect of Thickener on Measurement of Simulant Retention and Decontamination Performance for Materials*; CCDC CBC-TR-1580; Combat Capabilities Development Command Chemical Biological Center: Aberdeen Proving Ground, MD, 2019. UNCLASSIFIED Report (AD1073141), Public Release (AD1073141).
14. Smallwood, S.Q.; Mantooth, B.A.; Sheahy, M.L.; Pearl, T.P.; Chesebrough, M.J.; Ruth, J.L.; Piesen, J.C. *Effect of Using Stabilized VX on Measurement of Agent Retention and*

Decontamination Performance for Materials; ECBC-TR-1559; U.S. Army Combat Capabilities Development Command Chemical Biological Center: Aberdeen Proving Ground, MD, 2019. UNCLASSIFIED Public Release Report (AD1069284)

15. Mantooth, B.A.; Stevenson, S.M.; Varady, M.J.; Pearl, T.P.; Boyne, D.A.; Hulet, M.S.; Escarsega, J.; Levine, F. *Influence of Variable Sample Preparation on the Chemical Resistance of Paint Coatings Including Free Films, Accelerated Cure, and Pigment Types*; ECBC-TR-1551; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2018. UNCLASSIFIED Report, Distribution C (AD1063217).
16. Mantooth, B.A.; Myers, J.P.; Sheahy, M.L.; Pearl, T.P.; Chesebrough, M.J.; Ruth, J.L.; Piesen, J.C. *Effect of Chemical Purity on Measurement of Agent Resistance and Decontamination Performance for Materials*; ECBC-TR-1537; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2018. UNCLASSIFIED Report, Public Release (AD1059274).
17. Varady, M.J.; Mantooth, B.A.; Myers, J.; Boyne, D.; Hulet, M.; Pearl, T.P.; Chesebrough, M. *Decontamination System Performance Model Version 5.0: Model Development and Parameter Determination*; CBC-TR-1620; CCDC Chemical Biological Center: Aberdeen Proving Ground, MD, 2020. UNCLASSIFIED Distribution C FOUO (AD1093265).
18. Varady, M.J.; Mantooth, B.A.; Myers, J.; Boyne, D.; Hulet, M.; Pearl, T.P.; Chesebrough, M. *Decontamination System Performance Model: Case Studies for Sprayable Decontaminant Slurry*; CBC-TR-1627; CCDC Chemical Biological Center: Aberdeen Proving Ground, MD, 2020. UNCLASSIFIED Distribution C FOUO (AD1099358).
19. *Accuracy (Trueness and Precision) of Measurement Methods and Results Part 1: General Principles and Definitions*; 5725-1; ISO: 1994.
20. *Accuracy (Trueness and Precision) of Measurement Methods and Results Part 3: Intermediate Measures of the Precision of a Standard Measurement Method*; 5725-3; ISO: 1994.
21. *Accuracy (Trueness and Precision) of Measurement Methods and Results Part 2: Basic Method for the Determination of Repeatability and Reproducibility of a Standard Measurement Method*; 5725-2; ISO: 1994.

Appendix B: Data Transforms and Calculations

B.1 Data Transformations

Decontamination panel test results can often span six orders of magnitude (1,000,000×) or more depending on contamination levels and decontaminant efficacy. The better the decontaminant performs, the smaller the measured mass signal. Skewed data distributions can result due to low mean values and large variance, and also because these measurements cannot have negative values (i.e., mass is bounded by the limit of detection and must be greater than zero; the data domain is left-censored).[1] Consequently, the use of arithmetic mean and standard deviation statistics do not provide accurate descriptions of the data. Additionally, the results produced by chromatography instruments are known to be heteroscedastic (i.e., the standard deviation is proportional to the magnitude). Many of these challenges can be addressed with log transformation of the results.[1-4]

The data are transformed by taking the logarithm of the data before any statistical calculations. Standard statistical moments such as the mean (μ)* and standard deviation (σ) can be used to describe the shape of the resulting 'log-normal' distribution.† The resulting values are in the transformed units (e.g., $\log_{10}(\mu g)$) which can be inverse transformed back to the original units as

$$\mu = \frac{1}{n}\sum_{i=1}^{n}\left(log_{10}(x_i)\right) \quad \text{Eq. B-1}$$

$$\sigma = \sqrt{\frac{(log_{10}(x_i) - \mu)^2}{n-1}} \quad \text{Eq. B-2}$$

$$\mu^* = antilog(\mu) \quad \text{Eq. B-3}$$

$$\sigma^* = antilog(\sigma) \quad \text{Eq. B-4}$$

$$PI = antilog(\mu \pm z\sigma) \quad \text{Eq. B-5}$$

Where

- x_i is the input data (e.g., RA mass, μg)
- n number of samples
- μ is the log-transformed sample mean of the data (e.g., $\log_{10}(\mu g)$)
- σ is the log-transformed sample standard deviation of the data (e.g., $\log_{10}(\mu g)$)

---

* The symbols μ and σ are used here to represent the sample rather than population mean and standard deviation.

† When the data are log-transformed the skew of the data is reduced and a more symmetric normal distribution is obtained which is accurately represented with mean and standard deviation moments.

μ* is the inverse-transformed sample mean (e.g., μg)

σ* is the inverse-transformed sample standard deviation (e.g., μg)

PI is the inverse-transformed asymmetric prediction interval (e.g., μg)

The μ* value corresponds to the geometric mean of the data, which is also the median of the log-normal distribution. The inverse transformed standard deviation requires a different interpretation. Where normal data use μ ± σ, the characteristic of log transformation produces multiplicative rather than additive scaling. In this case μ* $^{\times}/$ σ*, where the $^{\times}/$ symbol indicates times or divided by, resulting in an upper bound of μ* × σ* and a lower bound of μ* / σ*.[1] This is the equivalent result of the PI calculation in Eq. B-5.

The use of arithmetic mean statistics to characterize a data set assumes the data follow a normal distribution. There are cases where the data appear normal (e.g., control samples with small standard deviations), but the treatment process samples produce significantly more variability (i.e., are skewed). It has been common practice to reject potential outliers to make the data seem more 'normal', however, this practice removes the actual skewed distribution and can hide the true behavior of the system under test. A demonstration of the accuracy of analyzing skewed data with and without a log-transform is provided in Section B.2.

It has been observed that normal distribution data are also well described by the log transformation. Therefore, whether the data are skewed or not, the log-transform statistics provide a good description of the data distribution.[2] Typically, control samples produce smaller standard deviations, and decontamination treatment conditions, especially those involving liquid agent on the material surface, tend to produce the skewed, log-normal distributions with much larger variability.

A typical approach to characterize the precision of a measurement is to use a normalized measure of the variance such as the coefficient of variation (CV) which is the ratio of the standard deviation divided by the mean. In the case of log-transformed data, the CV_{Log} is[1]

$$CV_{Log} = [antilog(\sigma^2) - 1]^{1/2} \qquad \text{Eq. B-6}$$

CV_{Log} and the log-transformed standard deviation are used throughout the report to describe relative variance in the data.

B.2 Statistical Analysis on Linear and Log Scales

Synthetic data was generated to illustrate the relationship of the CV on raw data (e.g., RA mass in μg) with the standard deviation of the log-transformed RA. Using the same technique as described in the Monte Carlo calculations, data were produced assuming a log normal data distribution with mean values (μ) of 1 and 3 $\log_{10}$(μg), corresponding to 10 and 1000 μg. Using the randn() function of MATLAB v2022a, 20 replicates of data with known σ in $\log_{10}$(μg) were produced (Figure B-1). The data were then inverse transformed, to represent the type of data collected by the RA analysis method. For each case the CV is calculated on the data in μg, and the standard deviation of the log-transformed results. Ideally the standard deviation of

the log-transformed results should match the input σ value, however, as random sampling with 20 replicates was used, the results will not match exactly.

The analysis of the data in the linear state is provided in Table B-1. The ratio of arithmetic mean to ideal mass shows that as σ increases the accuracy of the arithmetic mean decreases, with cases off by as much as a factor of 188. The middle graphs in Figure B-1 show that for higher values of σ, the resulting standard deviation indicates that mass values less than zero are part of the distribution, which is not physically possible. Table B-2 provides the statistical summary of the data when transformed on a log scale. Note that the calculated standard deviation shows close agreement with the σ value used to generate the data. When the arithmetic mean of the log-transformed data is inverse transformed (Eq. B-3), the result is equivalent to the geometric mean. For the log-transformed data the accuracy of the geometric mean to the ideal mass shows that this log-transformed result is significantly more accurate than the arithmetic mean of the linear scale data.

Next, in Table B-2, note that even though the mean value of the data is different the standard deviation of the log-transformed data matches the σ value used to generate the synthetic data. Because the log-transformed statistics provide a more accurate description of the data, it is useful to recognize that the standard deviation provides a measure of the relative variance of the data (Eq. B-6).[1] A standard deviation of 0.05 corresponds to a CV of 6%, which is often observed for dose confirmation samples (DCS). A standard deviation of 0.1 corresponds to a CV of about 16%, and a standard deviation of 0.2 corresponds to a CV of about 43%. These levels of standard deviation are observed for many of the conditions measured in this report. Some conditions provide standard deviations as high as 1, and very few cases at a standard deviation of 2.

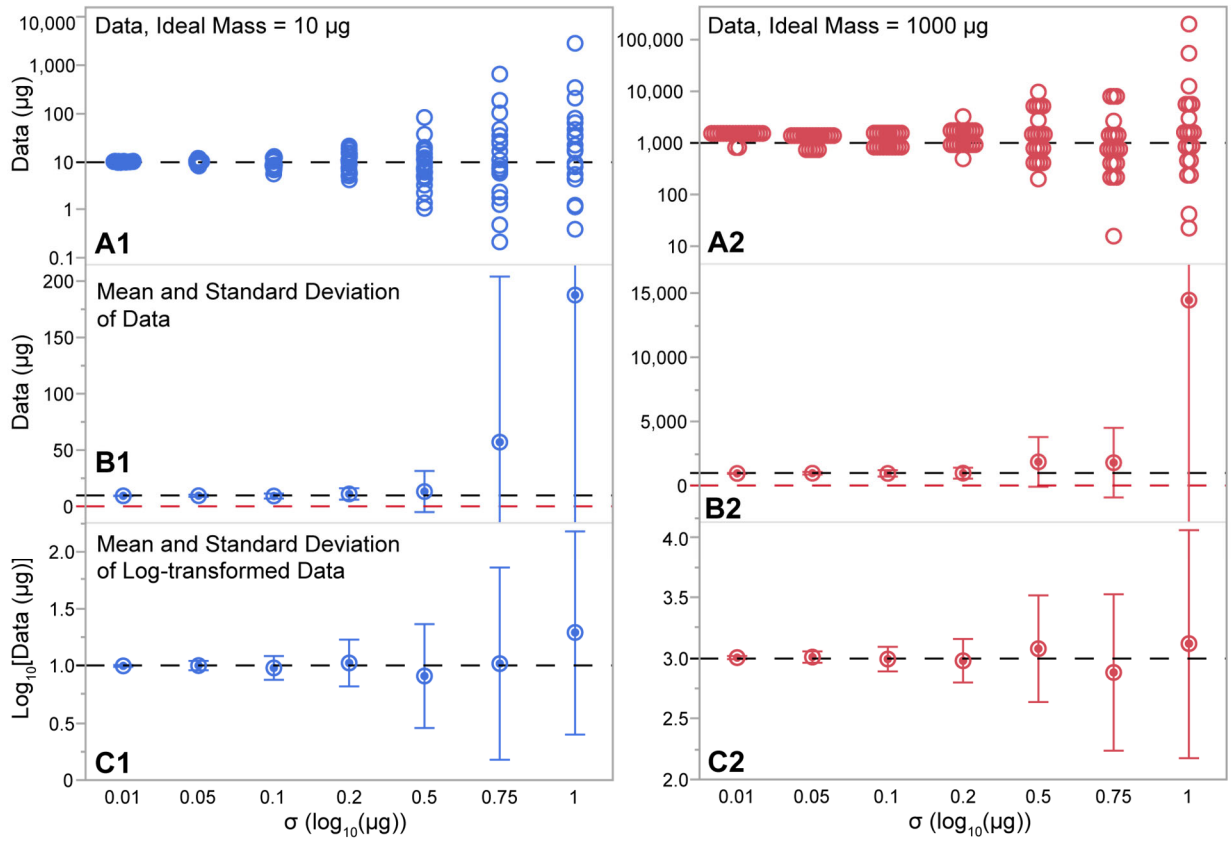


Figure B-1. Synthetic data to compare statistical analysis of direct and log-transforms of the data. The data are jittered in x to facilitate visualization. Top graphs (A) show the data points in µg and the synthetic mean value as a dashed lime, the middle graphs (B) show the mean and standard deviation of the data, the bottom graphs (C) show the standard deviation of the log-transformed data.

Table B-1. Synthetic data summary statistics on linear scale.

Defined Statistical Parameters (log_{10}(µg))		Ideal Mass (µg)	Data (µg)			
µ	σ		Mean	Std Dev	CV	Mean / Ideal Mass
1	0.01	10	9.94	0.20	0.02	0.99
	0.05	10	10.04	0.93	0.09	1.00
	0.1	10	9.79	2.20	0.23	0.98
	0.2	10	11.62	4.99	0.43	1.16
	0.5	10	13.73	18.16	1.32	1.37
	0.75	10	57.41	146.35	2.55	5.74
	1	10	187.39	626.69	3.34	18.74
3	0.01	1000	1004.6	28.8	0.03	1.00
	0.05	1000	1019.8	106.7	0.11	1.02
	0.1	1000	1003.3	245.0	0.24	1.00
	0.2	1000	1024.3	421.8	0.41	1.02
	0.5	1000	1900.2	1937.0	1.02	1.90
	0.75	1000	1839.2	2709.9	1.47	1.84
	1	1000	14494.0	44969.0	3.10	14.49

Table B-2. Synthetic data summary statistics on log-transformed scale.

Defined Statistical Parameters (log_{10}(µg))		Ideal Mass (µg)	log_{10}(Data(µg))		CV_{Log}*	Geometric Mean (µg)	Geo. Mean / Ideal Mass
µ	σ		Mean	Std Dev			
1	0.01	10	0.997	0.009	0.01	9.94	0.994
	0.05	10	1.000	0.041	0.06	10.00	1.000
	0.1	10	0.979	0.104	0.16	9.54	0.954
	0.2	10	1.023	0.204	0.32	10.54	1.054
	0.5	10	0.909	0.453	0.78	8.11	0.811
	0.75	10	1.017	0.840	2.02	10.40	1.040
	1	10	1.289	0.892	2.29	19.46	1.946
3	0.01	1000	3.002	0.012	0.02	1004.17	1.004
	0.05	1000	3.006	0.047	0.07	1014.28	1.014
	0.1	1000	2.990	0.101	0.15	977.05	0.977
	0.2	1000	2.976	0.179	0.28	945.77	0.946
	0.5	1000	3.075	0.439	0.75	1188.76	1.189
	0.75	1000	2.880	0.644	1.26	757.84	0.758
	1	1000	3.117	0.944	2.60	1308.93	1.309

*CV_{Log} is calculated from Eq. B-6

B.3 Base 10 Log Difference Calculation

Relative performance-metric calculations (such as log difference) are used to compare hazard mitigation technologies to a reference (e.g., positive control, reference technology, or alternate treatment condition). The relative decontaminant performance factor (*PF*) metric is defined as the ratio of a reference condition *R* to the technology condition *A*

$$PF \equiv \frac{R}{A} \quad \text{Eq. B-7}$$

For example, if condition *A* is more effective than condition *R*, i.e., the measured agent mass in *A* is less than in *R*, then the *PF* will be greater than 1 and condition *A* is *PF* times more effective than condition *R*. Alternatively, if the measured agent mass in *A* is more than in *R*, the *PF* is less than 1, and condition *A* is less effective than condition *R* by a factor of 1/*PF*. The data collected from these methods are left-censored (i.e., mass detected must be greater than zero) and may also span multiple orders of magnitude. To account for these data range considerations, a log difference (LD) calculation is utilized where LD is defined as the logarithm of *PF*.

$$LD = log_{10}(PF) = log_{10}\left(\frac{R}{A}\right) = log_{10}(R) - log_{10}(A) \quad \text{Eq. B-8}$$

Using this approach, an LD of zero indicates that conditions *A* and *R* are equivalent. If LD is greater than 0, condition *A* is LD orders of magnitude more effective than condition *R*. If LD is less than 0, condition *A* is LD orders of magnitude less effective than condition *R*. The LD is calculated as the difference of the arithmetic mean of the log_{10} transform of the reference and test conditions.

$$LD = \text{mean}[log_{10}(R)] - \text{mean}[log_{10}(A)] \quad \text{Eq. B-9}$$

The 95% CI for the difference in mean values, assuming unequal variance, is expressed as:

$$CI = t_{\left(\frac{\alpha}{2}, DF\right)} SE \quad \text{Eq. B-10}$$

$$DF = \frac{\left(\frac{{S_R}^2}{n_R} + \frac{{S_A}^2}{n_A}\right)^2}{\frac{\left({S_R}^2/n_R\right)^2}{(n_R - 1)} + \frac{\left({S_A}^2/n_A\right)^2}{(n_A - 1)}} \quad \text{Eq. B-11}$$

$$SE = \sqrt{\frac{{S_R}^2}{n_R} + \frac{{S_A}^2}{n_A}} \quad \text{Eq. B-12}$$

Where:

CI	is the confidence interval for LD (units vary)
t	is the t value (unitless)
α	is the probability level (unitless)
DF	is the degrees of freedom (unitless)
n_R	is the number of replicates for condition R (unitless)

n_A is the number of replicates for condition A (unitless)
SE is the standard error of the difference of the means (units vary)
S_R is the standard deviation of the log-transformed data for condition R (units vary)
S_A is the standard deviation of the log-transformed data for condition A (units vary)

CIs can be calculated for different probability levels, expressed as 1-α. In this case, to calculate a 95% CI, α = 0.05. The t value is a function of α and the DF in the system. The t value can be obtained from reference tables or from commercial software packages. The performance factor can be determined by the base 10 inverse transformation of the LD

$$PF = \text{antilog}(\text{LD}) \qquad \text{Eq. B-13}$$

References

1. Limpert, E.; Stahel, W.A.; Abbt, M. Log-normal distributions across the sciences: Keys and clues. *Bioscience* 2001, 51 (5), 341-352.
2. Limpert, E.; Stahel, W.A. Problems with Using the Normal Distribution - and Ways to Improve Quality and Efficiency of Data Analysis. *PLoS One* 2011, 6 (7).
3. Box, G.E.; Hunter, S.J.; Hunter, W.G. *Statistics for Experimenters: Design, Innovation, and Discovery*. 2nd ed.; John Wiley & Sons, Inc.: Hoboken, NJ, 2009; 664.
4. Aitchison, J.; Brown, J. *The Log-normal Distribution*. Cambridge University Press: Cambridge (UK), 1957.

Appendix C: Starting Challenge - Relationship of Environmental Contamination Density to Asset Contamination

C.1 Contamination Context

Decontamination requirements often specify the quantity of agent to be decontaminated as a starting challenge, expressed as grams per square meter (g/m^2). This value has historically been interpreted as grams of agent per square meter of *material* and consequently, contamination conditions of panels in tests have been specified this way.* However, the contamination density value is more likely associated with grams of agent per square meter of terrain, the distribution of agent in the environment. This interpretation has a significant influence on the quantity of agent that may be associated with an asset for a given contamination event. The following descriptions provide a new perspective on how to determine the contamination of an asset to enable scale-up calculations. The first step is to determine the total mass of agent that would be applied to the asset. The proposed approach defines an approximate area term that can be used with contamination density to determine the mass of liquid agent that contaminates the asset. Subsequent calculations will subdivide the total mass into drop distributions and apply drops to various regions of the asset to enable the per drop scale-up calculations.

The *starting challenge* is the environmental contamination density which describes the grams of agent per square meter of *terrain*. If an asset were present in the environment, some portion of its area will receive the agent that would otherwise have impacted the terrain. The total material area of an asset may be significantly different than the area that was contaminated, thus there is a significant difference between grams of agent per *material* area and grams of agent per *terrain* area. If the contamination source was imagined as a light source, the resulting *shadow* of the asset would describe the area of terrain that would have been contaminated if the asset was not present (Figure C-1). The mass of agent contaminating the asset would be the shadow area times the contamination density. The material area of the asset is constant, but the material area that could be contaminated may change significantly depending on the configuration of the assets (e.g., open or closed, for the multi-tool or a vehicle) and orientation of the asset (long face versus edge on). The same concept applies to larger scale assets such as vehicles (Figure 2).

* A 2 inch diameter flat panel presents a material area of 20.2 cm^2, when contaminated with 2 × 1 µL drops of VX (1.852 mg of VX) the resulting contamination density is calculated as 0.91 g/m^2.

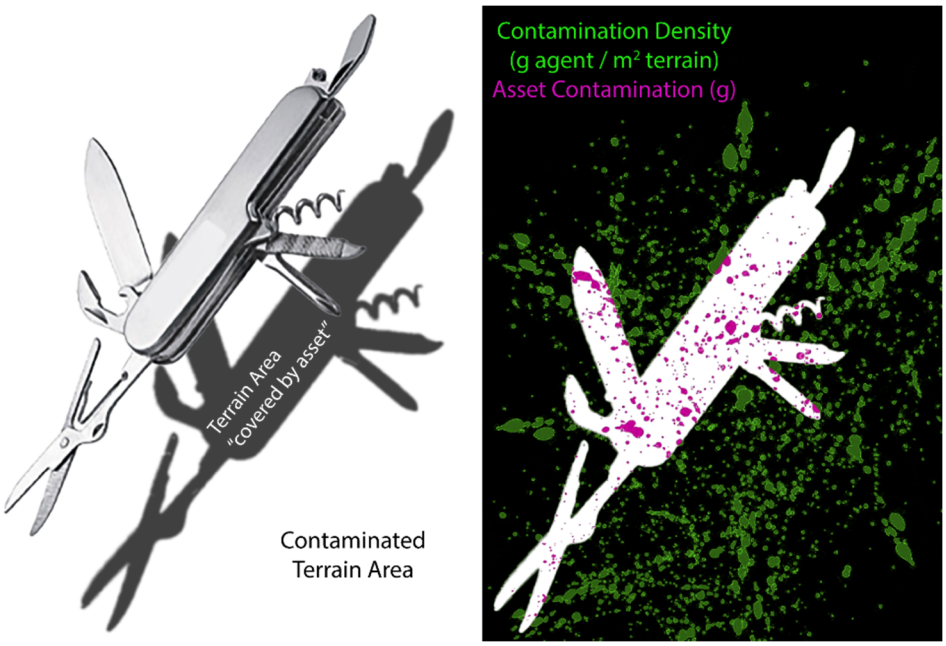


Figure C-1. Conceptual image of an item and the terrain area cast by its shadow (left). The asset would be contaminated by the (pink) drops that would have landed on the terrain had they not impacted the item.

The per drop normalization and drop scaling approach presented in Section 6 enable the use of laboratory data to calculate asset level responses based on the number of drops and the volume of each drop. This calculation approach enables the determination of asset level responses for any starting challenge (i.e., contamination density) based on the number of drops that interact with specific materials on the asset. Hazard mitigation performance was observed to be *independent of starting challenge*, but rather dependent on the count and volume of the agent drops.[1] This provides a more versatile approach for describing contamination of assets in many different situations and quantifying outputs from technology evaluations for relevancy in health-based assessments. Here, starting challenge is used to describe 'how contaminated' an asset is by identifying the mass of agent contaminating the asset, the calculations translate this mass into drop distributions to enable the scale-up calculations.

The approach of per drop normalization and scale up illustrates how the details of a contamination vignette (i.e., exactly how an asset is contaminated) plays a significant role in the potential for post-decontamination health effects. Many factors influence the number of drops that may impact the asset including the configuration of the asset (e.g., open doors on a vehicle, open knife on the multi-tool), the angle of the asset relative to the contamination source, the distance from the contamination source to the asset, and likely many other factors. Contamination can occur by many means, including, but not limited, to airborne liquid drops impinging on surfaces of materials and assets. Traversing contaminated terrain (e.g., cement road, dirt road, grassy field, etc.) could result in the transfer of contamination to the asset of interest. However, the ability to quantitatively describe contamination transfer is not currently robust and requires accounting for many other factors to consider in the contamination vignette.

The focus of this methodology assessment was to determine the accuracy of the scale-up calculations for highly controlled contamination. The following sections provide the

construct for how the direct contamination of an asset with liquid drops *could* be performed. The suggested approach is to use Monte Carlo (MC) random sampling methods, similar to those described in Section 6.2, to generate random drop volume distributions and randomly distribute the drops on the asset. The end goal is to generate a list of K drops with a volume, V_k, associated with each drop, and then identify the material-feature each drop is interacting with as described in the scale-up calculation in Section 6.1 and Table 32.

C.2 Mass Applied to an Asset

When liquid droplets are dispersed in an environment resulting in a contamination density for exposed surfaces, the agent mass associated with an object or asset in that environment can be expressed as

$$M_{contamination} = AC \qquad \text{Eq. C-1}$$

Where

$M_{contamination}$	is the total mass of agent that contaminated the asset (g)
A	is the projected area of asset that would receive contamination (m^2)
C	is the contamination density (g agent / m^2 terrain)

Determining the projected area for the asset may be non-trivial and is not addressed in detail here. Rather, initial efforts could make simplifications such as using the area for 1–3 sides of an asset (e.g., top, front edge, side edge) as an order of magnitude estimate for the area. The operational environment would likely include many permutations of how assets may be contaminated, detailed precision for the area may not be required to capture the range of possible areas to consider. Note that factors such as the distance of an asset from the contamination source can have significant influence on the 'local' contamination density that an asset would experience.

C.3 Drop Size Distributions

Contamination sources that disperse liquid drops generate a range of drop diameters, which tend to follow a log-normal distribution. The drop volumes may generally range from about 0.05 µL to 10 µL depending on many factors including agent properties and the nature of how the agent was dispersed. To generate the list of K drops and their volumes, a MC technique can be used to randomly sample drops from a specified drop distribution until the 'sampled' drop mass is equal to $M_{contamination}$. For example, in a 10 g/m^2 contamination density vignette, roughly 200,000 drops would need to be used to contaminate a generic vehicle.

It should be recognized that the drop size distribution at the point of contamination is a function of distance from the source and the environmental conditions. Large drops have ballistic trajectories and will deposit closer to the source, smaller drops may be suspended in the air (i.e., aerosols) and travel on air currents. Accounting for the drop size distribution at the asset level could be important, especially when the drop volume regression slope is significantly different than 1.0 (i.e., where efficacy varies with drop volume, see Appendix G). Without further data or operational details, point of generation drop size distributions may be an appropriate approximation for these types of calculations.

C.4 Probability of Contamination

To describe contamination of an asset, each drop on the list of *K* drops is associated with interacting with a specific material-feature (e.g., paint flat horizontal panel). RA source terms can vary by orders of magnitude as a function of material-feature type, therefore it is important to account for which specific material-features are contaminated. Given the large number of drops that may need to be managed, it is proposed that the probability of contamination is used in calculations (the likelihood of any drop to impact a given feature), which is proportional to the relative area of that feature. The relative area is defined with respect to the total area that was contaminated. It is not practical to map every detail of every asset that may be considered. Further, it is likely that more resolution/detail may not provide more actual accuracy. For example, the multi-tool used in the asset testing may be simplified to a box geometry as illustrated in Figure C-2.

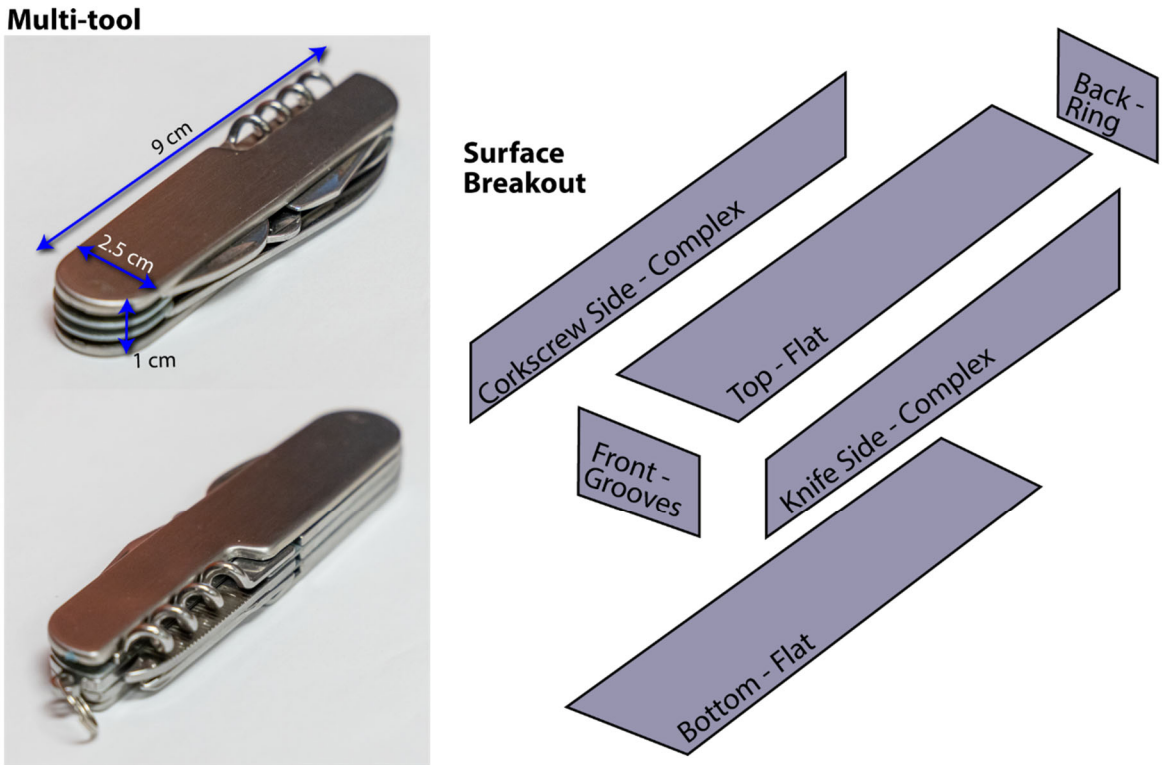


Figure C-2. Breakout illustration of multi-tool surfaces to determine probability of contamination.

Figure C-2 illustrates a simplification of the multi-tool into projected areas that are used to generate contamination probabilities, as shown in Table C-1. In this case, each face of the box is assigned a part name. Each part may be comprised of multiple components such as the corkscrew face having elements of flat surfaces, large features, and small features. Relative areas for each component were approximated for each part. For example, for the corkscrew side it was estimated that 30% of the part area can be represented using steel-flat panel results (i.e., 30% of drops interacting with this face would behave as flat panel results). The component area (cm^2) is estimated as the part area (cm^2) times the relative part area (scaled 0-1). Each component is then associated with a material of construction and the laboratory-tested source

material-feature that will be used to represent the feature. Surrogate or representative materials for many actual materials may need to be identified for the numerous materials that may be found on assets. For this asset, the material of construction is steel, and since the laboratory data in this study includes steel evaluated for multiple feature types, specific data for this component is available. In other cases, data for the material of construction may not be available and an alternative or surrogate laboratory-tested material will need to be used. The process is continued for each component, which includes assigning the percentage of drops that interact with specific material-feature combinations.

Table C-1. Asset source description and contamination probability

Part	Part Area (cm^2)	Component Description	Relative Part Area	Component Area (cm^2)	Actual Material-Feature	Source Material-Feature	Contaminated (1 or 0)	Contaminated Area (cm^2)	Contamination Probability
Top	22.5	Flat side	1	22.5	Steel-flat	Steel-flat	1	22.5	0.6618
Bottom	22.5	Flat side	1	22.5	Steel-flat	Steel-flat	0	0	0.0000
Front	2.5	Grooved surface	1	2.5	Steel-20 mil	Steel-20 mil	1	2.5	0.0735
Back	2.5	Rounded surface	0.75	1.875	Steel-flat	Steel-flat	0	0	0.0000
		Key ring	0.125	0.3125	Steel-2 mil	Steel-2 mil		0	0.0000
		Large groove	0.125	0.3125	Steel-20 mil	Steel-20 mil		0	0.0000
Knife Side	9	Small gaps	0.5	4.5	Steel-2 mil	Steel-2 mil	1	4.5	0.1324
		Large Gaps	0.5	4.5	Steel-20 mil	Steel-20 mil		4.5	0.1324
Corkscrew Side	9	Flat region	0.3	2.7	Steel-flat	Steel-flat	0	0	0.0000
		Large gaps	0.5	4.5	Steel-20 mil	Steel-20 mil		0	0.0000
		small Gaps	0.2	1.8	Steel-2 mil	Steel-2 mil		0	0.0000
Total	68	-	-	68	-	-	-	34	1.0

Once this process is completed for all parts and components, the parts that were contaminated may be specified. In this contamination vignette the top, front, and knife side of the tool were contaminated (indicated with a 1 or 0 in the contaminated column of Table C-1). The total contaminated area (34 cm^2) is the sum of the contaminated areas of each component, each of which is determined by the product of component area and the contaminated column. The contamination probability is calculated as the contaminated area for each component divided by the total contaminated area. For this particular vignette, a given drop has a 66.2% probability of interacting with the top-flat side, or a 13.2% probability of interacting with a small feature on the knife side, etc.

Random sampling methods can assign each drop *k* to a specific part-component according to the calculated contamination probabilities, resulting in an output similar to Table 32. This produces the description of contamination required to conduct the scale-up calculations as described in Section 6.1. In the context of MC methods, the generation of drop volume distributions and random assignment of drops to material-features (i.e., part-components) can be repeated many times to sample many contamination configurations (in addition to the multiple iterations for each drop distribution as conducted in this report). This process of repeated random sampling enables characterizing the range of asset-level responses that could be generated based on the random distribution of drops that will occur in operational conditions. This process is expected to provide a more complete description of the range of responses that could be

observed in DT testing or operational events where contamination processes are highly randomized – providing a better characterization of the hazard mitigation technology being evaluated.

It is recognized that the process of defining the parts and components of assets to determine contamination probability may become significantly more complicated as the asset becomes larger, such as vehicles. Further, future efforts may need to identify the level of detail required to provide acceptable accuracy since the output is a combination of the ability to accurately describe the contamination vignette and the parts, components, and contamination probability of the asset, and the laboratory source term data.

C.5 Applying Variable Contamination Capabilities

The application of these MC random sampling techniques can be used to show the influences of different contamination event factors on the potential for post-decontamination health effects. Using the tools presented here, each asset in a vignette could be individually analyzed accounting for different levels of contamination based on their proximity to the contamination source to identify. For example, it could be identified that the hazard mitigation technology could decontaminate 95% of all assets to an acceptable level, but the 5% that were the most contaminated would require additional efforts to sufficiently decontaminate. Previous approaches seem to focus on evaluating only the worst-case condition, which may represent only a fraction of contaminated assets. Focusing exclusively on worst case conditions may not provide an accurate picture of complete operational context. Technologies could provide sufficient decontamination on most assets, but not the worst contaminated. Using this type of approach could identify if this is the case.

The MC techniques presented here have a relatively small computational expense. Calculations generally take 1–10 minutes on a personal computer and are highly parallelizable. Many conditions can be analyzed using the laboratory data to represent different vignettes, which would allow for supporting Developmental Testing (DT). This approach provides a new analysis capability that enables building a better quantitative characterization of how decontamination technologies can enable the reduction of the protective posture by considering the range of conditions that may occur, rather than just compounded worst case contamination with worst case exposure that represent, for example, only 1% of cases.

References

1. Mantooth, B.A.; Pearl, T.P.; Stevenson, S.M.; Smallwood, S.Q.; Eikenberg, J.H.; Sheahy, M.L.; Gehring, D.G.; Chesebrough, M.J.; Ruth, J.L. *Normalization of Contamination Conditions for Measurement of Agent Retention and Decontamination Performance on Materials*; CCDC CBC-TR-1767; DEVCOM Chemical Biological Center: Aberdeen Proving Ground, MD, 2022. UNCLASSIFIED Report, Public Release (AD1170213).

Appendix D: Temperature and Humidity Summary Table

Table C-1 provides the temperature log summary as described in Section 2.6.

Table C-1. Temperature and humidity measurements during testing.

Report Section	Test ID	Laboratory Condition			Environmental Chamber		
		Mean T (°C)	Min/Max T (°C)	Mean RH	Mean T (°C)	Min/Max T (°C)	Mean RH
3	CAP016	21.7	20.5/22.3	55.47	21.27	19.7/21.6	63.11
	CAP017	21.98	21.5/22.2	52.77	21.66	21.5/22.6	55.18
	CAP034	20.52	20.2/20.8	46.07	21.02	20.8/21.2	45.22
4	CAP019	22.23	21.9/22.4	53.63	21.66	21.7/22.3	52.71
	CAP021	22.21	21.6/22.5	51.93	22.06	21.8/22.7	52.65
	CAP022	21.81	21.4/22.1	52.91	22.42	21.7/22.3	53.26
	CAP023-24*	22.78	21.6/24.0	48.16	22.97	22.0/24.1	48.38
	CAP026	22.2	21.0/23.3	53.11	22.47	21.6/23.6	53.2
5	CAP028	22.01	21.3/22.5	19.13	22.17	21.5/22.6	19.3
	CAP029	22.39	21.2/23.1	22.01	22.19	21.0/23.1	22.41
	CAP030	23.24	22.8/23.5	49.36	23.2	22.8/23.9	50.25
	CAP031-32*	23.16	21.8/23.6	49.19	23.28	21.9/23.7	49.6
	CAP033	22.84	22.2/23.4	31.32	22.84	22.0/23.5	31.2
7 and 8	CAP035	22.56	22.1/23.8	50.58	22.67	22.1/24.1	51.08
	CAP036	22.69	21.4/23.4	50.36	22.96	21.8/23.6	50.34
	CAP037	22.61	21.8/23.5	41.67	22.62	21.9/24.0	41.94
	CAP038	21.87	20.9/23.1	41.56	22.01	21.1/23.2	41.58
	CAP039	23.07	22.7/23.6	37.69	23.27	22.9/23.7	37.34
	CAP040	22.01	21.6/22.8	22.07	22.31	22.0/23.0	21.75
	CAP045	21.59	20.9/23.4	12.1	21.85	21.1/23.4	12.19
	CAP041	21.75	21.3/22.1	15.36	22.1	21.7/22.4	15.31
	CAP042	20.81	19.7/22.8	20.95	21.03	19.8/23.1	20.94
	CAP043	21.77	20.6/22.4	25.48	21.91	20.8/22.6	25.24
	CAP044	21.55	21.1/23.0	13.24	21.64	21.2/23.3	13.42
	CAP046	22.57	21.4/23.9	20.74	22.64	21.4/22.8	20.75
	CAP047	22.02	21.3/22.8	11.0	22.17	21.2/23.0	11.22

*Executed on the same day

Appendix E: Effects of Panel Orientation

The sensitivity analysis performed in Section 5 considered factors such as orientation and difference in application process for liquid decontaminants. Testing during method development on panel orientation are briefly summarized in Section E.1. Additionally, advanced multivariate statistical models, provided in Section E.2, were applied to the data collected in Section 5 to provide a more robust characterization of the parameters investigated.

E.1 Effect of Orientation for Flat Panel Testing

During method development flat panels were tested in horizontal and vertical orientations. Orientation can influence how both the agent and the decontaminant may flow during the treatment process. An example image of fluorescently labeled VX after a 60 min contamination duration depicts how the orientation influenced the final agent distribution before a treatment was conducted. Similar to the agent, panel orientation can influence if the liquid decontaminant resides on the material or flows off during the treatment process. The previous work demonstrated that orientation can influence the RA mass for VX.[1] A screening test was performed to evaluate how orientation may influence the RA with multiple agents and liquid treatment processes as described in Table E-1.

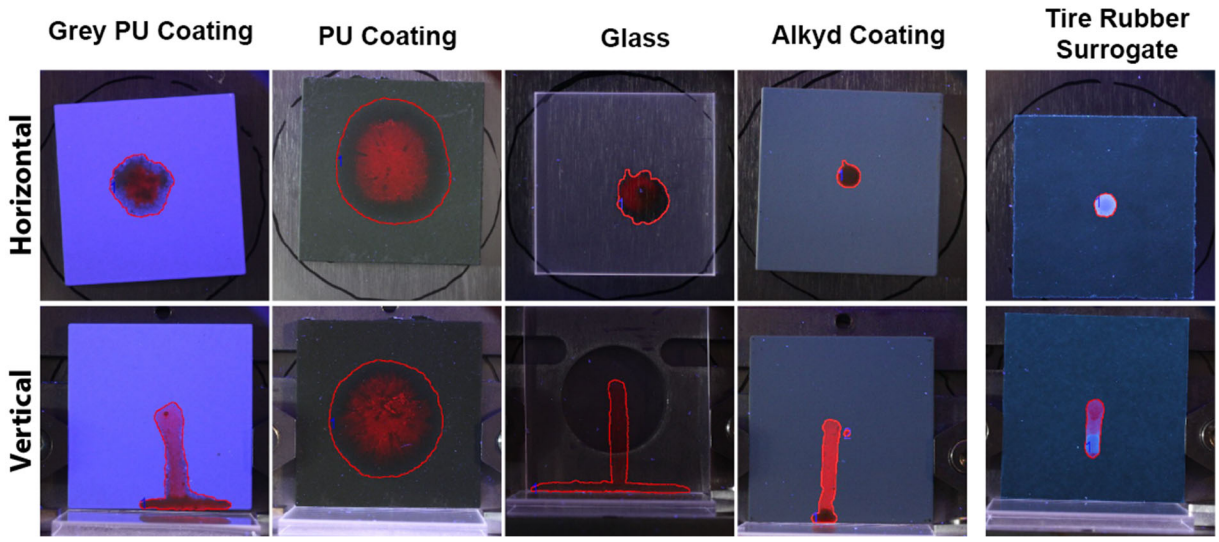


Figure E-1. Images of materials with fluorescently labeled 2 µL drops of VX on horizontal (top row) and vertical (bottom row) orientation panels after a 60 min contamination duration (reproduced from Ref[1]). Red outlines indicate where liquid agent was observed.

Table E-1. Test parameters to characterize the effect of orientation on RA.

Parameter	Conditions	Comments
Materials	PU coating, Silicone	Coating where agent tends to spread and elastomer where agent tends to remain as sessile drops
Agent	HD, GD, VX, MeS	Range of agents with different properties including surface tension, adhesion to materials, and volatility
Contamination	1 × 2 µL drop	Single drop application
Age Time	60 min	Typical contamination duration.
Panel variations	Flat Panel	Flat panels were 2 in diameter disks
Drop count ×volume (µL)	1 × 2 µL drop	Single drop application using a Gilson M10 pipette
Contamination Temperature	21 °C	Standard ambient temperature
Treatment	1) soapy water spray followed by water rinse (30 min residence) 2) bleach spray followed by water rinse (30 min residence)	Solutions were sprayed with the Preval sprayer for about 4 s.
Analysis Method	Retained Agent (RA)	Output includes RA for each panel, compare LD for horizontal versus vertical orientations.
Replication	3	Screening testing to identify order of magnitude effects used smaller replication to evaluate more conditions.

This test data was used to characterize how orientation across multiple contaminants influences the RA mass after two treatment processes. For example, is more mass retained in one orientation versus another, and do these trends vary with contaminant. The retained agent (RA) results and log difference (LD) of the horizontal orientation compared to the vertical orientation are provided in Figure E-2. For silicone, the results show that orientation did not have a statistically significant influence greater than ± 0.4 LD on RA. For the PU coating a range of responses were observed. For GD, HD and MeS on the PU coating, more RA was often observed for the horizontal orientation. The low replication of this screening study resulted in statistically insignificant LD (i.e., the 95% CI includes zero) for bleach in these cases. For VX on the PU coating, the opposite trend was observed, slightly more RA was observed for the vertical orientation. The work from previous studies showed that VX tends to adhere to materials and leave a liquid trail as the drop flows down the panel. This trail seems to result in higher RA mass and lower apparent decontamination efficacy.

The grey region in the LD plots corresponds to the ± 0.4 LD range indicated as being a significant enough difference to consider including in testing, otherwise one panel orientation can be tested and used to represent horizontal and vertical orientations in scale-up calculations. Note that the ± 0.4 LD range was as an initial estimate for identifying a significant difference. Testing panel orientation for all agent material combinations requires non-trivial resources. Future studies should consider analyzing scaled-up asset level results to identify if the ± 0.4 LD difference at the panel level is too low of a threshold. The ± 0.4 LD range may be small enough that when scaled up, the asset-level results may not show a significant difference if both orientations were used in the calculation compared to testing only one orientation (i.e., the return on investment for expending the resources to test both orientations may not be significant).

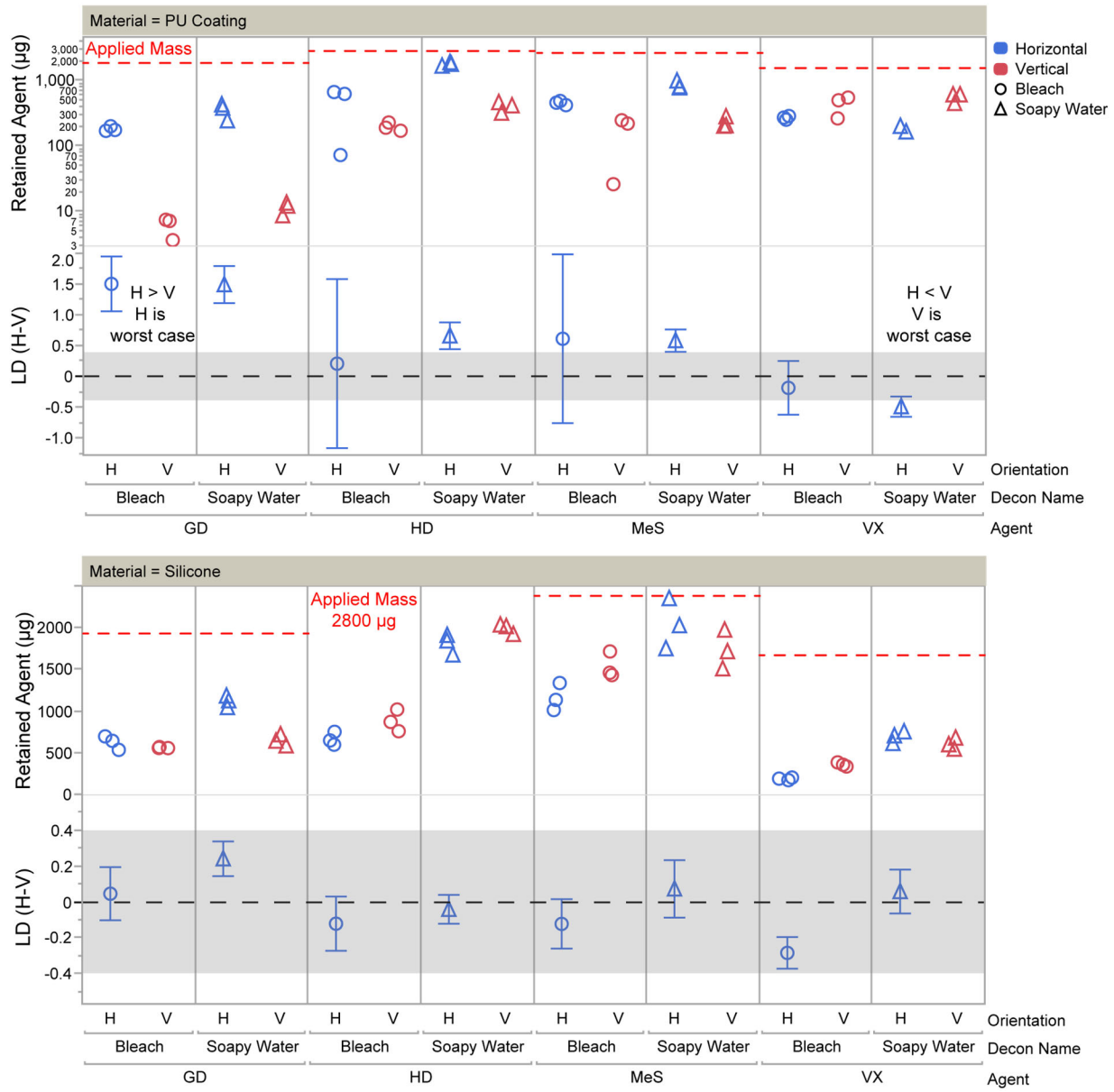


Figure E-2. RA results for liquid treatments in different orientations, bottom parts of each graph show the LD comparing horizontal (H) to vertical (V) RA, error bars represent the 95% CI on the LD. The grey shading indicates the ± 0.4 LD range.

E.2 Statistical Modeling of Sensitivity Data

The sensitivity analysis performed in Section 5 generated results as a function of orientation, spray angle, agent, and material. In Section 5, the data were analyzed pairwise to determine if there was statistical significance. The use of statistical models can utilize all of the laboratory data to construct stronger data comparisons. Further, the implementation of Monte Carlo techniques in the scale-up calculations require laboratory-based inputs. As described in Section 6, the Monte Carlo inputs are both the μ and σ of the data. It is possible to predict both μ and σ for an RA output using a statistical model as demonstrated here.

The sensitivity data was executed as a full factorial test; all test conditions were collected at the indicated replication levels. Statistical models can be used to quantify the effects of the categorical variables investigated. The statistical models provide more confident comparisons because of pooled sampling across all conditions, rather than the comparison of four replicates for two conditions.

A Loglinear Variance analysis was conducted using SAS JMP PRO V 17.0 which was used to predict the μ and σ of the log-transformed RA data. This analysis is provided as a *demonstration* to visualize the influence of the parameters investigated and serve as a proof-of-concept illustration for producing statistical model outputs for μ and σ. This is a demonstration of the capability, future efforts would need to quantify the accuracy of these predictions, as a result, only an overview of the statistical details are provided.

The starting statistical model for fixed effects treated all factors as categorical and included the main effects and all two-way interactions. The starting statistical model was reduced using the BiC criterion with a backwards step regression. The process of model reduction removed non influential effects from the model leaving just the factors with significant effects which are shown in Table E-1. The resulting model described 67% of the variance observed in the data (i.e., the adjusted $R^2 = 0.669$).

Table E-1. Statistical Model Parameters for Sensitivity Analysis

Type	Parameters
Main Effects	Agent, Shim Thickness, Bottom Material, Orientation
Cross-Effects	Agent * Orientation Shim Thickness * Bottom Material Shim Thickness * Orientation Bottom Material * Orientation

After fitting the fixed effects model the JMP LogLinear Variance platform was used to add the variance (random) effects to the fixed effects model so that the mean and standard deviation of the log-transformed RA data could be modeled simultaneously. The output of the LogLinear Variance statistical model is provided as a profiler showing how the predicted mean (top row) and predicted standard deviation (bottom row) of the $\log_{10}$(RA/drop) change across the variables (Figure E-). The vertical dashed red lines indicate the 'selected' parameter. For example, Figure E-A indicates that the predicted mean $\log_{10}$(RA) for VX on steel-flat panel in a horizontal orientation with 30° spray angle is $\mu = -0.47$ $\log_{10}$ μg. The bottom row provides the predicted standard deviation, $\sigma = 1.28$. These plots show how different parameters influence μ and σ. Comparing the agent column, the predicted mean response (top row *A*) for HD and VX

seem to overlap – similar RA mass is retained for either agent. Figure E-A2 shows how predicted σ varies across agents for this condition, VX has a larger predicted standard deviation. The effect of shim thickness (C) shows increasing retention for smaller shims, and that 2 mil shims have a much smaller predicted σ. Figure E-D shows that the difference between horizontal and vertical orientation is not statistically significant (error bars overlap).

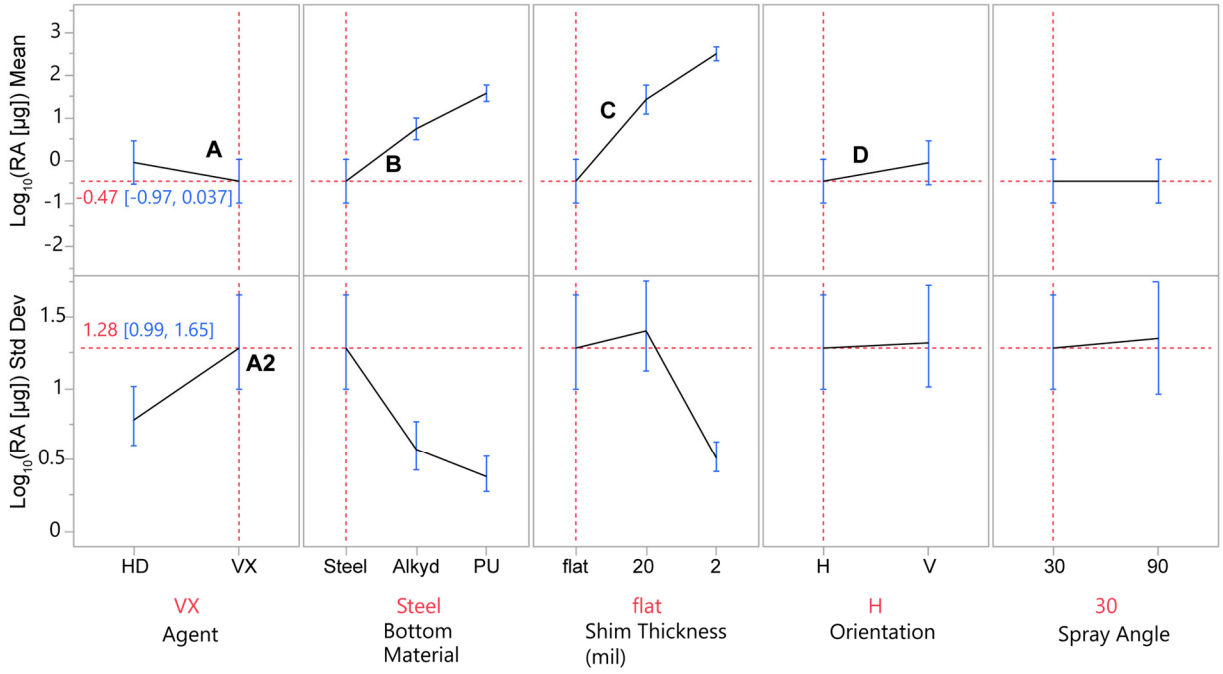


Figure E-3. Statistical model profiler for VX on steel, top row depicts the predicted mean $\log_{10}$(RA), the bottom row depicts the predicted standard deviation of the $\log_{10}$(RA).

A profiler view for VX on PU flat panel is illustrated in Figure E-4. The trends are significantly different compared to the steel results. Similar trends can be observed, smaller shims produce larger predicted μ and smaller predicted σ. The predicted μ response for orientation overlaps between horizontal and vertical, indicating there are no significant differences for orientation with VX on PU-flat panels.

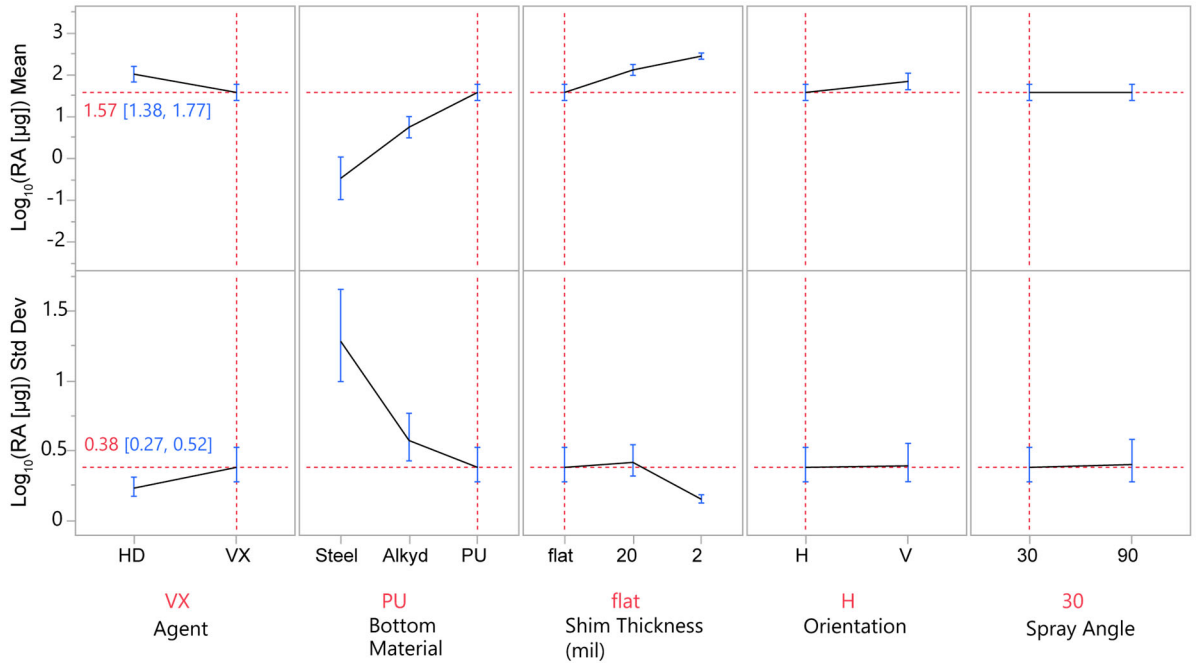


Figure E-4. Statistical model profiler for VX on PU coating, top row depicts the predicted mean $\log_{10}$(RA), the bottom row depicts the predicted standard deviation of the $\log_{10}$(RA).

These examples show that a statistical model can be developed for panel data across categorical variables. Further, Design of Experiment techniques can be used on these types of data to provide efficient test plans that would provide the predicted μ and predicted σ inputs to enable the Monte Carlo scale-up calculations without conducting full factorial testing.

References

1. Mantooth, B.A.; Willis, M.P.; Myers, J.P.; Lalain, T.A. *Development and Demonstration of the Complex Test Panel Methodology*; ECBC-TR-994; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2012. UNCLASSIFIED Report, Distribution C (ADB404306).

Appendix F: Monte Carlo Techniques

F.1 MATLAB code For Fixed Volume Asset Contamination

The introduction of Monte Carlo (MC) calculations is new to hazard mitigation evaluations. MC techniques typically require some amount of code or script to implement. The function MonteCarlo_FixedV.m that runs the calculations is provided in Figure F-1. This code was written in MATLAB version 2022a.

```
% This will take the input parameters and perform a MonteCarlo simulation
% this is in alignment with the shim panel Methodology Assessment where all drop
% volumes on the asset are fixed at either of 1 or 0.2 µL
%
% tableParam - is a matlab table structure containing drop counts to
% simulate and the source term mean and standard deviation values for fit
% and direction calculations
%
% n_sim - how many iterations to perform
%
% Type - 'Fit','IndirectV02','IndirectV1' - determines which statistical values are
used in the calculation
%
% Agent_prop - is a 1x2 matrix (1) = agent purity [0-1 scale],
%       (2) is the density in [g/ml]
%
% OUTPUT: sim_out - is a 5xn_sim array all elements are in mass µg
%
%   row 1=flat, 2=20 mil, 3=2 mil, 4 = total mass (M_Asset),
%   5 = Applied Mass (DCS)
%
%
% Author: Brent Mantooth
% 30 Nov, 2022

function [sim_out] = MonteCarlo_FixedV_v2(tableParam, n_sim, Type, Agent_prop)

%determine which statistical values to use
if(strcmp(Type,'Fit')) %pull from the regression
  src_mean = tableParam.FitMean(2:4); %flat
  src_std = tableParam.FitStd(2:4);
elseif(strcmp(Type,'IndirectV02')) %pull indirect V=0.2µL
  src_mean = tableParam.mean(2:4);
  src_std = tableParam.std(2:4);
elseif(strcmp(Type,'IndirectV1')) %pull indirect V=1µL
  src_mean = tableParam.imean(2:4);
  src_std = tableParam.istd(2:4);
else
  msgbox('Fit Type Not Found');
end

%get simulation conditions - this assumes FIXED drop volumes
DropV_mL = tableParam.DropV(1)/1000; %drop volume [mL]
DropV_uL = tableParam.DropV(1); %store a local copy of drop volume [µL]
```

```
%This is the mass of a drop in µg - used to upper bound limit random prediction
DropVMass = DropV_mL*Agent_prop(2) *1E6;
DropVMass_log10 = log10(DropVMass); %convert to log10 units

%Store local copy of DCS mean and stdev
DCS_mean = tableParam.FitMean(1);
DCS_std = tableParam.FitStd(1);
nDrops = tableParam.nDrops(1);
%create data to store simulation output
%sim_out stores the mass assocated with each feature, M_asset, and DCS
%row 1=flat, 2=20 mil, 3=2 mil, 4 = total mass, 5 = DCS
sim_out = zeros([n_sim,5]);

%get how many drops for each feature source term
%nDrop will be a 1x3 array
%row 1=DCS, 2=flat, 3=20 mil, 4=2 mil,
nDrop = tableParam.nDrops(2:4);

%Run MC Simulations
for i = 1:n_sim

  % for each Feature condition
  FeatureSum=zeros([1 3]);
  for j = 1:3
    %get how many drops to simulate on this material (from asset
    %variable)
    %return ndrop matrix of normally distributed random numbers
    rndSamp = randn(nDrop(j),1);

    %The randn function returns a sample of random numbers from a normal
    % distribution with mean 0 and variance 1.
    % we transform this to our distribution using the std and mean values
    %Note this is calculated in the log10 transform space

    %array calcultion for each drop using each feature j
    drop_data = src_std(j) .* rndSamp + src_mean(j);

    % enforce upper limit mass to total drop volume for any given drop,
    % large variance can allow mass values greater than the applied drop
    drop_data = min(drop_data,DropVMass_log10);

    % Inverse Transform to RA units in µg for each feature (j), note
    % the inverse transform MUST be done before the sum is taken
    FeatureSum(1,j) = sum(10.^drop_data);
  end %j

  %variable slicing to facilitate parallel execution with parfor (parallel
  %toolbox)
  sim_out(i,1) = FeatureSum(1,1);
  sim_out(i,2) = FeatureSum(1,2);
  sim_out(i,3) = FeatureSum(1,3);

  %accumulate total mass
  sim_out(i,4) = sum(FeatureSum(1,1:3));
```

```
  %Calculate randomized DCS Mass
  %return ndrop matrix of normally distributed random numbers - index 1 = DCS
  rndSamp = randn(nDrops,1);
  %array calcultion for each drop using each shim condition j
  drop_data = DCS_std .* rndSamp + DCS_mean;

  %Inverse Transform DCS results from log units back to normal units
  % RA total transformed back to RA units in µg - for DCS
  sim_out(i,5) = sum(10.^drop_data);

end % i - MC simulation

end %function
```

Figure F-1. MATLAB code that implements the MC calculations for fixed drop volumes.

F.2 Random Sampling Data Example

The implementation for MC methods generates a large sample of results with the same µ and σ as the laboratory data. An example of the panel data is provided in Figure F-2. In this case, five laboratory RA samples are used to calculate a $\log_{10}$ transformed µ of 2.83 and σ of 0.077. MC uses random sampling from a standard normal distribution (a normal distribution with a µ of 0 and σ of 1). The Matlab function *randn* is used to produce a random value drawn from the standard normal distribution. This value is used in the inverse transform as

$$RA = antilog(randn(\,) * \sigma + \mu) \qquad \text{Eq. F-1}$$

When many data points are displayed and overlap it is challenging to visualize data distributions. The data display in the left of Figure F-2 uses a visualization type known as a swarm plot. The MATLAB function *swarmchart* is used to display MC results where thousands of points may overlap. In this case, the data are jittered (i.e., offset) in the x-axis based on the kernel density estimate of y, similar to a histogram where more jitter (i.e., width) in x indicates more occurrences at the corresponding y value. This display does not make any assumptions about the data distribution (i.e., is nonparametric) and can reveal multi-modal conditions in the output. The progression of data from left to right in Figure F-2 shows how five replicates of laboratory data are used to calculate a µ and σ, then through randomly sampling with increasing iterations (n) build a distribution with the same µ and σ. Using this MC approach, many random samples produce representative distributions. The output for MC distributions with random sampling of different numbers of simulations (n) are shown in Figure F-2. This example illustrates random sampling for one agent-material-feature-treatment source term. To simulate M_{Asset} this process is repeated for each drop k, of agent drop volume V_k, using the appropriate material-feature source term μ_k and σ_k as inputs.

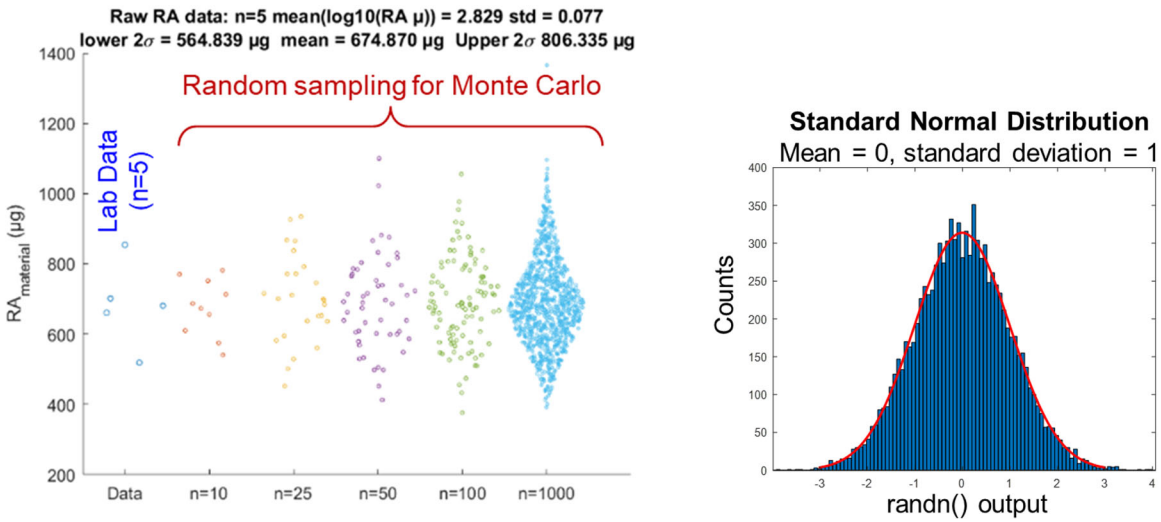


Figure F-2. Example of laboratory measured data to Monte Carlo output using a swarm plot (left). The graph on the right depicts the histogram of values produced by calling the randn() function many times.

This random sampling approach assumes that the laboratory data used provides a sufficiently accurate representation of μ and σ. The ability to produce an accurate σ value may require significant sampling (e.g., more than 15 samples). However, it is typically not practical to test all of the desired conditions with higher levels of sampling. The σ value will influence the dispersion of the asset level outputs which will influence analysis factors such as the upper bound tolerance interval. The influence of the σ value for one material-feature-treatment on an asset level output depends on the magnitude (i.e., μ value), and the abundance of contamination associated with the material-feature-treatment on the asset. For this initial implementation of calculations, the current laboratory sampling is assumed to provide sufficiently accurate μ and σ values.

Appendix G: Interpretation of Regression Slope and Accuracy of Indirect Calculation Assumptions

G.1 Interpretation of Regression Slope

The per drop regression (Eq. 5) is an empirical relationship relating the retained agent (RA) and the applied agent drop volume (V). When the regression is transformed to mass units (Eq. 6), the slope, m, describes the power law relationship as V^m (i.e., there is a nonlinear, power law relationship between agent drop volume and the RA). Previous work showed that for sessile drops, the liquid-material wetted area, which informs relative sorption and evaporation rates during contamination, determines RA and treatment process efficacy (Figure G-1).[1] Conversely, for materials where agent spreads significantly, the RA trends correlate with total mass of agent applied to the surface.[1] In both cases, the wetted area and the mass applied are correlated with V. A slope of 1.0 implies a direct (i.e., linear) relationship between the applied drop volume and the measured retained agent (RA) after a treatment process, this is always observed for dose confirmation samples (DCS). If the RA response is correlated specifically with liquid-material wetted area, the expected slope is 2/3.[1] The regression empirically describes the trends for both wetting/spreading and sessile drop cases with observed slopes that span 0.14–3.14.

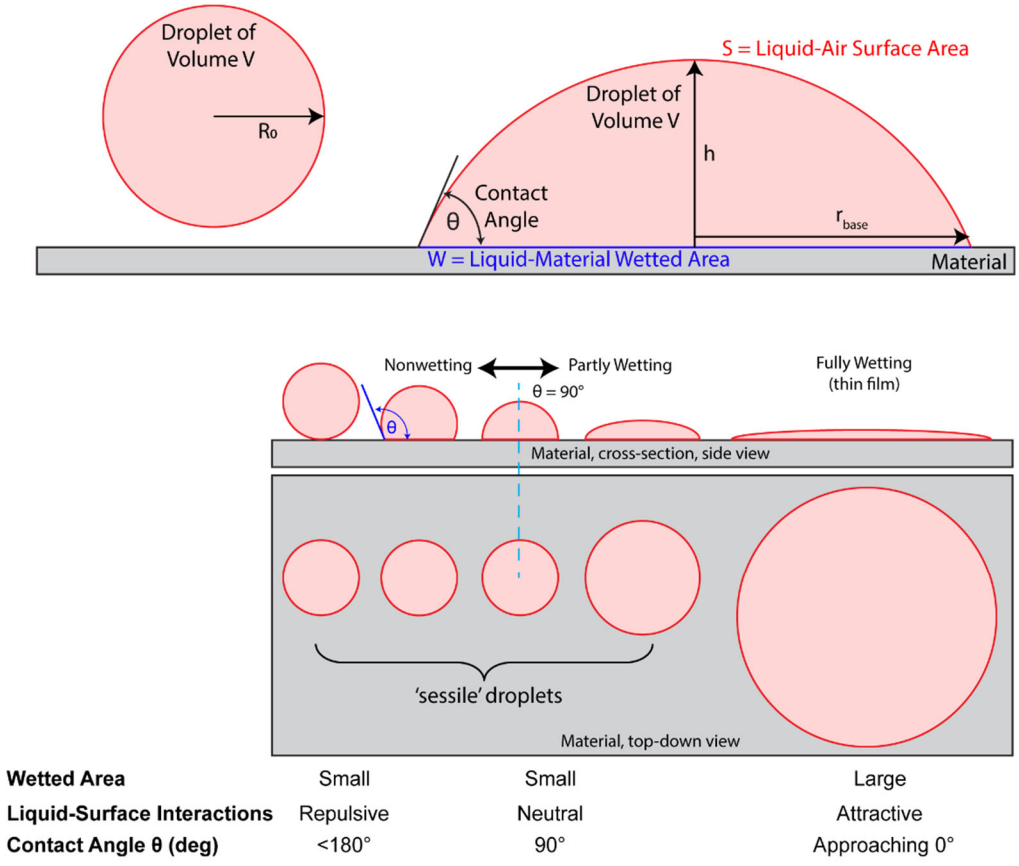


Figure G-1. Illustration of how contact angle can influence the appearance of a drop and the corresponding liquid-material and liquid-air interfacial areas.

For drops on a flat surface, the spherical cap assumption can be used to describe liquid-material interfacial areas (for drop volumes up to 16 µL for HD on glass, based on the Bond number[2]). However, the geometry of the gap for shim panels may result in capillary action that draws the agent into the feature, and this may vary with drop volume (Figure A-13). The wetted area may be influenced by the shim thickness (Figure G-2) and the drop may no longer exhibit a spherical cap geometry. These confined geometries likely alter the effects of evaporation, and trends with liquid-material wetted area. Additionally, and likely more significantly, the confined geometry limits the accessibility of the decontaminant to the agent.

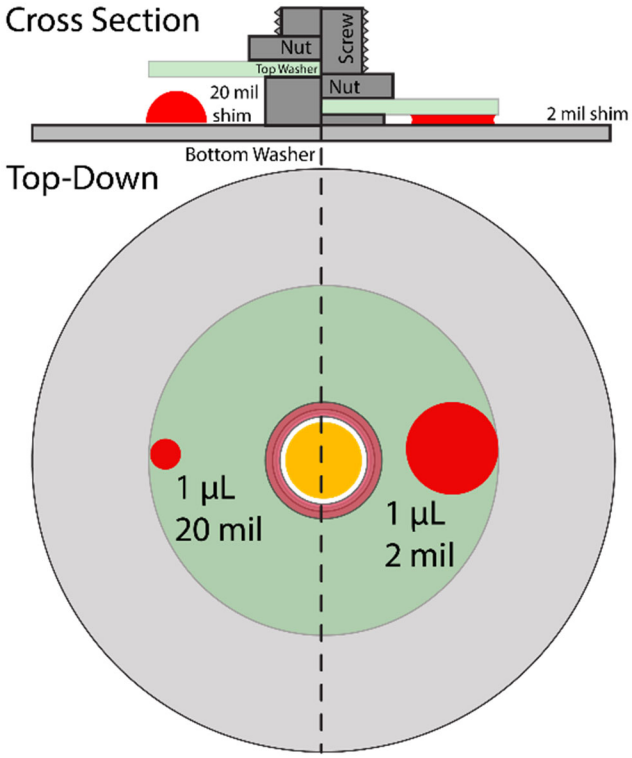


Figure G-2. Illustration of an agent drop in a 20 mil (left) or 2 mil (right) shim. Not drawn to scale.

Because the materials of interest are opaque, it is not possible to directly characterize the liquid wetted area in the material gap. In order to visualize the type of liquid interactions that occurs in the shim panel gap, a glass microscope slide was used as the top material so that an optical microscope could capture spreading behavior on relevant bottom materials.[3] In this case methyl salicylate was used as the contaminant, to which a small quantity of dye was added (Fluorosol Red 7348 [Koch Color; Bennett, CO]). For this visualization a 1 µL drop was applied to the PU coating and imaged once every second for 100 s (Figure G-3), using a Leica DVM6 optical microscope. For the 2 mil shim, the drop appears to contact the bottom and top material. Over time, a region around the drop becomes darker which is the result of hemiwicking,[4] where the liquid spreads along the surface roughness of the PU coating. For the 20 mil shim, the gap seems large enough that the drop did not touch the top glass material (e.g., Figure G-2), and rapidly spread on the PU material. In both cases, placing a drop at the material gap of the shim panel produced some quantity of agent inside and outside of the gap.

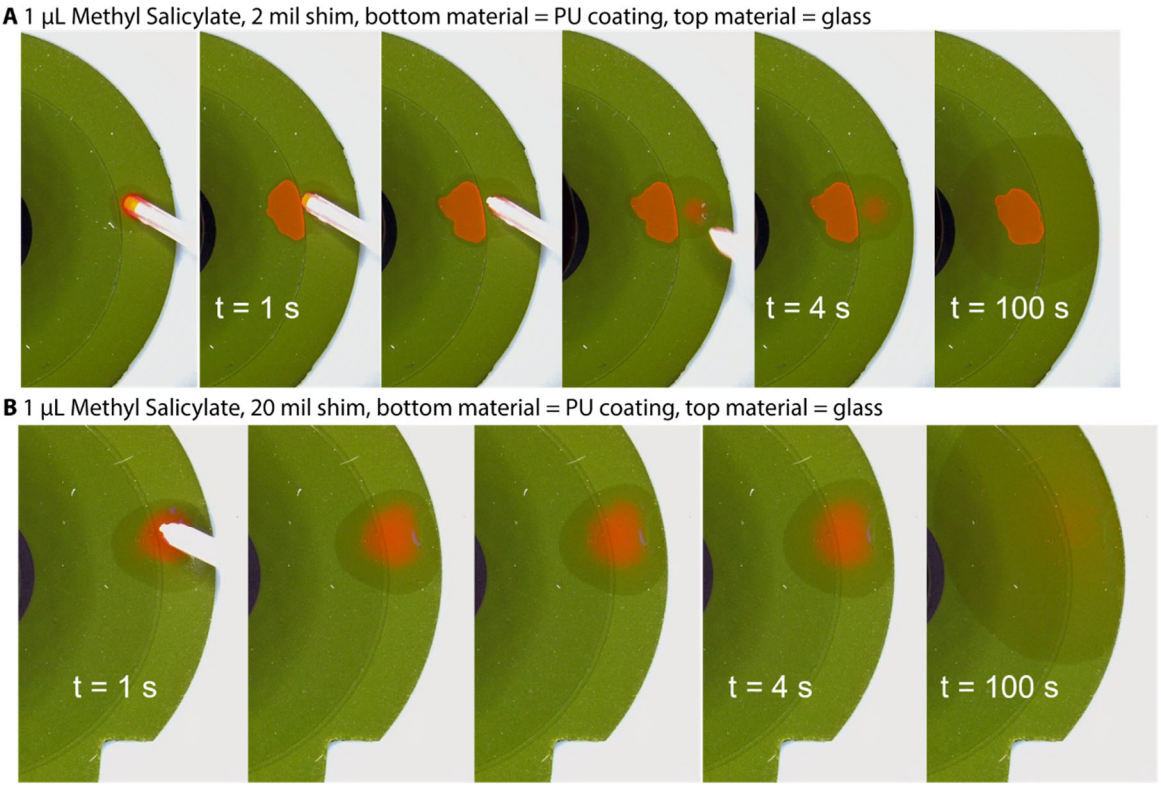


Figure G-3. Example visualization of drop distribution in a 2 mil shim (top), and 20 mil shim (bottom) from contamination (left) until 100 s later (right).

G.2 Drop Volume Trends

The *empirical* characterization of drop volume trends is a new capability that relates the applied agent drop volume to the measured RA/drop response. As discussed in Section G.1, there were hypotheses for why drop volume was influential toward the results for flat panel results relating either to the mass applied (regression slope equals 1) or relative surface area effects. The methodology assessment included liquid and vaporous treatments and introduced entrained agent effects with the shim panels. The trends associated with the regression slope across material-feature combinations are investigated.

Figure G-4 shows the slopes calculated for the liquid and vaporous treatments for all of the material-feature pairs, based on the regressions shown in Figure G-6. As the slope diverges from 1.0, the effect of drop volume on efficacy becomes larger. The flat panel results show that for all materials the vaporous treatment resulted in slopes less than one (larger drops have better efficacy), whereas the liquid treatments had slopes much closer to one. In most cases the shim panel results produce slopes greater than one (larger drops have worse efficacy). This could indicate that larger liquid drops flow deeper into the shim gap and are harder to access and remove, however, the current data cannot confirm this hypothesis.

As a general trend the alkyd and PU materials are thin coatings, the steel is an impermeable metal, and the silicone is a thick elastomer. The characteristic of how agent becomes distributed in the material likely influences the observed slope values. For the coatings and steel, the shim panel results produce slopes greater than one, however, the silicone produces a slightly different trend, possibly due to the sorption into the thickness of the material.

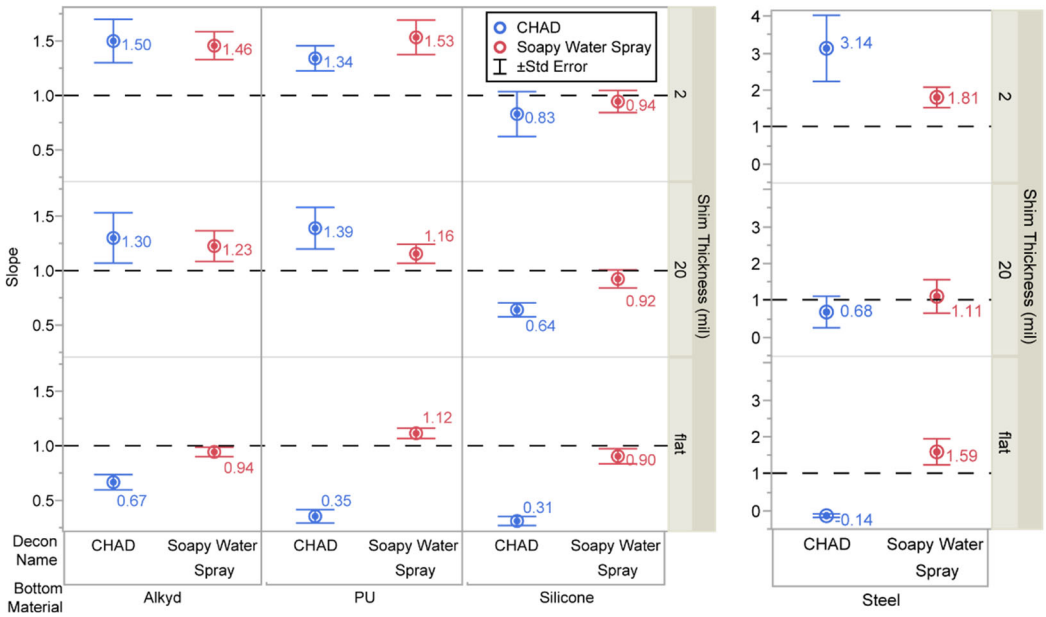


Figure G-4. The calculated slopes for each of the material-feature-treatment conditions tested in Section 7. The numerical values on the graph provide the slope value. The dashed line for a slope of 1 is provided as a reference for the assumption used by indirect fitting.

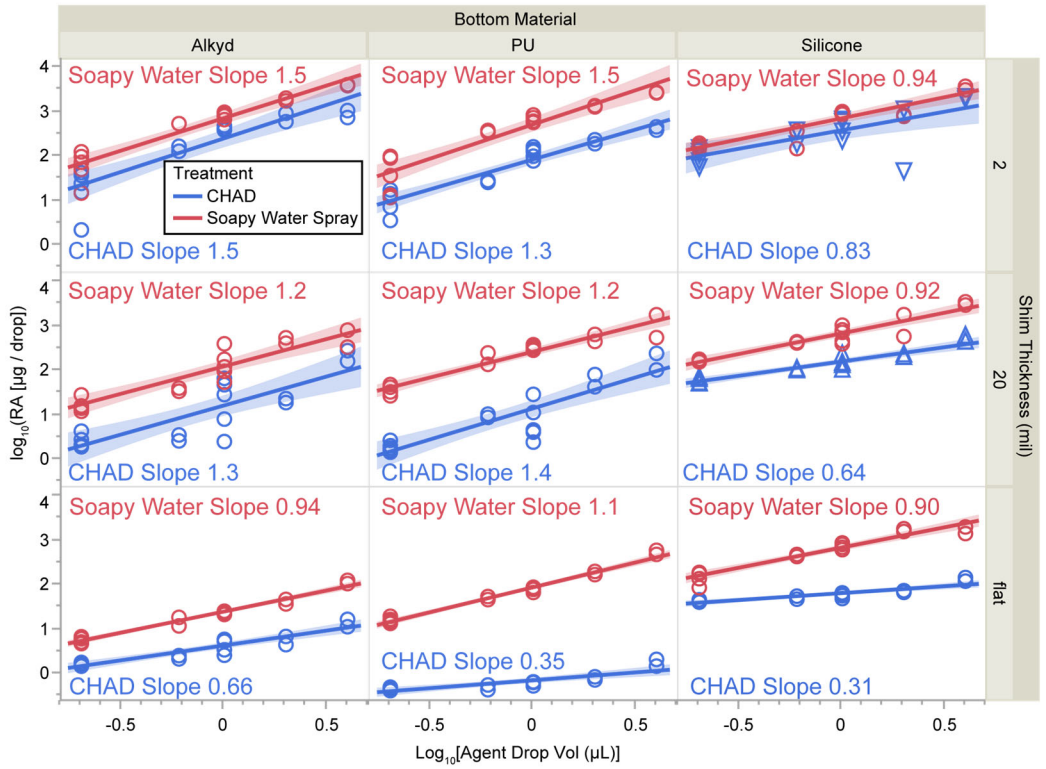


Figure G-5. The regression RA data for Alkyd coating, PU coating, and silicone across the features tested for CHAD and soapy water spray. The shaded region illustrates the CI on the fit.

LD calculations were performed for each material-feature-treatment as described in Section 7.2.2. Figure G-6 shows how the LD varies with agent drop volume using the fit technique. The flat panel response shows lower efficacy for smaller drops and larger LD for larger drop volumes (because the regression slope was less than 1.0). The shim panel results tend to show the opposite trend with higher LD for small drop volumes and lower LD for larger drop volumes. Silicone did diverge from this trend for CHAD, as expected based on the lower slope values. Figure G-7 shows similar plots for the soapy water spray treatment. The slope values near 1.0 result in more consistent LD across drop volumes for the flat panel conditions. The shim panel results had similar slope values to CHAD producing decreasing efficacy with larger agent drop volumes.

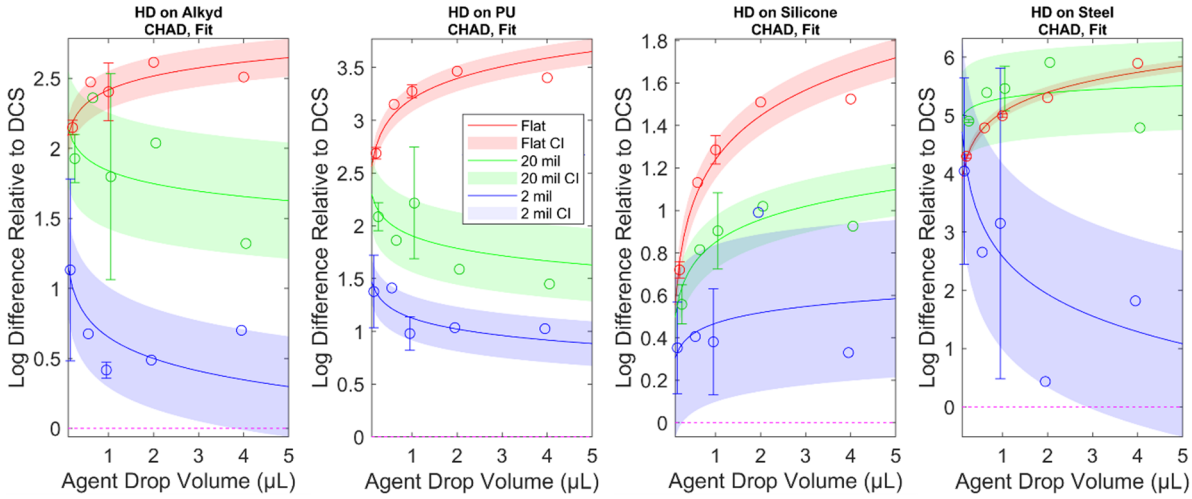


Figure G-6. LD relative to DCS for the tested materials for HD using CHAD. The circles represented LD calculated from the laboratory data.

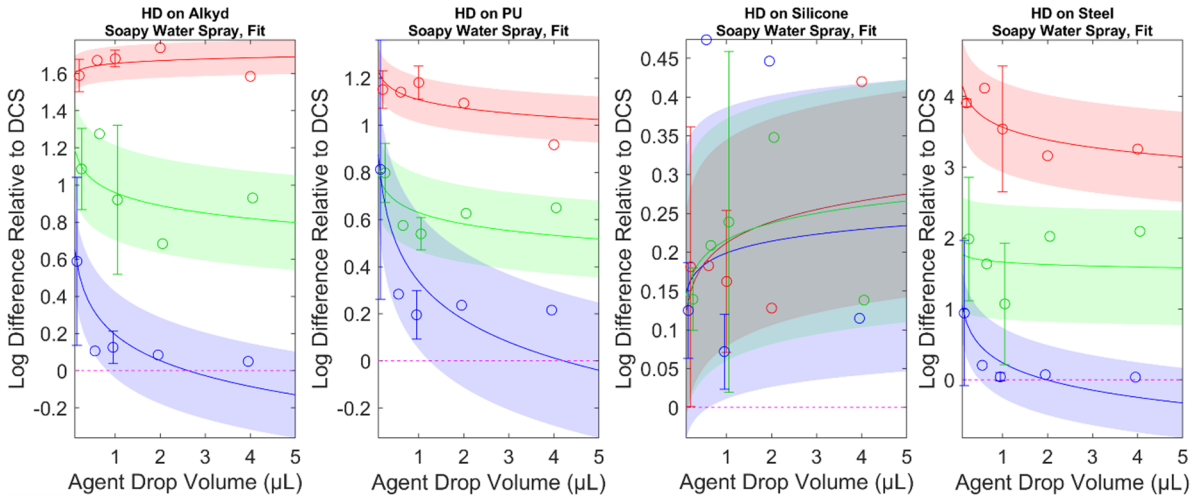


Figure G-7. Log difference relative to DCS for the tested materials for HD using soapy water spray. The circles represented LD calculated from the laboratory data.

G.3 Comparison of Source Term Outputs Across Calculation Techniques

The asset scale-up calculation utilized only the steel source terms. Section 11.1 illustrated the different values produced from the fit technique and indirect technique using different drop volumes. Here the source term outputs for other materials are provided to illustrate how the µ and σ terms compare. The graphs display the results after antilog transformation (µ* ± σ*, using Eq. B-3 and Eq. B-4).The results for alkyd coating, PU coating, and silicone treated with CHAD are provided in Figure G-8–Figure G-10. The results for alkyd coating, PU coating, and silicone treated with soapy water spray are provided in Figure G-11–Figure G-13.Recall that the direct method provides the same results as the indirect method when the indirect method uses the drop volume indicated for the data displayed.

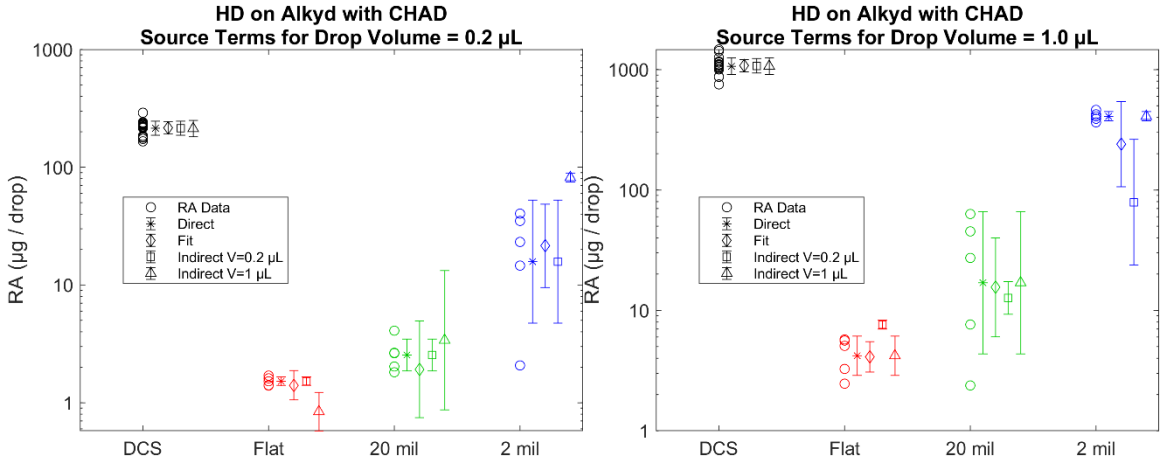


Figure G-8. Comparison of source term outputs (µ* ± σ*) for HD on Alkyd coating with CHAD for 0.2 and 1 µL drops. Open circles are the individual RA data for the indicated agent drop volume.

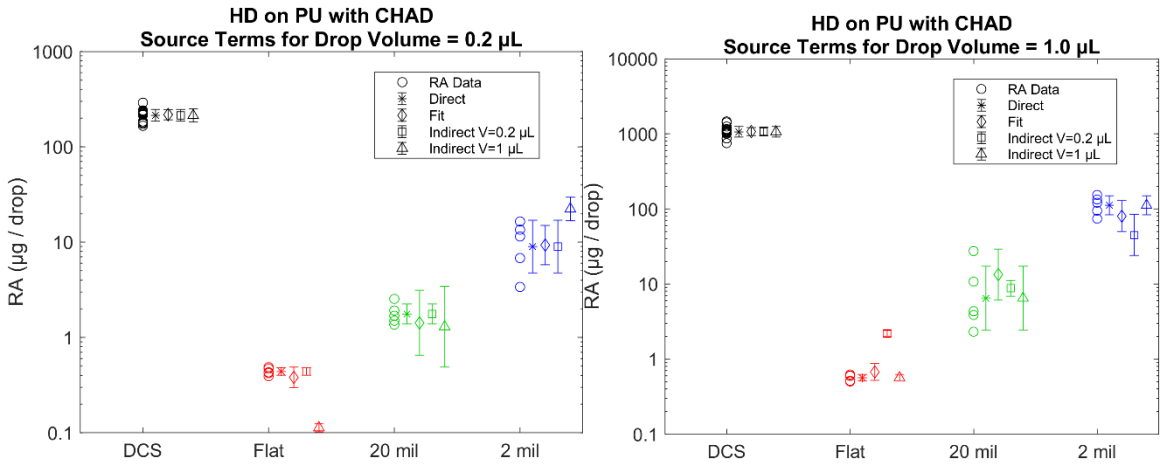


Figure G-9. Comparison of source term outputs for HD on PU coating with CHAD for 0.2 and 1 µL drops.

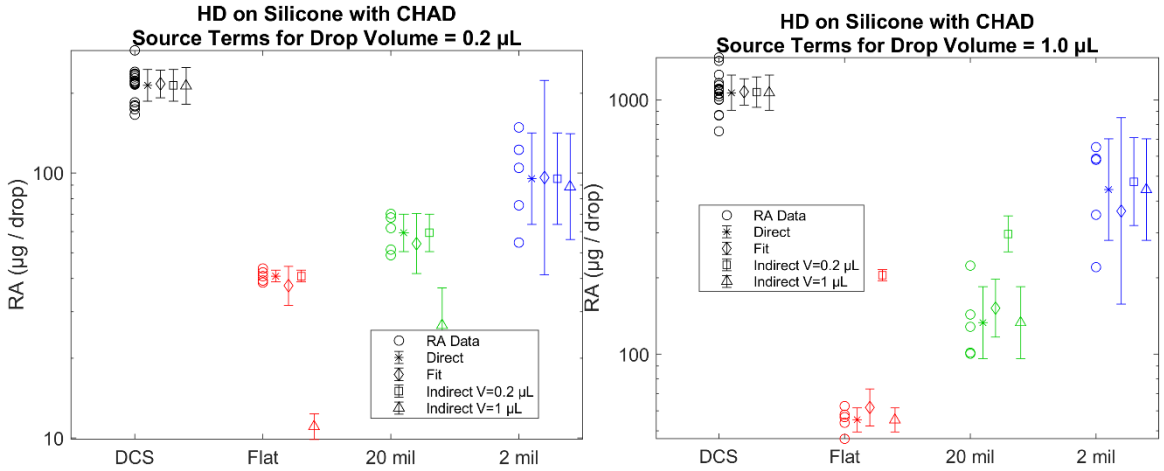


Figure G-10. Comparison of source term outputs for HD on silicone with CHAD for 0.2 and 1 µL drops.

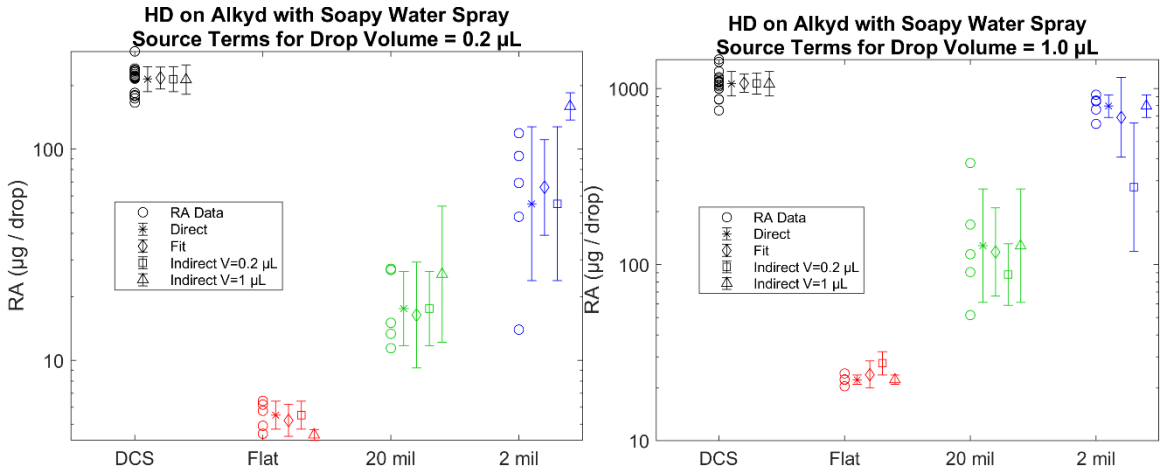


Figure G-11. Comparison of source term outputs (μ* ± σ*) for HD on Alkyd coating with soapy water spray for 0.2 and 1 µL drops.

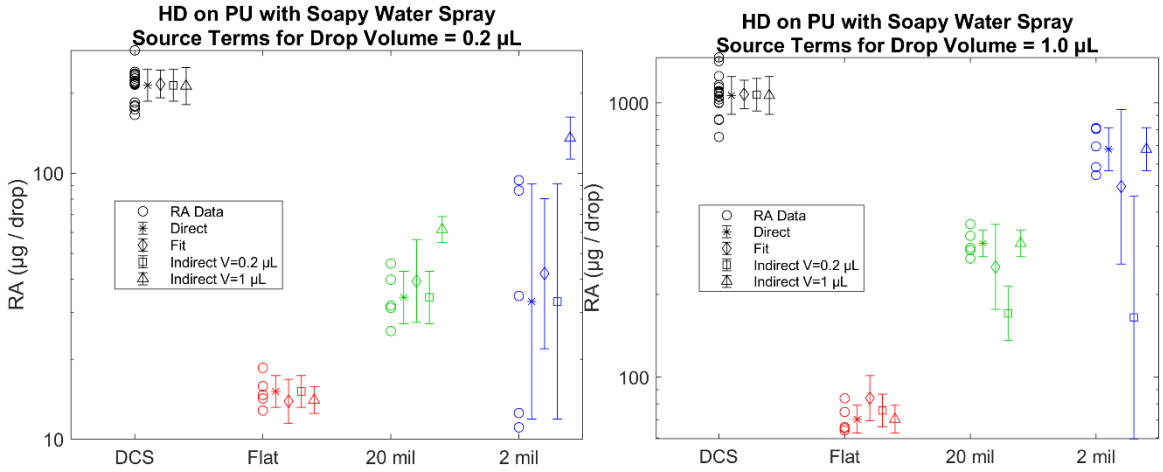


Figure G-12. Comparison of source term outputs for HD on PU coating with soapy water spray for 0.2 and 1 µL drops.

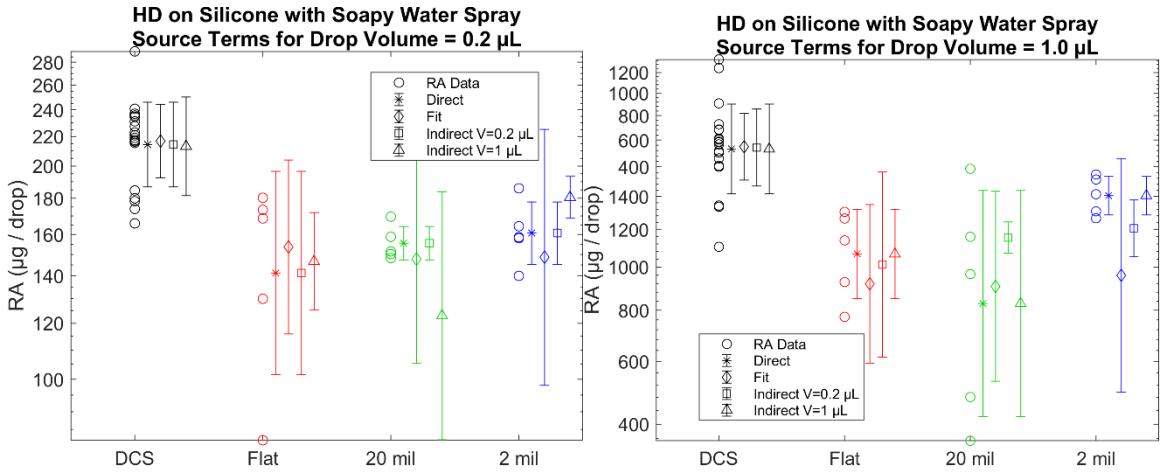


Figure G-13. Comparison of source term outputs for HD on silicone with soapy water spray for 0.2 and 1 µL drops.

Overall, these results illustrate that the fit technique provides a consistent representation of the test data across agent drop volumes. The indirect technique may produce deviation from the data when the output is extrapolated. The deviation of the indirect method could influence the accuracy of scale-up calculations similar to that demonstrated in Section 11.3.

References

1. Mantooth, B.A.; Pearl, T.P.; Stevenson, S.M.; Smallwood, S.Q.; Eikenberg, J.H.; Sheahy, M.L.; Gehring, D.G.; Chesebrough, M.J.; Ruth, J.L. *Normalization of Contamination Conditions for Measurement of Agent Retention and Decontamination Performance on Materials*; CCDC CBC-TR-1767; DEVCOM Chemical Biological Center: Aberdeen Proving Ground, MD, 2022. UNCLASSIFIED Report, Public Release (AD1170213).
2. Willis, M.P.; Mantooth, B.A.; Lalain, T.A. Novel Methodology for the Estimation of Chemical Warfare Agent Mass Transport Dynamics, Part I: Evaporation. *J. Phys. Chem. C* **2012,** 116 (1), 538-545.
3. Varady, M.J.; Mantooth, B.A.; Hawbaker, N.A.; Hinkle, A.R.; Hulet, M.S.; Ruth, J.; Sweat, M.L. *Nanofluids for Displacement of Entrained Contaminants from Restricted Geometries*; DEVCOM CBC-TR-1830; U.S. Army Combat Capabilities Development Command Chemical Biological Center: Aberdeen Proving Ground, MD, 2023. Public Release Report (AD1198339).
4. Varady, M.J.; Mantooth, B.A. Unsaturated Hemiwicking Dynamics on Surfaces with Irregular Roughness. *J. Colloid Interface Sci.* **2021,** 604, 104-112.

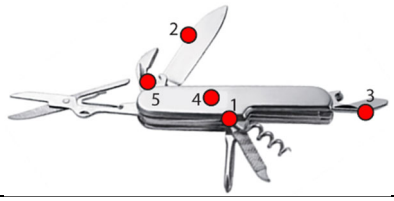
Appendix H: Asset Scale-up Calculations

The following content is the application of the asset calculation to represent the tested assets. The process is performed for each of the eight asset-agent drop volume-treatment conditions that were conducted. The summary of the accuracy of these fits was provided in Section 11.3.

H.1 Multi-tool Asset for 0.2 µL Drops and CHAD Treatment

The same sequence of analysis is conducted for each of the asset-drop volume-treatment combinations. The list of material-features and drop volumes used for the calculation is provided in Table H-1, based on the detailed description in Table 71. The contamination pattern uses five drops to deliver a total of 1 µL of agent to the asset.

Table H-1. Description of drop count and drop volumes used to represent the contamination of the multi-tool asset with 0.2 µL drops

Treatment	Material-Feature	Number of Drops	Drop Vol (µL)	Asset
Soapy water spray	steel-flat	1	0.2	
	steel-20 mil	2	0.2	
	steel-2 mil	2	0.2	
	total	5	1	

The source terms used for the calculations were summarized in Section 11.1. The seven replicates of the tested asset (Asset RA, as measured in Section 10.3) and the MC output of the 10,000 iterations for each source term calculation technique are shown in Figure H-1. The bell shape of the MC outputs is attributed to the below detection source terms contributing similar low mass values. These results show that the fit method predicted the M_{Asset} within a factor of R=0.45, the indirect fit using V=0.2 µL data within a factor of R=0.27, the indirect fit using V=1 µL data within a factor of R=13.7. The accuracy of the indirect fit using V=1 µL did not meet the acceptance criterion, note that this is the case where the 1 µL drop data is extrapolated to the 0.2 µL condition. The other the fit methods produced results within the acceptance criterion range.

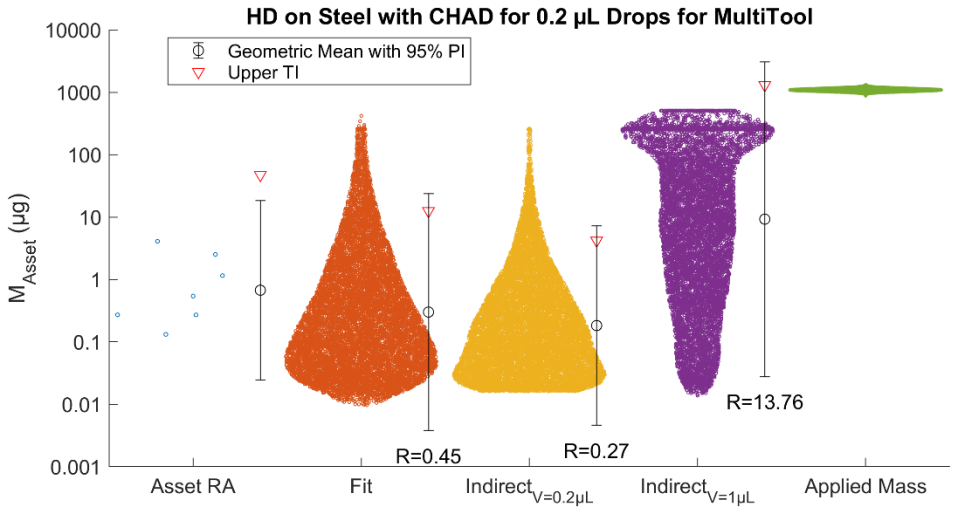


Figure H-1. MC output visualization for the multi-tool asset with 0.2 µL HD drops treated with CHAD. The circle with error bars to the right of each swarm plot represent the geometric mean and 95% PI for the data.

The tabulated outputs of the results are provided in Table H-2. The methodology output B_2 (Figure A-1) of asset level performance as quantified by LD from the applied mass is provided. These data show that CHAD provided 3.21 ± 0.51 LD for the tested asset compared to the applied agent mass, and that the fit predicted 3.56 ± 0.02 LD.

Table H-2. *M_{Asset}* output for the tested asset and calculation variations for the multi-tool asset with 0.2 µL HD drops treated with CHAD.

Type	Geometric Mean (µg)	Upper Bound TI (µg)	Upper Bound 95% PI (µg)	μ_{Output} ($\log_{10}(M_{Asset}$ µg))	σ_{Output} ($\log_{10}(M_{Asset}$ µg))	LD ± CI (relative to applied mass)
Asset RA	0.67	47.25	18.51	-0.17	0.55	3.21 ± 0.51
Fit	0.30	12.48	23.84	-0.52	0.97	3.56 ± 0.02
$Indirect_{V=0.2µL}$	0.18	4.20	7.24	-0.74	0.82	3.78 ± 0.02
$Indirect_{V=1µL}$	9.24	1303.90	3079.66	0.97	1.29	2.07 ± 0.03

The accuracy ratio results (R) comparing each analysis technique to the tested asset are provided in Table H-3. For these data the fit regression provided the most accurate estimate of M_{Asset} with an $R_{Absolute}$ of 2.24. For this asset condition, the accuracy of the indirect fit using V=1 µL did not meet the acceptance criterion; the $R_{Absolute}$ value indicates that this calculation technique predicted a value 13.7× greater than the tested asset RA. The other the fit methods produced results within the acceptance criterion range of $R_{Absolute} < 10$ (R is within 0.1–10).

For context on how well the PI and TI for the calculation techniques represent tested asset, ratio values for the quantity are also provided. If the upper bounds are compared to health-based requirements, then some measure of the accuracy should be considered. Here, the calculations are providing estimates much smaller than the indicated from the test data, likely

due to the differences in degrees of freedom in the calculation. No criterion were established for upper bound estimates.

Table H-3. Accuracy ratio of the calculation variations for the multi-tool asset with 0.2 µL HD drops treated with CHAD.

Type	R: Geo Mean	$R_{Absolute}$: Geo Mean	R: Upper TI	R: Upper PI
Fit / Asset	**0.45**	2.24	0.26	1.29
$Indirect_{V=0.2\mu L}$ / Asset	**0.27**	3.68	0.09	0.39
$Indirect_{V=1\mu L}$ / Asset	**13.76**	13.76	27.60	166.34

Bold blue values are the accuracy ratio that should be within the validation criterion range of 0.1–10.
Red values indicate results are outside the acceptance criterion.

To build trust in the accuracy of using the geometric mean, a histogram of the results are provided in Figure H-2, the red line shows a fitted normal distribution to the log-transformed results. Ideally, the red line should have similar shape and magnitude as the histogram. Deviations of the red line from the histogram indicate that the log-normal distribution may not be an accurate description of the data, and quartile (i.e., non-parametric) analysis may be a better description of the results.

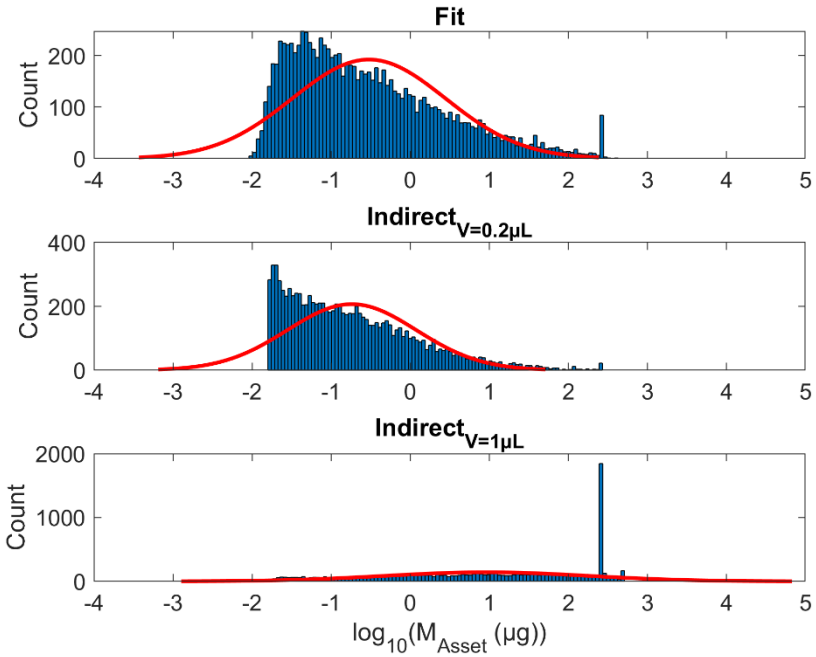


Figure H-2. Histogram of the M_{Asset} output fit to a log-normal distribution (red line), for the multi-tool asset with 0.2 µL HD drops treated with CHAD.

The geometric mean calculation assumes that the data are log-normal, with some deviations from normality seen in Figure H-2 (attributed to the below detection source terms). The MATLAB function *quantile* is used to identify the magnitude of M_{Asset} for specific quantiles such indicating that 95% of the data are less than an indicated value. The quantile output requires

more samples than the seven replicates collected for the Asset RA data, thus only the MC output is presented in the quantile output (Table H-4).

Table H-4. Quantile analysis of the M_{Asset} output for the multi-tool asset with 0.2 µL HD drops treated with CHAD.

Quantile	Fit Analysis (µg)	Indirect$_{V=0.2µL}$ (µg)	Indirect$_{V=1µL}$ (µg)
25%	70.963 (0.6x)	46.775 (0.4x)	630.507 (0.8x)
Median 50%	120.012 (1.0x)	103.988 (1.0x)	765.524 (1.0x)
75%	204.192 (1.7x)	256.276 (2.5x)	828.137 (1.1x)
95%	351.958 (2.9x)	361.151 (3.5x)	1014.490 (1.3x)

Values in parenthesis are the ratio of the value to the median.

The MC calculation tracks the source of agent mass, enabling the attribution of which material-features contributed to the total M_{Asset} output. Figure H-3 provides the relative contribution for each feature type. For example, the steel 2 mil feature was typically responsible for 87.5% of the M_{Asset} mass, and the steel 20 mil feature was typically responsible for 3.2% of the M_{Asset} mass, and the steel-flat response provided a 6% contribution to the M_{Asset} mass. A similar analysis can be done without normalization to the M_{Asset} mass to indicate, for example, that typically 0.15 µg of the 0.3 µg ($M_{Asset,\ fit\ geometric\ mean}$) retained by the asset was attributed to the steel-2 mil feature.

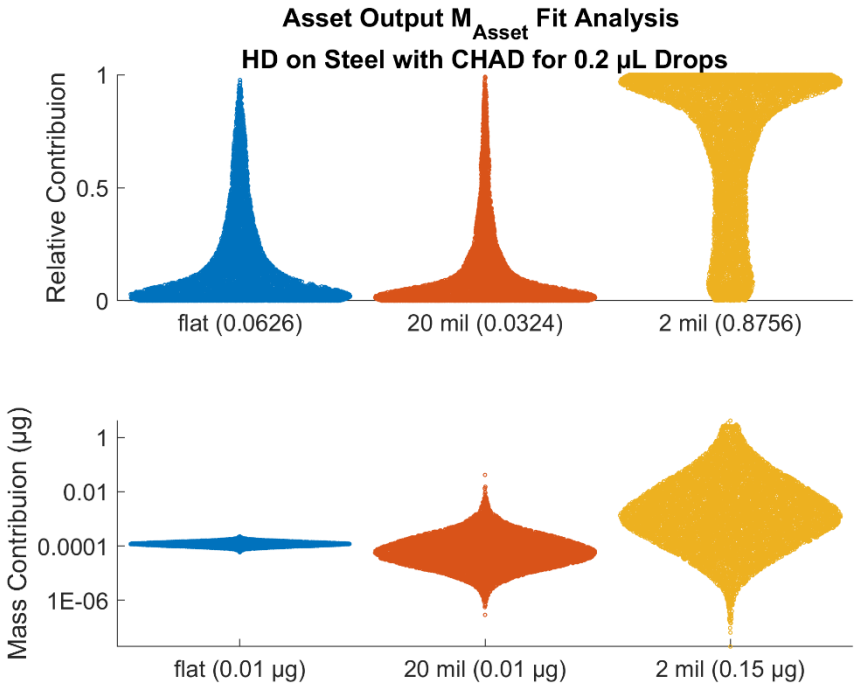


Figure H-3. Relative contribution and mass contribution graphs for each feature for the multi-tool asset with 0.2 µL HD drops treated with soapy water spray, values in parenthesis are the median response for the material-feature indicated.

The contribution calculations enable material attribution, here showing that the 2 mil shim panel was by far the major source of exposure. This relative contribution analysis also

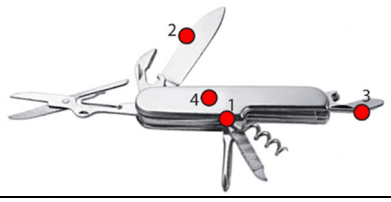
indicates that the accuracy of the of the M_{Asset} output is mostly influenced by the accuracy of the calculation technique for the 2 mil shim as it is the dominant source. For example, poor accuracy of the flat panel feature would have negligible contribution to the M_{Asset} output.

These data have demonstrated the ability of the analysis techniques to scale-up laboratory panel data to represent a small asset contaminated with 1 µL drops and treated with soapy water spray. In this case, the extrapolation of the 0.1 µL data to represent 1 µL drops did not provide the desired scale-up accuracy. However, the other calculation techniques met the criterion of $R_{Absolute} < 10$.

H.2 Multi-tool Asset for 1 µL Drops and CHAD Treatment

The same sequence of analysis is conducted for each of the asset-drop volume-treatment combinations. The list of material-features and drop volumes used for the calculation is provided in Table H-1, based on the detailed description in Table 71. The contamination pattern uses five drops to deliver a total of 1 µL of agent to the asset.

Table H-5. Description of drop count and drop volumes used to represent the contamination of the multi-tool asset with 1 µL drops

Treatment	Material-Feature	Number of Drops	Drop Vol (µL)	Asset
Soapy water spray	steel-flat	1	1	
	steel-20 mil	2	1	
	steel-2 mil	1	1	
	total	4	4	

The source terms used for the calculations were summarized in Section 11.1. The seven replicates of the tested asset (Asset RA, as measured in Section 10.3) and the MC output of the 10,000 iterations for each source term calculation technique are shown in Figure H-4. The bell shape of the MC outputs is attributed to the below detection source terms contributing similar low mass values. The flat tops of the distribution are a result of the large σ value that generated values greater than the applied mass, which were substituted with the applied mass. These results show that the fit method predicted the M_{Asset} within a factor of R=0.85, the indirect fit using V=0.2 µL data within a factor of R=0.02, the indirect fit using V=1 µL data within a factor of R=.33. The accuracy of the indirect fit using V=0.2 µL did not meet the acceptance criterion, note that this is the case where the 0.2 µL drop data is extrapolated to the 1 µL condition. The other the fit methods produced results within the acceptance criterion range.

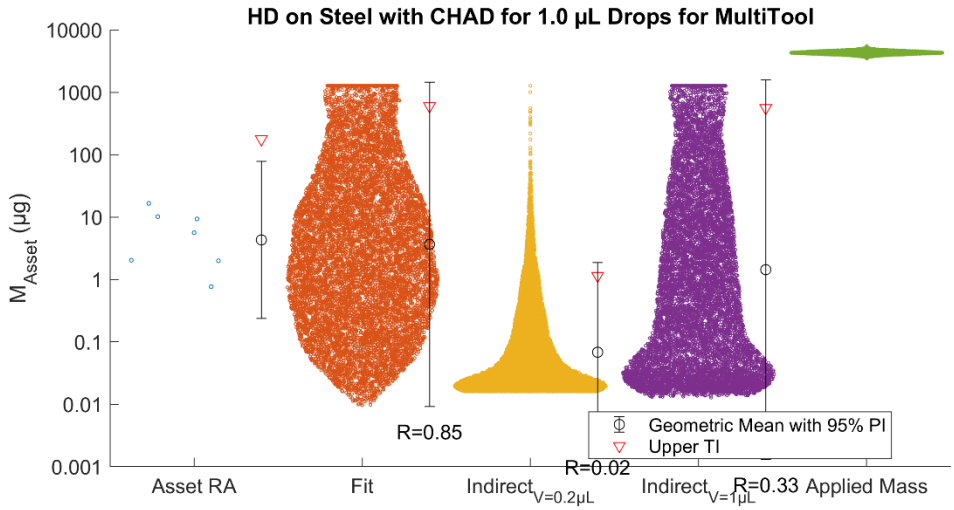


Figure H-4. MC output visualization for the multi-tool asset with 1 µL HD drops treated with CHAD. The circle with error bars to the right of each swarm plot represent the geometric mean and 95% PI for the data.

The tabulated outputs of the results are provided in Table H-6. The methodology output B_2 (Figure A-1) of asset level performance as quantified by LD from the applied mass is provided. These data show that CHAD provided 3.00 ± 0.44 LD for the tested asset compared to the applied agent mass, and that the fit predicted 3.07 ± 0.03 LD.

Table H-6. *M_{Asset}* output for the tested asset and calculation variations for the multi-tool asset with 1 µL HD drops treated with CHAD.

Type	Geometric Mean (µg)	Upper Bound TI (µg)	Upper Bound 95% PI (µg)	μ_{Output} ($\log_{10}(M_{Asset}$ µg))	σ_{Output} ($\log_{10}(M_{Asset}$ µg))	LD ± CI (relative to applied mass)
Asset RA	4.30	176.37	77.83	0.63	0.48	3.00 ± 0.44
Fit	3.65	598.37	1450.79	0.56	1.33	3.07 ± 0.03
$Indirect_{V=0.2µL}$	0.07	1.14	1.86	-1.17	0.73	4.80 ± 0.01
$Indirect_{V=1µL}$	1.43	558.88	1575.04	0.16	1.55	3.48 ± 0.03

The accuracy ratio results (R) comparing each analysis technique to the tested asset are provided in Table H-7. For these data the fit regression provided the most accurate estimate of M_{Asset} with an $R_{Absolute}$ of 1.18. For this asset condition, the accuracy of the indirect fit using V=0.2 µL did not meet the acceptance criterion; the $R_{Absolute}$ value indicates that this calculation technique predicted a value 63× less than the tested asset RA. The other the fit methods produced results within the acceptance criterion range of $R_{Absolute} < 10$ (R is within 0.1–10).

For context on how well the PI and TI for the calculation techniques represent tested asset, ratio values for the quantity are also provided. If the upper bounds are compared to health-based requirements, then some measure of the accuracy should be considered. Here, the calculations are providing estimates much smaller than the indicated from the test data, likely

due to the differences in degrees of freedom in the calculation. No criterion were established for upper bound estimates.

Table H-7. Accuracy ratio of the calculation variations for the multi-tool asset with 1 µL HD drops treated with CHAD.

Type	R: Geo Mean	$R_{Absolute}$: Geo Mean	R: Upper TI	R: Upper PI
Fit / Asset	**0.85**	1.18	3.39	18.64
$Indirect_{V=0.2µL}$ / Asset	**0.02**	63.07	0.01	0.02
$Indirect_{V=1µL}$ / Asset	**0.33**	3.00	3.17	20.24

Bold blue values are the accuracy ratio that should be within the validation criterion range of 0.1–10.
Red values indicate results are outside the acceptance criterion.

To build trust in the accuracy of using the geometric mean, a histogram of the results are provided in Figure H-5, the red line shows a fitted normal distribution to the log-transformed results. Ideally, the red line should have similar shape and magnitude as the histogram. Deviations of the red line from the histogram indicate that the log-normal distribution may not be an accurate description of the data, and quartile (i.e., non-parametric) analysis may be a better description of the results.

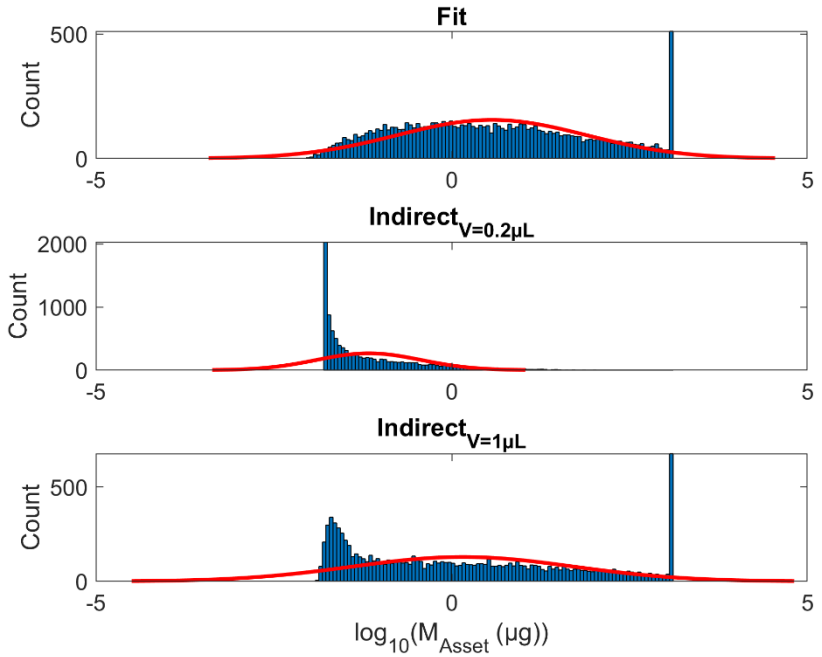


Figure H-5. Histogram of the M_{Asset} output fit to a log-normal distribution (red line), for the multi-tool asset with 1 µL HD drops treated with CHAD.

The geometric mean calculation assumes that the data are log-normal, with some deviations from normality seen in Figure H-5 (attributed to the below detection source terms). The MATLAB function *quantile* is used to identify the magnitude of M_{Asset} for specific quantiles such indicating that 95% of the data are less than an indicated value. The quantile output requires

more samples than the seven replicates collected for the Asset RA data, thus only the MC output is presented in the quantile output (Table H-4).

Table H-8. Quantile analysis of the M_{Asset} output for the multi-tool asset with 1 µL HD drops treated with CHAD.

Quantile	Fit Analysis (µg)	Indirect$_{V=0.2µL}$ (µg)	Indirect$_{V=1µL}$ (µg)
25%	0.311 (0.1x)	0.019 (0.6x)	0.050 (0.1x)
Median 50%	2.850 (1.0x)	0.034 (1.0x)	0.786 (1.0x)
75%	33.994 (11.9x)	0.150 (4.4x)	22.131 (28.2x)
95%	1185.685 (416.1x)	2.377 (70.2x)	1268.016 (1613.3x)

Values in parenthesis are the ratio of the value to the median.

The MC calculation tracks the source of agent mass, enabling the attribution of which material-features contributed to the total M_{Asset} output. Figure H-6 provides the relative contribution for each feature type. For example, the steel 2 mil feature was typically responsible for 98.8% of the M_{Asset} mass, and the steel 20 mil feature was typically responsible for 0.6% of the M_{Asset} mass, and the steel-flat response provided a 0.3% contribution to the M_{Asset} mass. A similar analysis can be done without normalization to the M_{Asset} mass to indicate, for example, that typically 2.79 µg of the 3.65 µg ($M_{Asset,\ fit\ geometric\ mean}$) retained by the asset was attributed to the steel-2 mil feature.

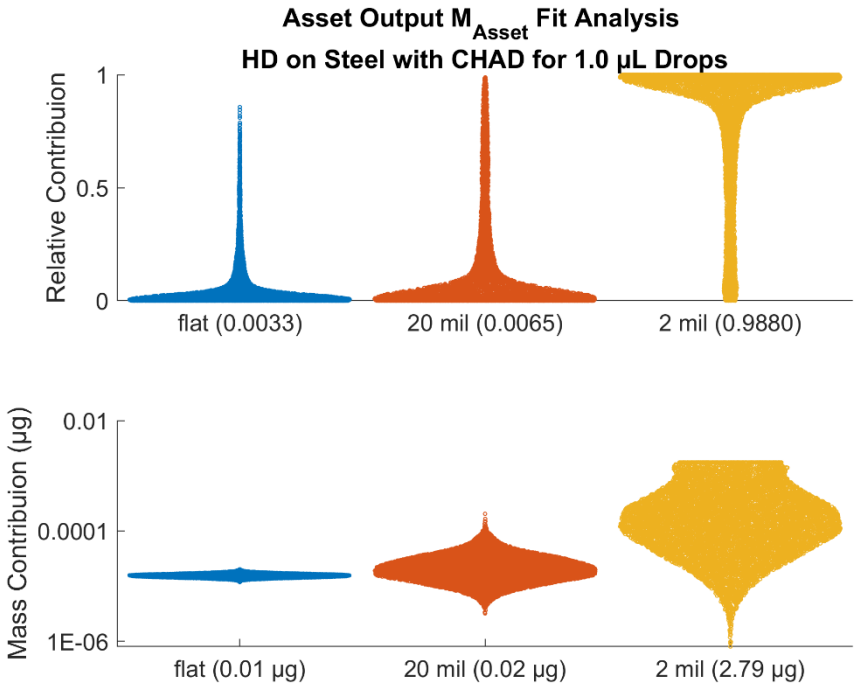


Figure H-6. Relative contribution and mass contribution graphs for each feature for the multi-tool asset with 1 µL HD drops treated with CHAD, values in parenthesis are the median response for the material-feature indicated.

The contribution calculations enable material attribution, here showing that the 2 mil shim panel was by far the major source of exposure. This relative contribution analysis also

indicates that the accuracy of the of the M_{Asset} output is mostly influenced by the accuracy of the calculation technique for the 2 mil shim as it is the dominant source. For example, poor accuracy of the flat panel feature would have negligible contribution to the M_{Asset} output.

These data have demonstrated the ability of the analysis techniques to scale-up laboratory panel data to represent a small asset contaminated with 1 µL drops and treated with soapy water spray. In this case, the extrapolation of the 0.2 µL data to represent 1 µL drops did not provide the desired scale-up accuracy. However, the other calculation techniques met the criterion of $R_{Absolute} < 10$.

H.3 Picatinny Rail Asset for 0.2 µL Drops with CHAD Treatment

The same sequence of analysis is conducted for each of the asset-drop volume-treatment combinations. The list of material-features and drop volumes used for the calculation is provided in Table H-9, based on the detailed description in Table 72. The contamination pattern uses five drops to deliver a total of 1 µL of agent to the asset.

Table H-9. Description of drop count and drop volumes used to represent the contamination of the multi-tool asset with 0.2 µL drops

Treatment	Material-Feature	Number of Drops	Drop Vol (µL)	Asset
Soapy water spray	steel-flat	1	0.2	
	steel-20 mil	1	0.2	
	steel-2 mil	3	0.2	
	total	5	1	

The source terms used for the calculations were summarized in Section 11.1. The seven replicates of the tested asset (Asset RA, as measured in Section 10.3) and the MC output of the 10,000 iterations for each source term calculation technique are shown in Figure H-7. The bell shape of the MC outputs is attributed to the below detection source terms contributing similar low mass values. These results show that the fit method predicted the M_{Asset} within a factor of R=0.37, the indirect fit using V=0.2 µL data within a factor of R=0.22, the indirect fit using V=1 µL data within a factor of R=17.22. The accuracy of the indirect fit using V=1 µL did not meet the acceptance criterion, note that this is the case where the 1 µL drop data is extrapolated to the 0.2 µL condition. The other the fit methods produced results within the acceptance criterion range.

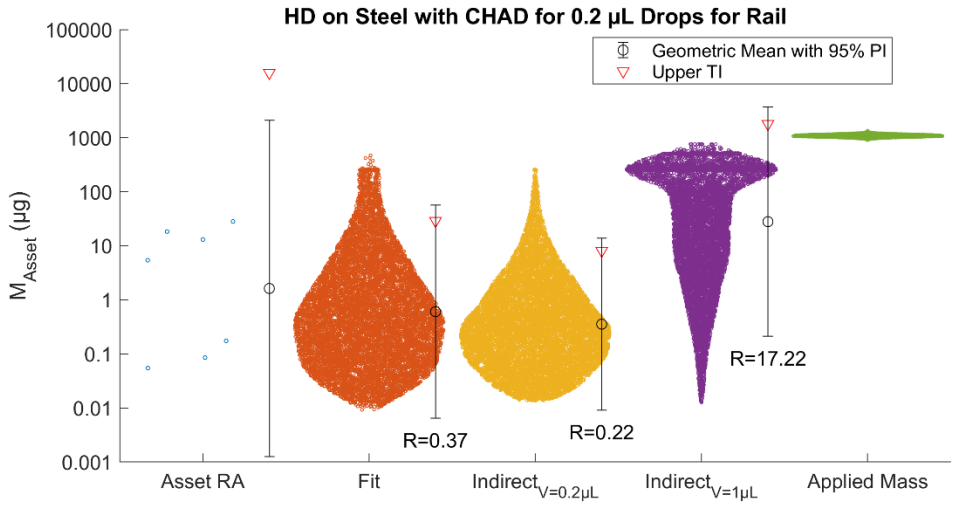


Figure H-7. MC output visualization for the Picatinny rail asset with 0.2 µL HD drops treated with CHAD. The circle with error bars to the right of each swarm plot represent the geometric mean and 95% PI for the data.

The tabulated outputs of the results are provided in Table H-10. The methodology output B_2 (Figure A-1) of asset level performance as quantified by LD from the applied mass is provided. These data show that CHAD provided 2.83 ± 1.10 LD for the tested asset compared to the applied agent mass, and that the fit predicted 3.26 ± 0.02 LD.

Table H-10. M_{Asset} output for the tested asset and calculation variations for the Picatinny rail asset with 0.2 µL HD drops treated with CHAD.

Type	Geometric Mean (µg)	Upper Bound TI (µg)	Upper Bound 95% PI (µg)	μ_{Output} ($\log_{10}(M_{Asset}$ µg))	σ_{Output} ($\log_{10}(M_{Asset}$ µg))	LD ± CI (relative to applied mass)
Asset RA	1.62	15866.93	2097.39	0.21	1.19	2.83 ± 1.10
Fit	0.60	28.97	56.72	-0.22	1.01	3.26 ± 0.02
$Indirect_{V=0.2µL}$	0.36	8.10	13.95	-0.45	0.81	3.49 ± 0.02
$Indirect_{V=1µL}$	27.97	1800.79	3711.58	1.45	1.08	1.59 ± 0.02

The accuracy ratio results (R) comparing each analysis technique to the tested asset are provided in Table H-11. For these data the fit regression provided the most accurate estimate of M_{Asset} with an $R_{Absolute}$ of 2.69. For this asset condition, the accuracy of the indirect fit using V=1 µL did not meet the acceptance criterion; the $R_{Absolute}$ value indicates that this calculation technique predicted a value 17.2× greater than the tested asset RA. The other the fit methods produced results within the acceptance criterion range of $R_{Absolute} < 10$ (R is within 0.1–10).

For context on how well the PI and TI for the calculation techniques represent tested asset, ratio values for the quantity are also provided. If the upper bounds are compared to health-based requirements, then some measure of the accuracy should be considered. Here, the calculations are providing estimates much smaller than the indicated from the test data, likely

due to the differences in degrees of freedom in the calculation. No criterion were established for upper bound estimates.

Table H-11. Accuracy ratio of the calculation variations for the Picatinny rail asset with 0.2 µL HD drops treated with CHAD.

Type	R: Geo Mean	$R_{Absolute}$: Geo Mean	R: Upper TI	R: Upper PI
Fit / Asset	**0.37**	2.69	0.002	0.027
$Indirect_{V=0.2\mu L}$ / Asset	**0.22**	4.57	0.001	0.007
$Indirect_{V=1\mu L}$ / Asset	**17.22**	17.22	0.11	1.77

Bold blue values are the accuracy ratio that should be within the validation criterion range of 0.1–10.
Red values indicate results are outside the acceptance criterion.

To build trust in the accuracy of using the geometric mean, a histogram of the results are provided in Figure H-8, the red line shows a fitted normal distribution to the log-transformed results. Ideally, the red line should have similar shape and magnitude as the histogram. Deviations of the red line from the histogram indicate that the log-normal distribution may not be an accurate description of the data, and quartile (i.e., non-parametric) analysis may be a better description of the results.

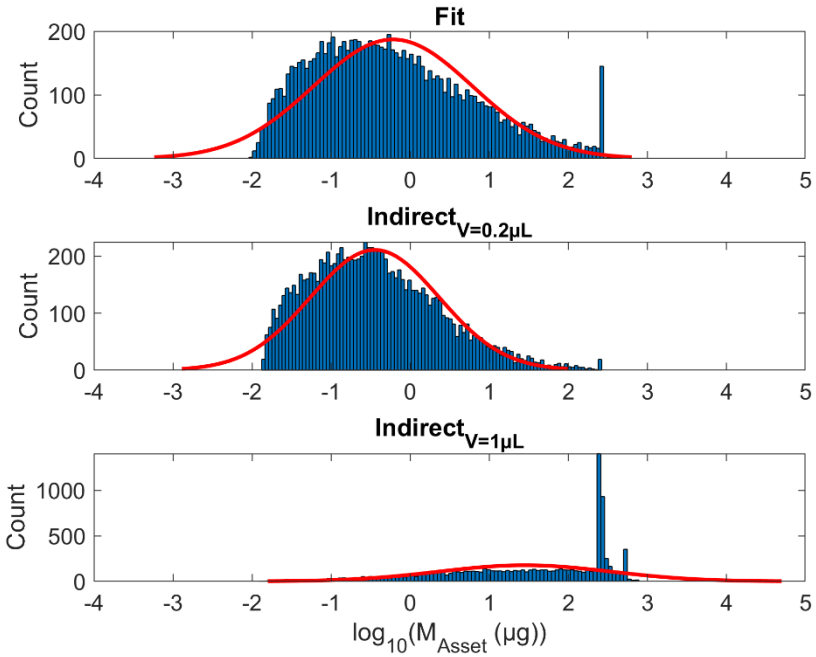


Figure H-8. Histogram of the M_{Asset} output fit to a log-normal distribution (red line), for the Picatinny rail asset with 0.2 µL HD drops treated with CHAD.

The geometric mean calculation assumes that the data are log-normal, with some deviations from normality seen in Figure H-2 (attributed to the below detection source terms). The MATLAB function *quantile* is used to identify the magnitude of M_{Asset} for specific quantiles such indicating that 95% of the data are less than an indicated value. The quantile output requires

more samples than the seven replicates collected for the Asset RA data, thus only the MC output is presented in the quantile output (Table H-12).

Table H-12. Quantile analysis of the M_{Asset} output for the Picatinny rail asset with 0.2 µL HD drops treated with CHAD.

Quantile	Fit Analysis (µg)	Indirect$_{V=0.2µL}$ (µg)	Indirect$_{V=1µL}$ (µg)
25%	0.099 (0.2x)	0.085 (0.3x)	5.093 (0.1x)
Median 50%	0.451 (1.0x)	0.294 (1.0x)	53.579 (1.0x)
75%	2.675 (5.9x)	1.163 (4.0x)	254.325 (4.7x)
95%	47.993 (106.4x)	11.133 (37.9x)	442.053 (8.3x)

Values in parenthesis are the ratio of the value to the median.

The MC calculation tracks the source of agent mass, enabling the attribution of which material-features contributed to the total M_{Asset} output. Figure H-9 provides the relative contribution for each feature type. For example, the steel 2 mil feature was typically responsible for 96.2% of the M_{Asset} mass, and the steel 20 mil feature was typically responsible for 0.4% of the M_{Asset} mass, and the steel-flat response provided a 2.6% contribution to the M_{Asset} mass. A similar analysis can be done without normalization to the M_{Asset} mass to indicate, for example, that typically 0.43 µg of the 0.6 µg ($M_{Asset,\ fit\ geometric\ mean}$) retained by the asset was attributed to the steel-2 mil feature.

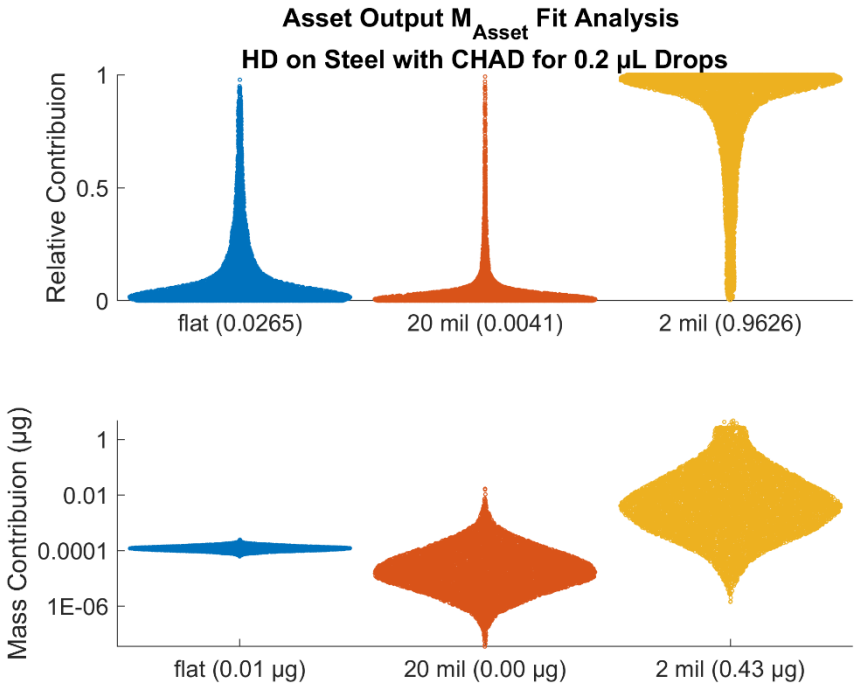


Figure H-9. Relative contribution and mass contribution graphs for each feature for the Picatinny rail asset with 0.2 µL HD drops treated with CHAD, values in parenthesis are the median response for the material-feature indicated.

The contribution calculations enable material attribution, here showing that the 2 mil shim panel was by far the major source of exposure. This relative contribution analysis also

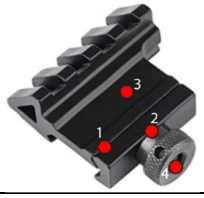
indicates that the accuracy of the of the M_{Asset} output is mostly influenced by the accuracy of the calculation technique for the 2 mil shim as it is the dominant source. For example, poor accuracy of the flat panel feature would have negligible contribution to the M_{Asset} output.

These data have demonstrated the ability of the analysis techniques to scale-up laboratory panel data to represent a small asset contaminated with 1 µL drops and treated with soapy water spray. In this case, the extrapolation of the 1 µL data to represent 0.2 µL drops did not provide the desired scale-up accuracy. However, the other calculation techniques met the criterion of $R_{Absolute} < 10$.

H.4 Picatinny Rail Asset with 1 µL Drops and CHAD Treatment

The same sequence of analysis is conducted for each of the asset-drop volume-treatment combinations. The list of material-features and drop volumes used for the calculation is provided in Table H-13, based on the detailed description in Table 72. The contamination pattern uses five drops to deliver a total of 1 µL of agent to the asset.

Table H-13. Description of drop count and drop volumes used to represent the contamination of the Picatinny rail asset with 1 µL drops

Treatment	Material-Feature	Number of Drops	Drop Vol (µL)	Asset
Soapy water spray	steel-flat	1	1	
	steel-20 mil	0	1	
	steel-2 mil	3	1	
	total	4	4	

The source terms used for the calculations were summarized in Section 11.1. The seven replicates of the tested asset (Asset RA, as measured in Section 10.3) and the MC output of the 10,000 iterations for each source term calculation technique are shown in Figure H-10. The bell shape of the MC outputs is attributed to the below detection source terms contributing similar low mass values. These results show that the fit method predicted the M_{Asset} within a factor of R=0.18, the indirect fit using V=0.2 µL data within a factor of R=0.00, the indirect fit using V=1 µL data within a factor of R=0.11. The accuracy of the indirect fit using V=0.2 µL did not meet the acceptance criterion, note that this is the case where the 0.2 µL drop data is extrapolated to the 1 µL condition. The other the fit methods produced results within the acceptance criterion range.

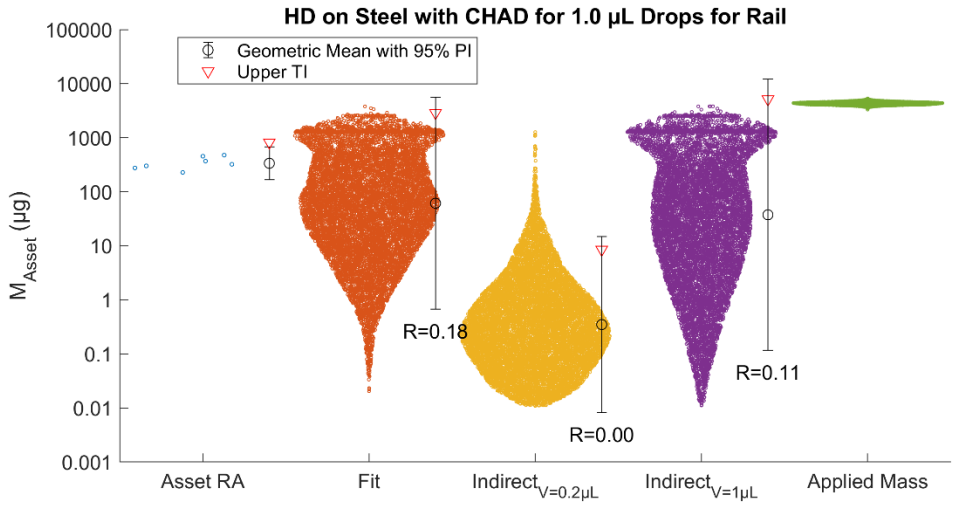


Figure H-10. MC output visualization for the Picatinny rail asset with 1 µL HD drops treated with CHAD. The circle with error bars to the right of each swarm plot represent the geometric mean and 95% PI for the data.

The tabulated outputs of the results are provided in Table H-14. The methodology output B_2 (Figure A-1) of asset level performance as quantified by LD from the applied mass is provided. These data show that CHAD provided 1.11 ± 0.11 LD for the tested asset compared to the applied agent mass, and that the fit predicted 1.85 ± 0.02 LD.

Table H-14. M_{Asset} output for the tested asset and calculation variations for the Picatinny rail asset with 1 µL HD drops treated with CHAD.

Type	Geometric Mean (µg)	Upper Bound TI (µg)	Upper Bound 95% PI (µg)	μ_{Output} ($\log_{10}(M_{Asset}$ µg))	σ_{Output} ($\log_{10}(M_{Asset}$ µg))	LD ± CI (relative to applied mass)
Asset RA	336.53	820.06	673.97	2.53	0.12	1.11 ± 0.11
Fit	61.36	2875.20	5607.92	1.79	1.00	1.85 ± 0.02
$\text{Indirect}_{V=0.2\mu L}$	0.35	8.51	14.82	-0.46	0.83	4.09 ± 0.02
$\text{Indirect}_{V=1\mu L}$	37.48	5149.92	12107.81	1.57	1.28	2.06 ± 0.03

The accuracy ratio results (R) comparing each analysis technique to the tested asset are provided in Table H-15. For these data the fit regression provided the most accurate estimate of M_{Asset} with an $R_{Absolute}$ of 5.48. For this asset condition, the accuracy of the indirect fit using V=0.2 µL did not meet the acceptance criterion; the $R_{Absolute}$ value indicates that this calculation technique predicted a value 963.7× less than the tested asset RA. The other the fit methods produced results within the acceptance criterion range of $R_{Absolute} < 10$ (R is within 0.1–10).

For context on how well the PI and TI for the calculation techniques represent tested asset, ratio values for the quantity are also provided. If the upper bounds are compared to health-based requirements, then some measure of the accuracy should be considered. Here, the calculations are providing estimates much smaller than the indicated from the test data, likely

due to the differences in degrees of freedom in the calculation. No criterion were established for upper bound estimates.

Table H-15. Accuracy ratio of the calculation variations for the Picatinny rail asset with 1 µL HD drops treated with CHAD.

Type	R: Geo Mean	$R_{Absolute}$: Geo Mean	R: Upper TI	R: Upper PI
Fit / Asset	**0.18**	5.48	3.51	8.32
$Indirect_{V=0.2µL}$ / Asset	**0.001**	963.35	0.01	0.02
$Indirect_{V=1µL}$ / Asset	**0.11**	8.98	6.28	17.96

Bold blue values are the accuracy ratio that should be within the validation criterion range of 0.1–10.
Red values indicate results are outside the acceptance criterion.

To build trust in the accuracy of using the geometric mean, a histogram of the results are provided in Figure H-11, the red line shows a fitted normal distribution to the log-transformed results. Ideally, the red line should have similar shape and magnitude as the histogram. Deviations of the red line from the histogram indicate that the log-normal distribution may not be an accurate description of the data, and quartile (i.e., non-parametric) analysis may be a better description of the results.

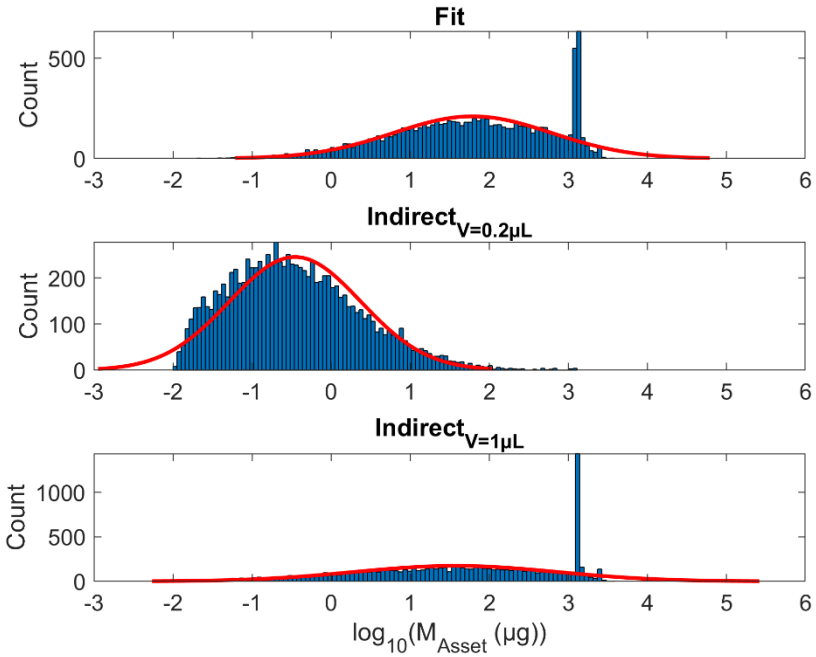


Figure H-11. Histogram of the M_{Asset} output fit to a log-normal distribution (red line), for the Picatinny rail asset with 1 µL HD drops treated with CHAD.

The geometric mean calculation assumes that the data are log-normal, with some deviations from normality seen in Figure H-11 (attributed to the below detection source terms). The MATLAB function *quantile* is used to identify the magnitude of M_{Asset} for specific quantiles such indicating that 95% of the data are less than an indicated value. The quantile output requires

more samples than the seven replicates collected for the Asset RA data, thus only the MC output is presented in the quantile output (Table H-16).

Table H-16. Quantile analysis of the M_{Asset} output for the Picatinny rail asset with 1 µL HD drops treated with CHAD.

Quantile	Fit Analysis (µg)	Indirect$_{V=0.2µL}$ (µg)	Indirect$_{V=1µL}$ (µg)
25%	12.248 (0.2x)	0.083 (0.3x)	4.487 (0.1x)
Median 50%	68.974 (1.0x)	0.290 (1.0x)	48.650 (1.0x)
75%	431.251 (6.3x)	1.165 (4.0x)	589.264 (12.1x)
95%	1321.855 (19.2x)	10.688 (36.9x)	1373.806 (28.2x)

Values in parenthesis are the ratio of the value to the median.

The MC calculation tracks the source of agent mass, enabling the attribution of which material-features contributed to the total M_{Asset} output. Figure H-12 provides the relative contribution for each feature type. For example, the steel 2 mil feature was typically responsible for 99.9% of the M_{Asset} mass, and the steel 20 mil feature was typically responsible for 0% of the M_{Asset} mass, and the steel-flat response provided a 0.01% contribution to the M_{Asset} mass. A similar analysis can be done without normalization to the M_{Asset} mass to indicate, for example, that typically 69 µg of the 61 µg ($M_{Asset,\ fit\ geometric\ mean}$) retained by the asset was attributed to the steel-2 mil feature. The use of median statistics for the material-feature contributions enabled the contribution value to be greater than the total asset geometric mean.

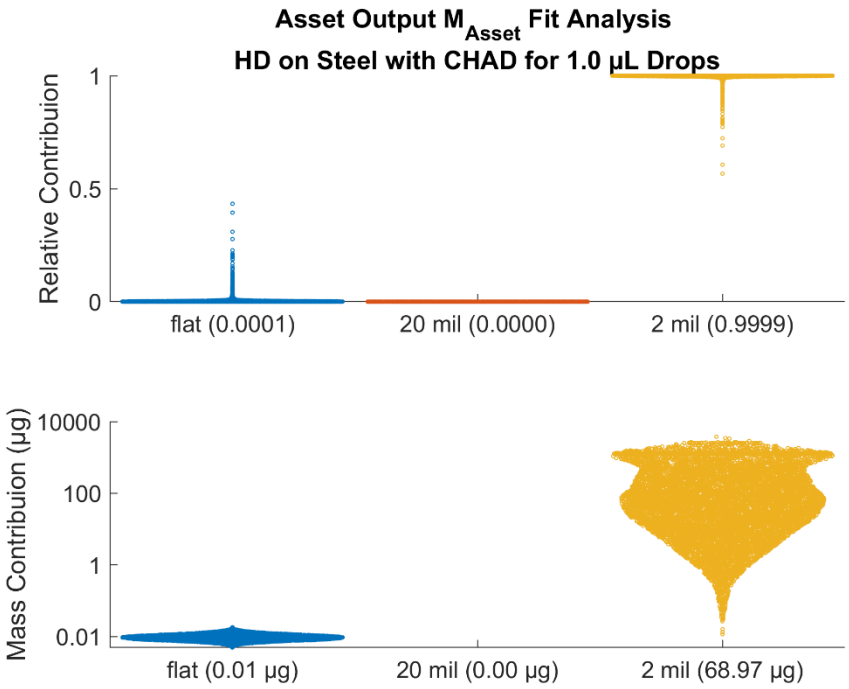


Figure H-12. Relative contribution and mass contribution graphs for each feature for the Picatinny rail asset with 1 µL HD drops treated with CHAD, values in parenthesis are the median response for the material-feature indicated.

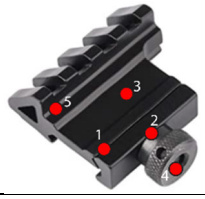
The contribution calculations enable material attribution, here showing that the 2 mil shim panel was by far the major source of exposure. This relative contribution analysis also indicates that the accuracy of the of the M_{Asset} output is mostly influenced by the accuracy of the calculation technique for the 2 mil shim as it is the dominant source. For example, poor accuracy of the flat panel feature would have negligible contribution to the M_{Asset} output.

These data have demonstrated the ability of the analysis techniques to scale-up laboratory panel data to represent a small asset contaminated with 1 µL drops and treated with soapy water spray. In this case, the extrapolation of the 0.1 µL data to represent 1 µL drops did not provide the desired scale-up accuracy. However, the other calculation techniques met the criterion of $R_{Absolute} < 10$.

H.5 Picatinny Rail Asset for 0.2 µL Drops with Soapy Water Spray Treatment

The same sequence of analysis is conducted for each of the asset-drop volume-treatment combinations. The list of material-features and drop volumes used for the calculation is provided in Table H-17, based on the detailed description in Table 72. The contamination pattern uses five drops to deliver a total of 0.2 µL of agent to the asset.

Table H-17. Description of drop count and drop volumes used to represent the contamination of the Picatinny rail asset with 0.2 µL drops

Treatment	Material-Feature	Number of Drops	Drop Vol (µL)	Asset
Soapy water spray	steel-flat	1	0.2	
	steel-20 mil	1	0.2	
	steel-2 mil	3	0.2	
	total	5	1	

The source terms used for the calculations were summarized in Section 11.1. The seven replicates of the tested asset (Asset RA, as measured in Section 10.3) and the MC output of the 10,000 iterations for each source term calculation technique are shown in Figure H-13. The bell shape of the MC outputs is attributed to the below detection source terms contributing similar low mass values. These results show that the fit method predicted the M_{Asset} within a factor of R=0.86, the indirect fit using V=0.2 µL data within a factor of R=0.79, the indirect fit using V=1 µL data within a factor of R=4.76. All fit methods produced results within the acceptance criterion range.

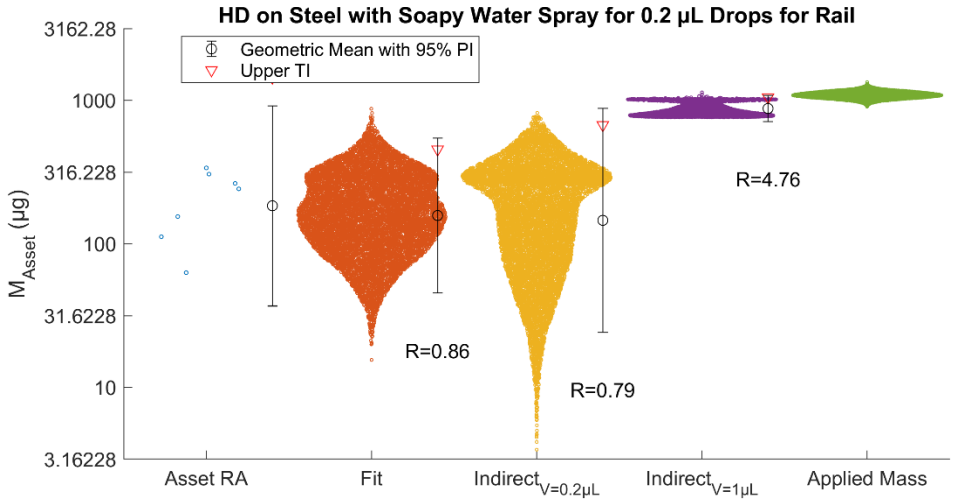


Figure H-13. MC output visualization for the Picatinny rail asset with 0.2 µL HD drops treated with soapy water spray. The circle with error bars to the right of each swarm plot represent the geometric mean and 95% PI for the data.

The tabulated outputs of the results are provided in Table H-18. The methodology output B_2 (Figure A-1) of asset level performance as quantified by LD from the applied mass is provided. These data show that soapy water spray provided 0.77 ± 0.25 LD for the tested asset compared to the applied agent mass, and that the fit predicted 0.84 ± 0.01 LD.

Table H-18. M_{Asset} output for the tested asset and calculation variations for the Picatinny rail asset with 0.2 µL HD drops treated with soapy water spray.

Type	Geometric Mean (µg)	Upper Bound TI (µg)	Upper Bound 95% PI (µg)	μ_{Output} ($\log_{10}(M_{Asset}$ µg))	σ_{Output} ($\log_{10}(M_{Asset}$ µg))	LD ± CI (relative to applied mass)
Asset RA	184.18	1441.92	916.40	2.27	0.27	0.77 ± 0.25
Fit	157.60	454.65	546.49	2.20	0.28	0.84 ± 0.01
$Indirect_{V=0.2µL}$	145.85	672.92	877.57	2.16	0.40	0.87 ± 0.01
$Indirect_{V=1µL}$	876.93	1049.59	1082.86	2.94	0.05	0.09 ± 0.00

The accuracy ratio results (R) comparing each analysis technique to the tested asset are provided in Table H-19. For these data the fit regression provided the most accurate estimate of M_{Asset} with an $R_{Absolute}$ of 1.17. All the fit methods produced results within the acceptance criterion range of $R_{Absolute} < 10$ (R is within 0.1–10).

For context on how well the PI and TI for the calculation techniques represent tested asset, ratio values for the quantity are also provided. If the upper bounds are compared to health-based requirements, then some measure of the accuracy should be considered. Here, the calculations are providing estimates much smaller than the indicated from the test data, likely due to the differences in degrees of freedom in the calculation. No criterion were established for upper bound estimates.

Table H-19. Accuracy ratio of the calculation variations for the Picatinny rail asset with 0.2 µL HD drops treated with soapy water spray.

Type	R: Geo Mean	$R_{Absolute}$: Geo Mean	R: Upper TI	R: Upper PI
Fit / Asset	**0.86**	1.17	0.32	0.60
$Indirect_{V=0.2µL}$ / Asset	**0.79**	1.26	0.47	0.96
$Indirect_{V=1µL}$ / Asset	**4.76**	4.76	0.73	1.18

Bold blue values are the accuracy ratio that should be within the validation criterion range of 0.1–10.
Red values indicate results are outside the acceptance criterion.

To build trust in the accuracy of using the geometric mean, a histogram of the results are provided in Figure H-14, the red line shows a fitted normal distribution to the log-transformed results. Ideally, the red line should have similar shape and magnitude as the histogram. Deviations of the red line from the histogram indicate that the log-normal distribution may not be an accurate description of the data, and quartile (i.e., non-parametric) analysis may be a better description of the results.

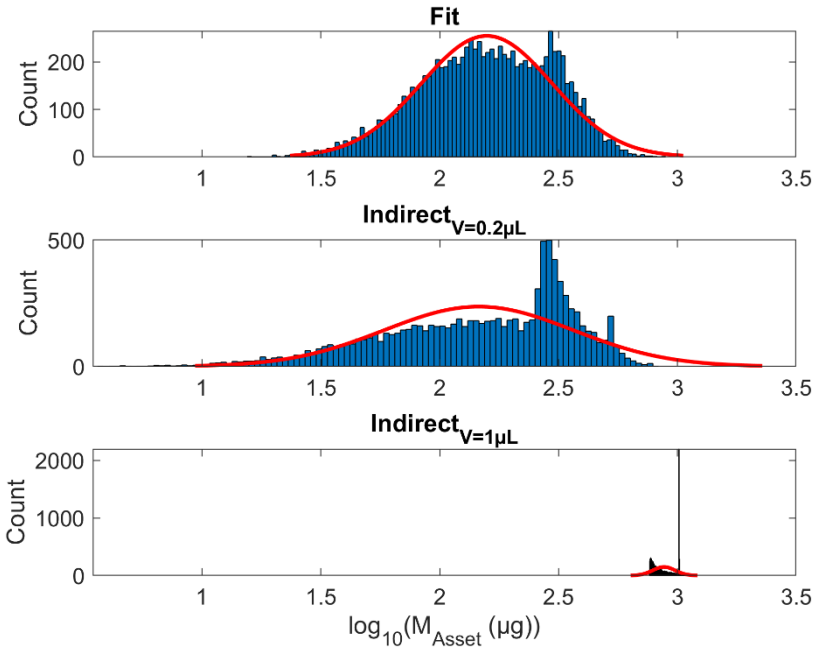


Figure H-14. Histogram of the M_{Asset} output fit to a log-normal distribution (red line), for the Picatinny rail asset with 0.2 µL HD drops treated with soapy water spray.

The geometric mean calculation assumes that the data are log-normal, with some deviations from normality seen in Figure H-14 (attributed to the below detection source terms). The MATLAB function *quantile* is used to identify the magnitude of M_{Asset} for specific quantiles such indicating that 95% of the data are less than an indicated value. The quantile output requires more samples than the seven replicates collected for the Asset RA data, thus only the MC output is presented in the quantile output (Table H-20).

Table H-20. Quantile analysis of the M_{Asset} output for the Picatinny rail asset with 0.2 µL HD drops treated with soapy water spray.

Quantile	Fit Analysis (µg)	Indirect$_{V=0.2µL}$ (µg)	Indirect$_{V=1µL}$ (µg)
25%	100.381 (0.6x)	77.942 (0.4x)	792.880 (0.9x)
Median 50%	160.844 (1.0x)	175.642 (1.0x)	850.827 (1.0x)
75%	262.710 (1.6x)	297.431 (1.7x)	1014.410 (1.2x)
95%	407.557 (2.5x)	491.104 (2.8x)	1015.769 (1.2x)

Values in parenthesis are the ratio of the value to the median.

The MC calculation tracks the source of agent mass, enabling the attribution of which material-features contributed to the total M_{Asset} output. Figure H-15 provides the relative contribution for each feature type. For example, the steel 2 mil feature was typically responsible for 97.2% of the M_{Asset} mass, and the steel 20 mil feature was typically responsible for 2.7% of the M_{Asset} mass, and the steel-flat response provided a 0.01% contribution to the M_{Asset} mass. A similar analysis can be done without normalization to the M_{Asset} mass to indicate, for example, that typically 143 µg of the 157 µg ($M_{Asset,\ fit\ geometric\ mean}$) retained by the asset was attributed to the steel-2 mil feature.

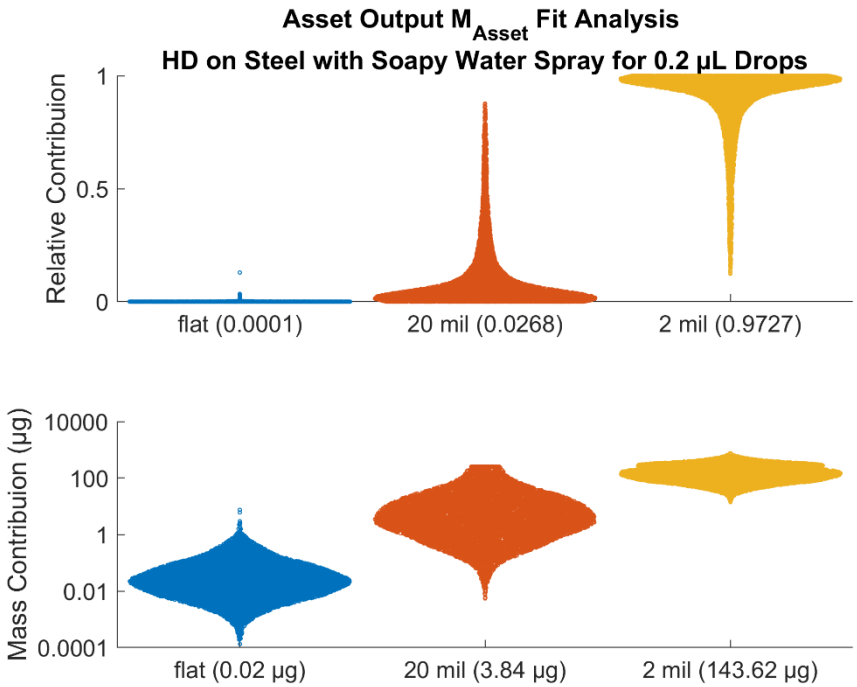


Figure H-15. Relative contribution and mass contribution graphs for each feature for the Picatinny rail asset with 0.2 µL HD drops treated with soapy water spray, values in parenthesis are the median response for the material-feature indicated.

The contribution calculations enable material attribution, here showing that the 2 mil shim panel was by far the major source of exposure. This relative contribution analysis also indicates that the accuracy of the of the M_{Asset} output is mostly influenced by the accuracy of the calculation technique for the 2 mil shim as it is the dominant source. For example, poor accuracy of the flat panel feature would have negligible contribution to the M_{Asset} output.

These data have demonstrated the ability of the analysis techniques to scale-up laboratory panel data to represent a small asset contaminated with 1 µL drops and treated with soapy water spray. In this case, all calculation techniques met the criterion of $R_{Absolute} < 10$.

H.6 Picatinny Rail Asset with 1 µL Drops and Soapy Water Spray Treatment

The same sequence of analysis is conducted for each of the asset-drop volume-treatment combinations. The list of material-features and drop volumes used for the calculation is provided in Table H-21, based on the detailed description in Table 72. The contamination pattern uses five drops to deliver a total of 1 µL of agent to the asset.

Table H-21. Description of drop count and drop volumes used to represent the contamination of the Picatinny rail asset with 1 µL drops

Treatment	Material-Feature	Number of Drops	Drop Vol (µL)	Asset
Soapy water spray	steel-flat	1	1	
	steel-20 mil	0	1	
	steel-2 mil	3	1	
	total	4	4	

The source terms used for the calculations were summarized in Section 11.1. The seven replicates of the tested asset (Asset RA, as measured in Section 10.3) and the MC output of the 10,000 iterations for each source term calculation technique are shown in Figure H-16. The bell shape of the MC outputs is attributed to the below detection source terms contributing similar low mass values. These results show that the fit method predicted the M_{Asset} within a factor of R=1.00, the indirect fit using V=0.2 µL data within a factor of R=0.09, the indirect fit using V=1 µL data within a factor of R=1.52. The accuracy of the indirect fit using V=0.2 µL did not meet the acceptance criterion, note that this is the case where the 0.2 µL drop data is extrapolated to the 1 µL condition. The other the fit methods produced results within the acceptance criterion range.

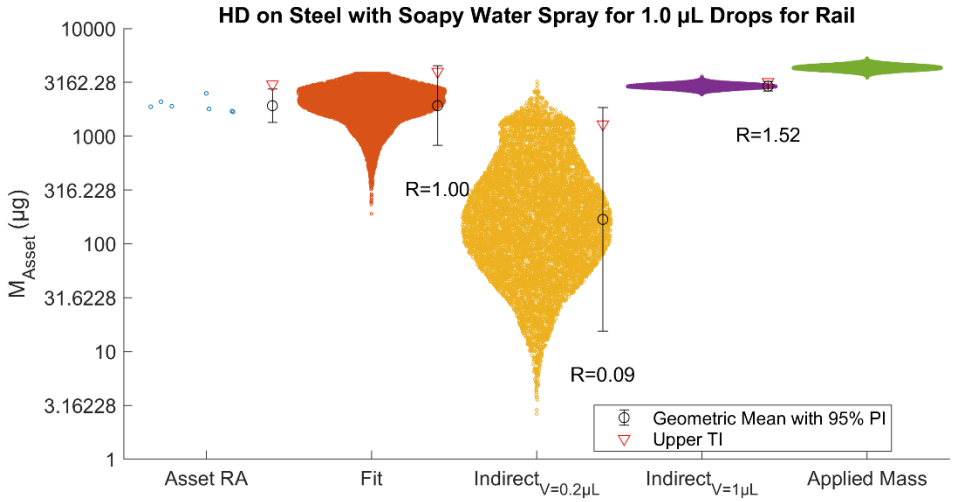


Figure H-16. MC output visualization for the Picatinny rail asset with 1 µL HD drops treated with soapy water spray. The circle with error bars to the right of each swarm plot represent the geometric mean and 95% PI for the data.

The tabulated outputs of the results are provided in Table H-22. The methodology output B_2 (Figure A-1) of asset level performance as quantified by LD from the applied mass is provided. These data show that soapy water spray provided 0.35 ± 0.05 LD for the tested asset compared to the applied agent mass, and that the fit predicted 0.35 ± 0.001 LD.

Table H-22. M_{Asset} output for the tested asset and calculation variations for the Picatinny rail asset with 1 µL HD drops treated with soapy water spray.

Type	Geometric Mean (µg)	Upper Bound TI (µg)	Upper Bound 95% PI (µg)	μ_{Output} ($\log_{10}(M_{Asset}$ µg))	σ_{Output} ($\log_{10}(M_{Asset}$ µg))	LD ± CI (relative to applied mass)
Asset RA	1919.33	3032.73	2742.02	3.28	0.06	0.35 ± 0.05
Fit	1923.82	3982.08	4518.29	3.28	0.19	0.35 ± 0.00
$Indirect_{V=0.2µL}$	168.80	1291.32	1838.58	2.23	0.53	1.41 ± 0.01
$Indirect_{V=1µL}$	2913.64	3188.36	3238.64	3.46	0.02	0.17 ± 0.00

The accuracy ratio results (R) comparing each analysis technique to the tested asset are provided in Table H-23. For these data the fit regression provided the most accurate estimate of M_{Asset} with an $R_{Absolute}$ of 1.0. For this asset condition, the accuracy of the indirect fit using V=0.2 µL did not meet the acceptance criterion; the $R_{Absolute}$ value indicates that this calculation technique predicted a value 11.7× less than the tested asset RA. The other the fit methods produced results within the acceptance criterion range of $R_{Absolute} < 10$ (R is within 0.1–10).

For context on how well the PI and TI for the calculation techniques represent tested asset, ratio values for the quantity are also provided. If the upper bounds are compared to health-based requirements, then some measure of the accuracy should be considered. Here, the calculations are providing estimates much smaller than the indicated from the test data, likely due to the differences in degrees of freedom in the calculation. No criterion were established for upper bound estimates.

Table H-23. Accuracy ratio of the calculation variations for the Picatinny rail asset with 0.2 µL HD drops treated with soapy water spray.

Type	R: Geo Mean	$R_{Absolute}$: Geo Mean	R: Upper TI	R: Upper PI
Fit / Asset	**1.00**	1.00	1.31	1.65
$Indirect_{V=0.2µL}$ / Asset	**0.09**	11.37	0.43	0.67
$Indirect_{V=1µL}$ / Asset	**1.52**	1.52	1.05	1.18

Bold blue values are the accuracy ratio that should be within the validation criterion range of 0.1–10.
Red values indicate results are outside the acceptance criterion.

To build trust in the accuracy of using the geometric mean, a histogram of the results are provided in Figure H-17, the red line shows a fitted normal distribution to the log-transformed results. Ideally, the red line should have similar shape and magnitude as the histogram. Deviations of the red line from the histogram indicate that the log-normal distribution may not be an accurate description of the data, and quartile (i.e., non-parametric) analysis may be a better description of the results.

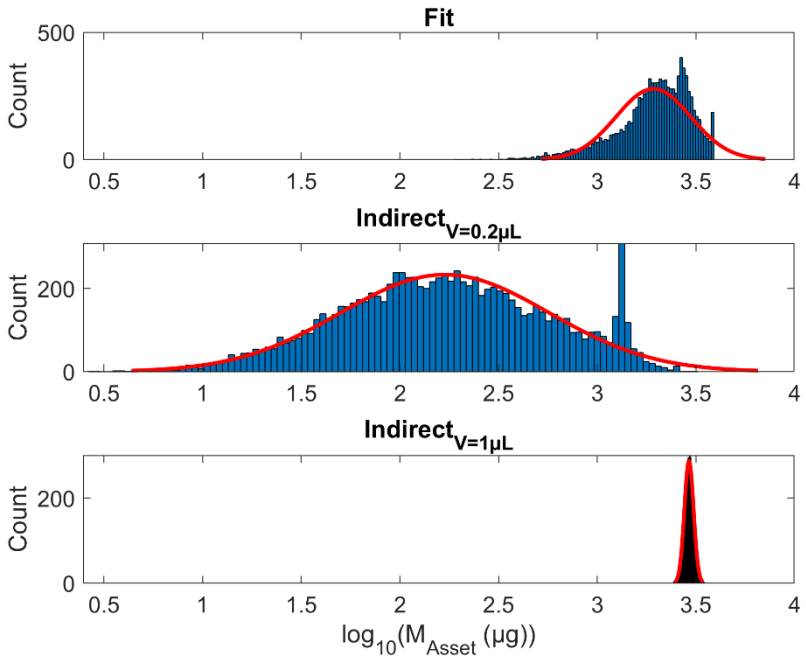


Figure H-17. Histogram of the M_{Asset} output fit to a log-normal distribution (red line), for the Picatinny rail asset with 1 µL HD drops treated with soapy water spray.

The geometric mean calculation assumes that the data are log-normal, with some deviations from normality seen in Figure H-17 (attributed to the below detection source terms). The MATLAB function *quantile* is used to identify the magnitude of M_{Asset} for specific quantiles such indicating that 95% of the data are less than an indicated value. The quantile output requires more samples than the seven replicates collected for the Asset RA data, thus only the MC output is presented in the quantile output (Table H-24).

Table H-24. Quantile analysis of the M_{Asset} output for the Picatinny rail asset with 1 µL HD drops treated with soapy water spray.

Quantile	Fit Analysis (µg)	Indirect$_{V=0.2µL}$ (µg)	Indirect$_{V=1µL}$ (µg)
25%	1562.332 (0.8x)	71.918 (0.4x)	2809.686 (1.0x)
Median 50%	2063.986 (1.0x)	166.965 (1.0x)	2912.451 (1.0x)
75%	2657.245 (1.3x)	397.528 (2.4x)	3022.695 (1.0x)
95%	3383.988 (1.6x)	1311.736 (7.9x)	3183.956 (1.1x)

Values in parenthesis are the ratio of the value to the median.

The MC calculation tracks the source of agent mass, enabling the attribution of which material-features contributed to the total M_{Asset} output. Figure H-18 provides the relative contribution for each feature type. For example, the steel 2 mil feature was typically responsible for 99.9% of the M_{Asset} mass, and the steel 20 mil feature was typically responsible for 0% of the M_{Asset} mass, and the steel-flat response provided a 0.02% contribution to the M_{Asset} mass. A similar analysis can be done without normalization to the M_{Asset} mass to indicate, for example, that typically 2062 µg of the 1923 µg ($M_{Asset,\ fit\ geometric\ mean}$) retained by the asset was attributed to the steel-2 mil feature. The use of median statistics for the material-feature contributions enabled the contribution value to be greater than the total asset geometric mean.

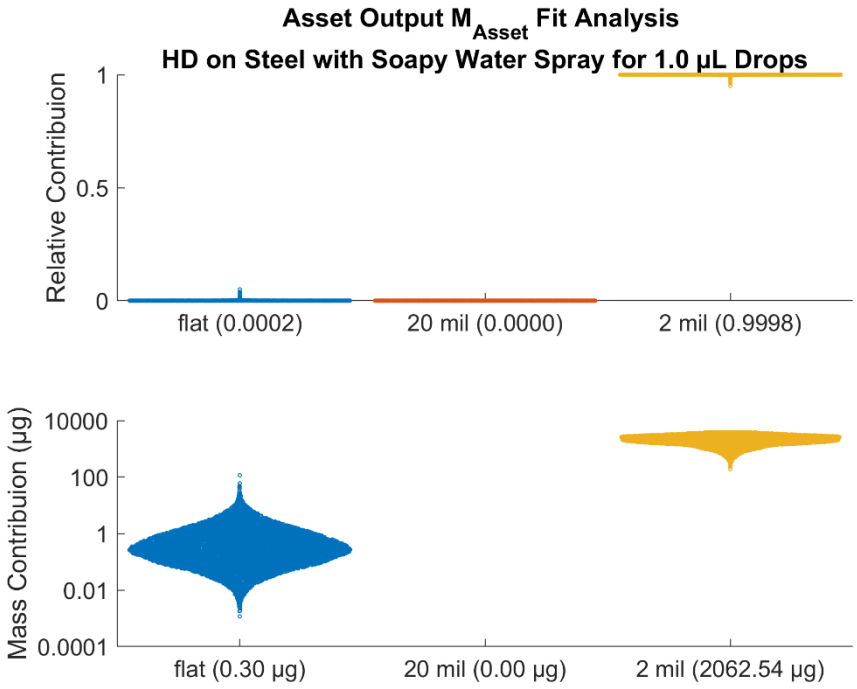


Figure H-18. Relative contribution and mass contribution graphs for each feature for the Picatinny rail asset with 0.2 µL HD drops treated with soapy water spray, values in parenthesis are the median response for the material-feature indicated.

The contribution calculations enable material attribution, here showing that the 2 mil shim panel was by far the major source of exposure. This relative contribution analysis also indicates that the accuracy of the of the M_{Asset} output is mostly influenced by the accuracy of the

calculation technique for the 2 mil shim as it is the dominant source. For example, poor accuracy of the flat panel feature would have negligible contribution to the M_{Asset} output.

These data have demonstrated the ability of the analysis techniques to scale-up laboratory panel data to represent a small asset contaminated with 1 µL drops and treated with soapy water spray. In this case, the extrapolation of the 0.1 µL data to represent 1 µL drops did not provide the desired scale-up accuracy. However, the other calculation techniques met the criterion of $R_{Absolute} < 10$.

DISTRIBUTION LIST

The following individuals and organizations were provided with one electronic copy of this report:

U.S. Combat Capabilities Development
Command Chemical Biological Center
(DEVCOM CBC)
CB Protection and Decontamination Division
Decontamination Sciences Branch
FCDF-CBR-PD
ATTN: Mantooth, B
Morrissey, K.
Caretti, D.

DEVCOM CBC
Technical Library
FCDF-CBR-L
ATTN: Foppiano, S.
Stein, J.

Defense Technical Information Center
ATTN: DTIC OA

Defense Threat Reduction Agency
DTRA-RF-IAR
J9-CBS
ATTN: Bass, C.
Lawson, G.

U.S. ARMY

U.S. ARMY COMBAT CAPABILITIES DEVELOPMENT COMMAND
CHEMICAL BIOLOGICAL CENTER