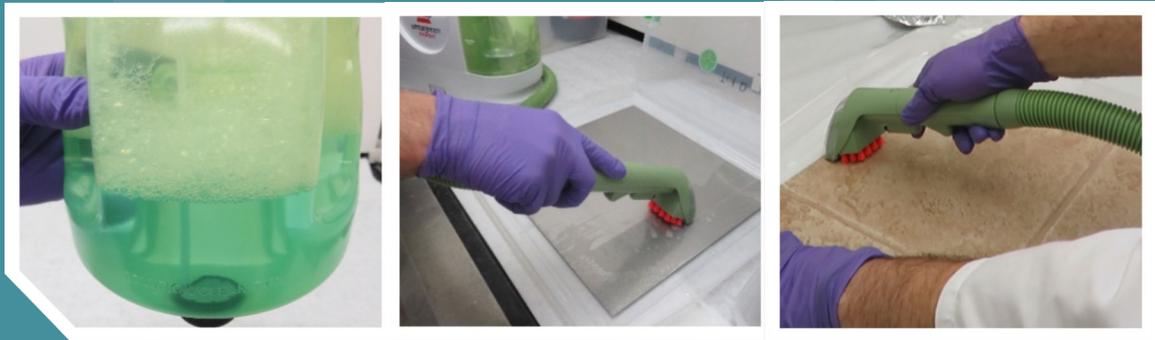


## Wet-Vacuum-Based Surface Sampling Method for Chemical Agents



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# **Wet-Vacuum-Based Surface Sampling Method for Chemical Agents**

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## Disclaimer

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## Acronyms and Abbreviations

2-CEPS	2-chloroethyl phenylsulfide
AEMD	Air and Energy Management Division (EPA)
ANOVA	analysis of variance
CC	continuous calibration
CF	conversion factor
cm	centimeter(s)
cm <sup>2</sup>	square centimeter(s)
cm <sup>3</sup>	cubic centimeter(s)
CMAD	Consequence Management and Advisory Division (EPA)
CRM	Certified Reference Material
CS	control spike
CT	contact time
CWA	chemical warfare agent
D	depth
DCM	dichloromethane
DCMD	Decontamination and Consequence Management Division (EPA)
DF	film thickness
DI	deionized
DLT	dirty [sample] liquid tank
DQI	data quality indicator
DUP	duplicate injection
EC	end check
EPA	U.S. Environmental Protection Agency
ft <sup>2</sup>	square foot
g	gram(s)
GC	gas chromatography
GC/FID	gas chromatography/flame ionization detector
GC/MS	gas chromatography/mass spectrometry
h	hour(s)
H	height
HD	sulfur mustard
HPLC	high performance liquid chromatography
HSRP	Homeland Security Research Program (EPA)
HT	holding time
ICAL	initial calibration
ICV	initial calibration verification
IPA	isopropyl alcohol
IR	infrared
IS	internal standard
ISO	International Organization for Standardization
L	liter(s)
LB	laboratory blank

LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LLE	liquid–liquid extraction
LOQ	limit of quantitation
LT	lapse time
m	meter(s)
M	molar
m <sup>2</sup>	square meter(s)
MAC	multiarea composite
mg	milligram(s)
µg	microgram(s)
min	minute(s)
mL	milliliter(s)
µL	microliter(s)
NHSRC	National Homeland Security Research Center (EPA)
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
NMAM	NIOSH Manual of Analytical Methods
OEM	Office of Emergency Management (EPA)
OLEM	Office of Land and Emergency Management (EPA)
ORD	Office of Research and Development (EPA)
OSL	Organic Support Laboratory
PB	procedural blank
PI	principal investigator
PW	plywood
QC	quality control
R <sup>2</sup>	coefficient of determination
RLV	reporting limit verification
RPD	relative percent difference
RSD	relative standard deviation
RTP	Research Triangle Park, North Carolina, USA
s	second(s)
SA	single [medium-size] area
SD	standard deviation
SEE	Senior Environmental Employee
SL	sampling liquid
slpm	standard liter(s) per minute
S/N	signal-to-noise ratio
SS	stainless steel
TAT	turnaround time
TC	test coupon
TEP	triethylphosphate
TIC	toxic industrial chemical
TOT	total operational time

VF	vinyl flooring
W	width
WT	wetting tank
X <sup>SE</sup>	sampling efficiency

## **Acknowledgments**

This research effort is part of the U.S. Environmental Protection Agency's (EPA's) Homeland Security Research Program (HSRP) to develop novel surface sampling approaches. Here, a wet-vacuum technology is considered for the sampling of various toxic industrial chemicals from contaminated building material surfaces. This methodology was optimized for sampling of chemicals with various solubility in water, using a discrete and a composite sample collection approach. The optimized wet-vacuum sampling technology, which can be applied to sample for chemicals over a large area, was also compared to standard wipe-based sampling methods. The results of this work would inform responders, governments, and health departments in their guidance development for sampling recommendations for medium to large surface areas contaminated with toxic chemicals.

This effort was directed by the principal investigator (PI) from the Office of Research and Development's (ORD's) National Homeland Security Research Center (NHSRC), with support of a project team consisting of staff from across EPA. The contributions of the following individuals have been a valued asset throughout this effort:

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## Executive Summary

A large-area accidental or intentional chemical release may require extensive environmental sampling for hazard characterization and mapping, and, after the decontamination is completed, may require reliable post-decontamination clearance sampling to assess if surface cleanup goals were met. Such complex environmental sampling may necessitate collection of large numbers of samples. Traditional sampling methods can significantly hinder the remediation process because they are time- and labor-intensive.

This study investigated a novel surface sampling approach based on the use of a commercially-available wet-vacuum cleaning method as a sampling mechanism. This novel surface sampling approach utilizes the ability of one piece of equipment to dispense a solution (the wetting agent) onto a surface and to vacuum the applied wetting agent, including any dissolved chemical, for collection into the same system. This approach was investigated for collection of various classes of chemicals from indoor building surfaces. Target chemicals with varying degrees of solubility in water were collected from nonporous to porous and permeable substrates using water or organic solvent wetting agents. The performance of an optimized wet-vacuum sampling method using a commercial off-the-shelf wet-vacuum unit was evaluated for multivariate (chemical and surface type, surface contamination level, and wetting agent type) sampling of medium-size (approximately 1000 cm<sup>2</sup>) and large-size (approximately 5000 cm<sup>2</sup>) areas and compared to existing wipe-based sampling methods and/or modifications thereof.

The main findings of this study are:

- Wet-vacuum-based methods, utilizing a commercially available cleaner and isopropyl alcohol (IPA) wetting solvent provide a better than 75% recovery for sampling of various classes of chemicals with varied solubility in water at tens of milligrams per square meter (mg/m<sup>2</sup>) surface concentrations.
- Wet-vacuum method performance is lower for collection of chemicals from semiporous materials (wood, vinyl), compared to nonporous materials, and for sampling of surfaces contaminated with lower microgram per square meter (µg/m<sup>2</sup>) contamination levels.
- The efficiency of aqueous wetting agent-based wet-vacuum sampling is affected by the solubility of the chemical in water. The addition of surfactant improves recovery of selected water-insoluble chemicals but generally does not improve the sampling efficiency at lower contaminant surface concentrations.
- In comparison to wipe-based sampling methods, the wet-vacuum methodology with IPA as a wetting solvent offers eight- to tenfold improvement in turnaround time needed to collect and prepare surface sample for analysis. The aqueous wetting agent methods offers an approximately twofold reduction of the turnaround time, as compared to wipe-based methodology.

Results indicate that the wet-vacuum sampling method was very efficient for collection of chemicals from nonporous substrates based upon only a fraction of the chemical mass recovered remaining on the surfaces post-sampling. However, the vacuum cleaner components were confirmed to contain the chemical after use suggesting the wet-vacuum units should not be considered for reuse and should be handled as contaminated waste.

The main limitations of this new technique are: (1) its limited applicability to sampling of highly porous surfaces (like wipe-based surface sampling) and (2) low sample recoveries when the level of the chemical surface contamination is significantly below the 1-10 mg/m<sup>2</sup> surface concentration level.

Depending on the surface decontamination threshold, this methodology may need to be further optimized for application to post-contamination sampling. Despite the significant reduction of turnaround time, as compared to conventional wipe-based sampling, the wet-vacuum method generates relatively expensive contaminated sampling equipment that cannot easily be reused or decontaminated. In addition to the cost of equipment (approximately \$120-\$140 per unit), the cost of proper disposal of post-sampling waste, including the wet-vacuum units, should be taken into consideration when utilizing commercial vacuum cleaner devices.

The optimized wet-vacuum sampling method as described here can be considered a good prospective addition to the wipe-based surface sampling methods when responding to a large-area chemical release or incident. Outcomes of the systematic testing of this surface sampling method provide field responders with additional tools to characterize large-area contamination following an accidental chemical release or terror incident.

## Table of Contents

Disclaimer .....	ii
Acronyms and Abbreviations .....	iii
Acknowledgments .....	vi
Executive Summary .....	vii
Figures .....	xi
Tables .....	xiii
1.0 Introduction .....	16
1.1 Project Objectives .....	16
2.0 Experimental Approach.....	18
2.1 Test Facility.....	18
2.2 Experimental Design .....	18
3.0 Materials and Methods.....	19
3.1 Test Materials .....	19
3.2 Chemicals .....	20
3.3 Contamination of Coupons .....	21
3.4 Test Setup .....	23
3.5 Wet-Vacuum Test Equipment.....	24
3.6 Surface Wetting Agents .....	25
3.6.1 Wetting Agent Application Methods .....	26
3.7 Method Development Tests.....	26
3.7.1 Extraction of target chemicals from water and water-surfactant solutions.....	26
3.7.2 Wipe-based methods for surface sampling of target chemicals.....	28
3.8 Surface Sampling Tests .....	29
3.8.1 Phase I: Selection of operational parameters for wet-vacuum sampling.....	29
3.8.2 Phase II: Application of commercial wet-vacuum systems for surface sampling .....	30
3.8.3 Phase III: Multivariate characterization of wet-vacuum-based sampling methods .....	31
3.8.3.1 Operational characterization of commercial wet-vacuum cleaner-based sampling .....	31
3.8.3.1.1 Medium-area sampling .....	31
3.8.3.1.2 Large-area sampling .....	32
3.9 Supplementary Surface Concentration Verification Tests .....	33
3.9.1 Verification of surface concentrations by direct extraction .....	33
3.10 Supplementary Tests .....	33
3.10.1 Operational time estimates .....	33
3.10.2 Flow and temperature profiles of wet-vacuum system .....	34
3.10.2.1 Measurement of vacuum airflow .....	34
3.10.2.2 Temperature measurement of wet-vacuum unit.....	34
4.0 Sampling and Analysis.....	35
4.1 Surface Sampling Methods.....	35
4.1.1 Medium-size area wet-vacuum sampling.....	35
4.1.2 Large-area wet-vacuum sampling .....	35
4.1.3 Small-area wipe-based sampling .....	37
4.2. Extraction Methods.....	38
4.2.1 Extraction of surface wipes .....	38

4.2.2 Extraction of water and water-based wetting agents .....	38
4.3 Preparation of Samples for Analysis .....	40
4.4 Instrumental Analyses .....	41
4.5 Data Reduction Procedures .....	44
4.5.1 Chemical concentration in sampling liquid calculations .....	44
4.5.2 Sampling efficiency calculations .....	45
5.0 Results .....	46
5.1 Analysis of Sampling Liquids .....	46
5.2 Proof of Concept and Initial Optimization of Surface Sampling Method – Phase I .....	48
5.3 Initial Evaluation of Commercial Wet-Vacuum Cleaners – Phase II .....	50
5.3.1 Comparison of commercial wet-vacuum cleaners .....	50
5.3.2 Optimization of the commercial wet-vacuum cleaner–based sampling .....	53
5.3.3 Wipe sampling optimization .....	53
5.4 Operational-scale Testing of Wet-Vacuum Sampling Efficiency – Phase III; Medium-size Area .....	54
5.4.1 Medium-size area sampling at default surface concentration .....	54
5.4.1.1 Residual chemical on wet-vacuumed surface and in wet-vacuumsing unit .....	55
5.4.2 Medium-size area sampling at low (below default) surface concentration .....	56
5.4.3 Surface concentration verification by direct extraction .....	58
5.4.4 Comparison of wet-vacuum sampling to wipe-based sampling methods .....	59
5.5 Operational-scale Testing of Wet-Vacuum Sampling Efficiency – Phase III; Large-size (Composite) Area .....	60
5.5.1 Operational considerations for large area composite sampling .....	60
5.6 Operational time comparisons .....	61
5.7 Supplementary Characterization Tests .....	62
5.7.1 Bissell wet-vacuum airflow .....	62
5.7.2 Wet-vacuum temperature profile .....	62
6.0 Quality Assurance and Quality Control .....	65
6.1 Test Equipment Calibration .....	65
6.2 Data Quality Results for Critical Measurements .....	65
7.0. Summary .....	67
8.0. References .....	69
Appendix A: Supporting Information .....	71
A-1 Wet-Vacuum Sampling Procedure .....	71
A-2 Parameters and Conditions for Instrumental analysis .....	73
A-3 Sample-Specific Test Results .....	76

## Figures

Figure 2-1. General experimental scheme of wet-vacuum sampling optimization and testing .....	18
Figure 3-1. Patterns for discrete droplet application of chemicals onto the medium-sized test surfaces: (a) wet-vacuum sampling, (b) wipe-based sampling, and (c) bulk extraction of positive control sample .....	22
Figure 3-2. Microdroplet application of chemical (a) and examples of wet (b) and dried out (or post-weathering) (c) chemical droplet pattern. Example shown is 2-CEPS on stainless steel .....	22
Figure 3-3. Clean test coupons prepared for testing: (a) stainless steel, (b) laminate flooring, (c) vinyl flooring, and (d) plywood .....	23
Figure 3-4. Assembled wet-vacuum sampling apparatus consisting of connector, Nalgene bottle, tubing, and custom-made nozzle .....	24
Figure 3-5. Commercial wet-vacuums used in this study: (a) Rug Doctor and (b) Bissell. ....	25
Figure 3-6. Stainless-steel surface before (a) and after (b) wipe sampling. ....	28
Figure 3-7. Surface spike controls ready for extraction. ....	33
Figure 4-1. Experimental design and sample flow for SA sampling. TC - test coupon; PB – procedural blank; CT – contact time; WT – wetting tank; DLT – dirty liquid tank .....	35
Figure 4-2. Experimental design and sample flow for MAC sampling. TC – test coupon; PB – procedural blank; CT – contact time; WT – wetting tank; DLT – dirty liquid tank .....	36
Figure 4-3. Experimental design and sample flow for small area sampling. TC – test coupon; PB – procedural blank; CT – contact time .....	37
Figure 4-4. Dawn Ultra® water SL immediately after conclusion of sampling (a), during settling phase in the dirty liquid tank (b), and settled liquid (aliquot in beaker) ready for LLE (b). ....	39
Figure 4-5. Drying of the SL extract .....	40
Figure 5-1. Results from LLE of TEP and 2-CEPS in control samples of aqueous sample liquids; (a) SuperSoap®-water LLE, (b) Dawn Ultra®-water LLE, (c) water LLE; HT, analytical HT defined as from control sample preparation to extraction. ....	47
Figure 5-2. Selection of operational parameters of laboratory-scale wet-vacuum sampling. (a) Selection of wetting solvent, (b) selection of lapse time for IPA, and (c) results of wet-vacuum sampling of various test surfaces using 50 mL IPA and LT = 10 s. ....	49
Figure 5-3. Results of the initial evaluation of commercial vacuum cleaners for wet-vacuum sampling of various test surfaces; sampling was performed using 50 mL of water, LT=10 s; material types: stainless steel (a); laminate flooring (b); vinyl flooring (c) .....	51
Figure 5-4. Results of the initial evaluation of commercial vacuum cleaners for wet-vacuum sampling of various test surfaces as a function of the solubility of the chemical in water .....	52
Figure 5-5. Comparison of sampling efficiency of SA wet-vacuum and wipe-based method for 2-CEPS (a) and TEP (b); WS – wipe sampling, WV – wet-vacuum sampling .....	59
Figure 5-6. Comparison of total operational time for wet-vacuum and wipe-based sampling methods; WS – wipe sampling; WV – wet-vacuum sampling .....	61
Figure 5-7. Airflow monitoring of Bissell unit simulated large-area sampling; flow rate is reported at US EPA standard ambient sampling conditions of 101.325 kPa and 25 °C; slpm – standard liters per minute .....	62
Figure 5-8. Temperature monitoring of Bissell exhaust during simulated sampling. Sampling performed under the chemical hood. (a) start of vacuum sampling (t = 0 min); (b) after 1 min of vacuum sampling (t = 1	

min); (c) after 2 min of sampling (t = 2 min); (d) after 3 min of sampling (t = 3 min); (e) at the end of sampling (t = 3 min 24 s); f, 15 min after sampling ended (t = 19 min 24 s)..... 63

Figure 5-9. Temperature in various zones of Bissell vacuum cleaner after 20 min sampling using water. (a) Central system temperature near motor; (b) Liquid waste collection tank temperature..... 64

Figure A-1 Spray nozzle (A) and spray trigger (B) locations ..... 72

## Tables

Table 3-1. Specifications of Building Materials .....	19
Table 3-2. Physicochemical Properties and Functional Surrogate Classification of Target Chemicals .....	20
Table 3-3. Analytical Standards for Target Chemicals .....	21
Table 3-4. Chemical Reagents .....	21
Table 3-5. Surface Wetting Agents for Wet-Vacuum Sampling .....	26
Table 3-6. Experimental Parameters of Original and Modified Reference Methods for Wipe Sampling and Extraction of Wipes .....	28
Table 3-7. Experimental Parameters of Phase I Testing .....	29
Table 3-8. Experimental Parameters of Phase II Commercial Wet-Vacuum Cleaner Testing – Initial Evaluation of Rug Doctor and Bissell Vacuum Cleaners .....	30
Table 3-9. Experimental Parameters of Phase II Commercial Vacuum Cleaner Testing for Improved Recovery of Organic Solvent-based Wetting Agent .....	30
Table 3-10. Experimental Parameters of Phase III Multivariate Characterization of Commercial Vacuum Cleaner Testing – Single Medium-Area Sampling .....	32
Table 3-11. Experimental Parameters of Phase III Multivariate Characterization of Commercial Vacuum Cleaner Testing – Large-Area Composite Sampling .....	32
Table 4-1. Chemical-Specific Experimental Conditions for Extractions of Aqueous SLs .....	39
Table 4-2. Instrumental Methods Used for Analysis of Target Analytes .....	41
Table 4-3. QC Checks for Instrumental Analyses Performed by External Laboratory .....	42
Table 4-4. Initial and Continuing Laboratory Proficiency Results .....	43
Table 5-1. Results for Wipe Sampling and Extraction Optimization Tests for 2-CEPS and TEP .....	53
Table 5-2. Test Results of Medium-Sized Area Wet-Vacuum Sampling of 2-CEPS from a Nonporous and Permeable Material at the Default Surface Concentration .....	54
Table 5-3. Test Results of Medium-Sized Area Wet-Vacuum Sampling of TEP from Nonporous and a Permeable Material .....	55
Table 5-4. Test Results of Medium-Sized Area Wet-Vacuum Sampling of 2-CEPS at Low Concentrations .....	56
Table 5-5. Test Results of Medium-Sized Area Wet-Vacuum Sampling of TEP at Low Concentrations .....	57
Table 5-6. Test Results for Direct Extraction of 2-CEPS from Stainless Steel .....	58
Table 5-7. Test Results for Direct Extraction of TEP from Stainless steel .....	58
Table 5-8. Test Results of Large-Sized Area Wet-Vacuum Sampling of 2-CEPS and TEP from Stainless-steel Material (n=3) .....	60
Table 6-1. Instrument Calibration Frequency .....	65
Table 6-2. Acceptance Criteria for Critical Measurements and Corresponding Test Results .....	66
Table A-1. GC/FID Analyses of Nitrobenzene with Modified NIOSH* 2005 (EMSL Analytical, Inc.) .....	73
Table A-2. GC/FID Analyses of Phenol Modified NIOSH Method 2546 (EMSL Analytical, Inc.) .....	73
Table A-3. GC/FID Analyses of TEP Using Modified NIOSH 5034 (EMSL Analytical, Inc.) .....	74
Table A-4. GC/FID Analyses of TEP Modified NIOSH 5038 (EMSL Analytical, Inc.) .....	74
Table A-5. GC/MS Analyses of TEP and 2-CEPS Samples in IPA (EPA OSL) .....	75
Table A-6. GC/MS Analyses of TEP and 2-CEPS in Hexane (EPA OSL) .....	75

Table A-7. Liquid–Liquid Extraction Efficiency of Selected Chemicals from Wetting Agents .....	76
Table A-8. Results of Direct Analysis of Phenol in Various Wetting Agents .....	77
Table A-9. Results of Direct Analysis of TEP in IPA .....	77
Table A-10. Results of Direct Analysis of 2-CEPS in IPA.....	77
Table A-11. Phase I Operational Parameter Optimization – Selection of Wetting Agent for Sampling of Phenol from Nonporous Reference Material.....	78
Table A-12. Phase I Operational Parameters Optimization – Selection of Wetting Agent for Sampling of Nitrobenzene from Nonporous Reference Material .....	79
Table A-13. Phase I Operational Parameters Optimization – Selection of Wetting Agent for Sampling of TEP from Nonporous Reference Material .....	80
Table A-14. Phase I Operational Parameters Optimization – Selection of the Wetting Agent Surface Contact Time (Lapse Time) for IPA Sampling of Phenol from Nonporous Reference Material.....	81
Table A-15. Phase I Operational Parameters Optimization – Selection of the Wetting Agent Surface Contact Time (Lapse Time) for IPA Sampling of Nitrobenzene from Nonporous Reference Material .....	82
Table A-16. Phase I Operational Parameters Optimization – Selection of the Wetting Agent Surface Contact Time (Lapse Time) for IPA Sampling of TEP from Nonporous Reference Material .....	83
Table A-17. Phase I Operational Parameters Optimization – Evaluation of IPA Wetting Agent for Sampling of Phenol from Semiporous and Porous Materials .....	84
Table A-18. Phase I Operational Parameters Optimization – Evaluation of IPA Wetting Agent for Sampling of Nitrobenzene from Semiporous and Porous Materials.....	85
Table A-19. Phase I Operational Parameters Optimization – Evaluation of IPA Wetting Agent for Sampling of TEP from Semiporous and Porous Materials.....	86
Table A-20. Phase II Evaluation of Commercial Wet-Vacuums for Sampling of Phenol from Stainless Steel .....	87
Table A-21. Phase II Evaluation of Commercial Wet-Vacuums for Sampling of Phenol from Laminate Flooring.....	88
Table A-22. Phase II Evaluation of Commercial Wet-Vacuums for Sampling of Phenol from Vinyl Flooring .....	89
Table A-23. Phase II Evaluation of Commercial Wet-Vacuums for Sampling of Nitrobenzene from Stainless Steel.....	90
Table A-24. Phase II Evaluation of Commercial Wet-Vacuums for Sampling of Nitrobenzene from Laminate Flooring.....	91
Table A-25. Phase II Evaluation of Commercial Wet-Vacuums for Sampling of Nitrobenzene from Vinyl Flooring.....	92
Table A-26. Phase II Evaluation of Commercial Wet-Vacuums for Sampling of TEP from Stainless Steel Coupon .....	93
Table A-27. Phase II Evaluation of Commercial Wet-Vacuums for Sampling of TEP from Laminate Flooring .....	94
Table A-28. Phase II Evaluation of Commercial Wet-Vacuums for Sampling of TEP from Vinyl Flooring.....	95
Table A-29. Phase II Optimization of Wetting Solvent and Chemical Recovery Using Bissell Little Green ProHeat Wet-Vacuum – Phenol on Stainless-steel Coupon .....	96
Table A-30. Phase II Optimization of Wetting Solvent and Chemical Recovery Using IPA and Bissell Little Green ProHeat Wet-Vacuum – TEP on Stainless-steel Coupon .....	97

Table A-31. Phase III Optimized IPA Wet-Vacuum Method Performance for 100% Surface Concentration Reference Material Sampling Baseline of 2-CEPS, including Mass Balance Tests..... 98

Table A-32. Phase III Optimized TEP Wet-Vacuum Method Performance for 100% Surface Concentration Reference Material Sampling Baseline of TEP, including Mass Balance Tests..... 99

## 1.0 Introduction

In the event of a large-area chemical incident, many environmental samples may be collected, preserved, and analyzed to successfully identify and map the hazard and select the appropriate decontamination strategy. Characterization and clearance sampling procedures and associated analyses can present a high fiscal and logistical burden to responsible agencies. This is especially the case when using a statistical sampling design as such design often requires the generation of large numbers of samples to achieve reasonable confidence in, for example, hotspot detection or, post-decontamination, the cleanliness of an area. Limited laboratory capacity and costly analysis, especially for chemical warfare agents (CWAs), make statistically-based sampling strategies difficult to implement as part of the remediation and clearance process.

Pre- and post-decontamination environmental sampling of chemical agents typically involves the use of surface wipes for collection of a contaminant from a single discrete location (often an area less than one square foot) or from multiple locations combined into one sample (composite sampling), followed by extraction of the sampling medium and instrumental analysis of extracts [1-3]. Operationally and logistically, it is often challenging to sample, extract, and analyze large quantities of such environmental samples quickly, especially when sample cleanup, fractionation, or solvent exchange is performed. Aside from revising sampling strategies, for example, via direct *in situ* analysis, composite multilocation sampling may be an alternative approach that can present many advantages such as reduced response sampling time, fewer samples to process, and coverage of large sampling areas that would improve detection of widespread contamination [1-4]. A direct collection of the surface contamination into a solvent can reduce sampling costs and efforts by eliminating the wipe extraction and preparation steps for such an extract prior to analysis by readily available instrumental techniques (for example, gas chromatography/mass spectrometry [GC/MS]). Direct collection of the surface contamination into a solvent also eliminates the use of a wipe material, which sometimes requires a pre-cleaning cycle and additional Quality Assurance/Quality Control steps.

In this study, a wet-vacuum cleaning method was evaluated for the surface sampling of nonporous to porous building surfaces spiked with chemicals exhibiting differing degrees of water solubility. The wet-vacuum sampling efficiency was studied using a prototype and two commercially available devices. A relatively mild but moderately volatile organic solvent (isopropyl alcohol [IPA]), water, and water with surfactant solutions were evaluated as the surface wetting and sampling agents. The optimized wet-vacuum sampling method efficiency was compared to traditional wipe-based sampling methods and/or modifications thereof. Research outcomes from this study benefit responders as it will allow them to consider high-capacity composite sampling approaches for the sampling of chemical agents from environmental surfaces.

### 1.1 Project Objectives

The purpose of this project was to provide responding agencies with information on the effectiveness of a wet-vacuum sampling approach for the collection of toxic industrial chemicals (TICs) and CWA surrogates deposited on building materials with different porosities. This research is part of the U.S. Environmental Protection Agency's (EPA's) Homeland Security Research Program (HSRP) efforts to develop time- and cost-effective high-capacity sampling approaches to be used in large areas contaminated with chemical agents. This new methodology uses commercial, off-the-shelf cleaning equipment (handheld wet-dry vacuum units) for composite sample collection and composite sample analysis with various wetting

solutions and solvents that are pertinent to field sampling in response to an accidental chemical release or chemical terror incident.

The primary objectives of this research were to:

- Develop a medium- to large-area surface sampling method that uses an easily accessible, off-the-shelf device and surface sampling agents that can be used in the field.
- Determine the feasibility and effectiveness of the proposed method by measuring medium- and large-area surface sampling efficiencies through measurement of the mass of chemical deposited on the surface as a function of chemical surface loading, wetting agent, and material type.
- Compare wet-vacuum surface sampling efficiencies to the efficiency of conventional (wipe-based) sampling methods. These comparisons were performed in conjunction with initial estimates of differences in operational time and sample turnaround time between these two types of sampling approaches.

The secondary objectives were to:

- Provide information on residual chemical concentrations that remain inside the wet-vacuum system after sampling.
- Provide information on residual chemical concentrations that remain on surfaces *after* wet-vacuum sampling.
- Provide initial information on the general wet-vacuum operational parameters that can be relevant to field applications (e.g., temperature profile, flow, potential for reuse).

Outcomes of the systematic testing of this surface sampling methodology will provide field responders with additional tools to characterize large-area contamination following an accidental chemical release or terror incident. This information provides the scientific basis for a potentially significant reduction in the time and cost of post-decontamination sampling events of selected chemical agents. This project supports the strategic goals of improving the capability to respond to chemical incidents that affect buildings and the outdoor environments as part of EPA's Homeland Security Research Program.

## 2.0 Experimental Approach

### 2.1 Test Facility

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

### 2.2 Experimental Design

This study was performed in three consecutive phases. The first proof-of-concept phase (Phase I) consisted of the bench-scale determination of basic test parameters such as the selection of type and volume of the wetting solvent and evaluation of material-specific surface lapse times. This surface lapse time is defined here as the time between application of the wetting solvent and the wet-vacuum of this agent. Phase II involved the wet-vacuum method optimization for improved recovery of the selected wetting solvent and target chemicals using a single discrete area sampling. Phase III involved the operational-scale deployment of the optimized method for sampling of various surface concentrations of TICs using organic solvent and water-based wetting agents for medium-sized (929 cm<sup>2</sup>) and a composite sampled area of five medium-sized areas (5 x 929 cm<sup>2</sup>). The general experimental approaches for each phase of this work are shown in Figure 2-1. Details of each experimental phase are described in Sections 3.8.1 through 3.8.3.

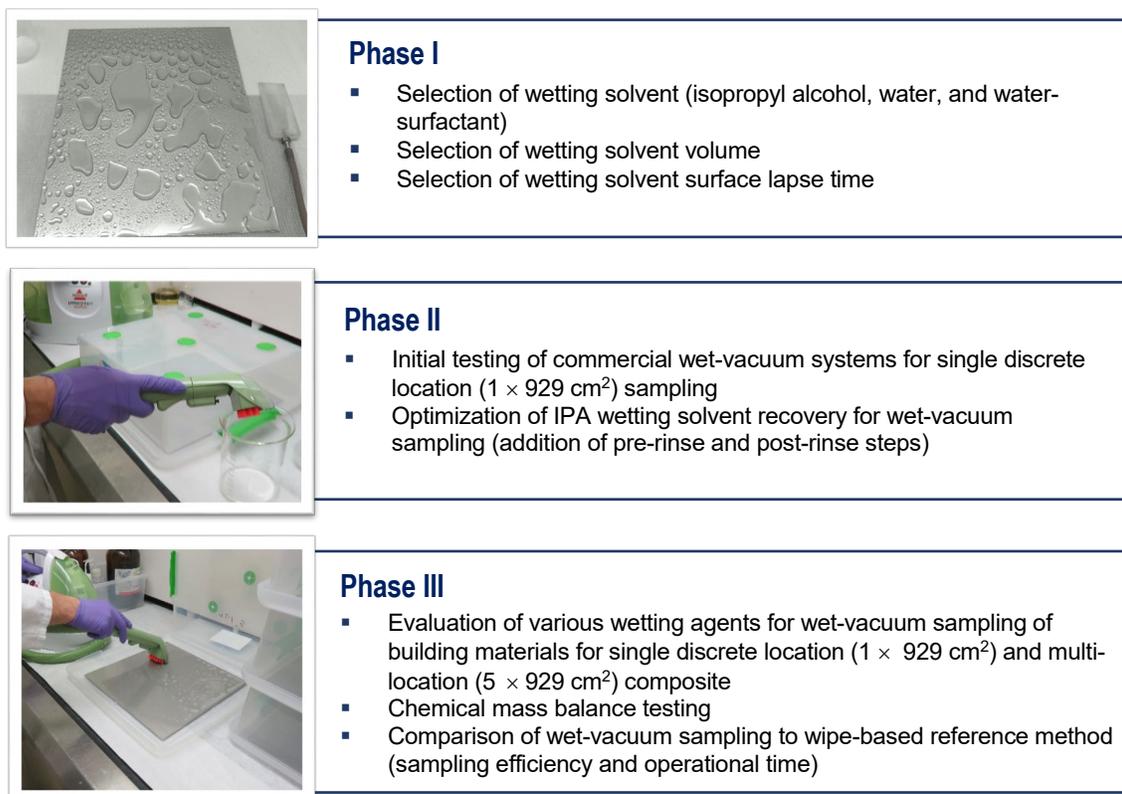


Figure 2-1. General experimental scheme of wet-vacuum sampling optimization and testing

### 3.0 Materials and Methods

An ideal surface sampling methodology should generate accurate and reproducible data when used on environmental surfaces for which it was designed. The analytical data must meet the quantitative and qualitative detection criteria that are relevant to the applied method. For this study, the sampling method was designed for the defensible determination of a surface chemical contamination level. Wet-vacuum sampling approaches may consider horizontal surfaces such as flooring materials or countertops or vertical surfaces such as walls. In this project, the focus was on horizontal surfaces with flooring materials as the most prevailing material type that would require sampling to characterize the (residual) level of chemical contamination following a contamination event.

#### 3.1 Test Materials

Several types of building materials with different porosities and permeabilities were selected for evaluation of sampling procedures (Table 3-1). Multipurpose stainless steel was a surrogate for smooth nonpermeable building surfaces (e.g., sinks, countertops). Due to its inertness, low porosity, and excellent corrosion resistance [5], stainless steel was also used as a good reference material for optimization of sampling approaches. Three types of flooring (vinyl, laminate and plywood) were selected as representative of semipermeable (laminate) and permeable (vinyl and plywood) building materials. The building material specifications are given in Table 3-1. A low efficiency of wetting solvent collection was observed when sampling target chemicals on plywood during the Phase I method development (results are described in Section 5.2). Hence, this material was not used in the subsequent wet-vacuum sampling optimization and testing (Phases II and III).

**Table 3-1. Specifications of Building Materials**

Material	Description	Manufacturer/ Supplier Name/Location/Country	Coupon Size, L x W (cm)*	Material Preparation
Stainless steel	Multipurpose stainless steel (1.2 x 1.2 m), type 304, #2B mill (unpolished), 0.091 cm thick	McMaster-Carr Douglasville, GA, USA	35.56 × 35.56	Cut into coupons and remove any lubricant/grease from shearing with acetone. Wipe dry.  Immediately before use, remove particles and dust by wiping clean with acetone and then water. Wipe dry.
Vinyl flooring	2.4 x 3.6 m Casa Grande beige precut sheet vinyl, residential grade, low gloss, stain resistant, scratch resistant, 0.050 cm	Tarkett, Inc. Whitehall, PA, USA	35.56 × 35.56	Cut into coupons. Remove particles by wiping clean with water and wipe dry.
Laminate flooring	Project Source 20.5 x 120 cm natural oak smooth laminate wood planks	Clarion Laminates LLC Shipperville, PA, USA	35.56 × 35.56	Attach to the plywood base and cut into coupons. Remove particles by wiping clean with water and wipe dry.
Plywood	1.2 x 2.4 m Plytanium® untreated pine plywood, 1.27 cm thick	Georgia-Pacific Building Products Atlanta, GA, USA	35.56 × 35.56	Cut into coupons. Remove particles by wiping clean with water and wipe dry.

\*Actual effective test area was center 30.48 cm × 30.48 cm (929 cm<sup>2</sup>) or 12 inches (") x 12".

Stainless-steel and vinyl coupons were cut to the correct length and width from larger sheets using heavy-duty power hydraulic shears. Plywood panels were precut to desired dimensions using a table saw.

All coupons were cleaned prior to testing using the procedures described in Table 3-1. Test boxes (dimensions: 45.72 cm × 45.72 cm × 15.88 cm; Stor-N-Slide square box with lid, product no. 491530; IRIS USA, Inc., Pleasant Prairie, WI, USA) that held one individual coupon were cleaned using laboratory-grade detergent solution, wiped with acetone and water, and wiped dry.

### 3.2 Chemicals

The target chemicals used in this study were selected using two criteria: (1) the chemicals selected must be representative of a wide range of water solubility, from very slightly soluble (range: grams per liter or less) to very soluble (range: hundreds of grams per liter to completely miscible) and (2) the chemicals selected must be representative of various classes of TICs, including CWA surrogates.

The relevant physical and chemical properties and functional surrogate classification of target chemicals are listed in Table 3-2. Information on the neat chemical standard sourcing is summarized in Table 3-3. Information on internal standard and surrogate compound analytical standards used in this study is given in Section 4.4. Other chemical reagents used as wetting solvents, drying agents or extraction solvents are listed in Table 3-4.

**Table 3-2. Physicochemical Properties and Functional Surrogate Classification of Target Chemicals**

Target Chemical	2-Chloroethyl phenyl sulfide (2-CEPS)	Nitrobenzene	Phenol	Triethyl phosphate (TEP)
CAS registry number	5535-49-9	98-95-3	108-95-2	78-40-0
Other common names and/or abbreviations	2-CEPS	Nitrobenzol	Hydroxybenzene	TEP
<b>Physicochemical Properties*</b>				
Molecular weight	172.7	123.1	94.1	182.2
Chemical formula	C <sub>8</sub> H <sub>9</sub> ClS	C <sub>6</sub> H <sub>5</sub> NO <sub>2</sub>	C <sub>6</sub> H <sub>6</sub> O	C <sub>6</sub> H <sub>15</sub> O <sub>4</sub> P
Density (g/cm <sup>3</sup> )	1.174	1.20	1.06	1.072
Vapor pressure (Pa)†	2.53	40	46	52
Solubility in water (grams (g)/liter (L))†	0.084	2.1†	83	500
<b>Water Solubility and Functional Surrogate Class</b>				
Functional surrogate class	Surrogate of CWA (sulfur mustard [HD]) [6,7]	Toxic industrial chemical	Toxic industrial chemical	Toxic industrial chemical
*Data from <a href="https://pubchem.ncbi.nlm.nih.gov">https://pubchem.ncbi.nlm.nih.gov</a> ; <a href="https://www.sigmaaldrich.com">https://www.sigmaaldrich.com</a>				
†At 25 °C				

**Table 3-3. Analytical Standards for Target Chemicals**

Target Chemical	2-Chloroethyl phenyl sulfide	Nitrobenzene	Phenol	Triethyl phosphate
Manufacturer	Sigma Aldrich St. Louis, MO, USA	Sigma-Aldrich St. Louis, MO, USA	Sigma-Aldrich St. Louis, MO, USA	Sigma-Aldrich St. Louis, MO, USA
Product No.	417602-5ML	48547	P9346-100mL	538728-1L
Purity, %	98	100	≥89.0	≥99.8

**Table 3-4. Chemical Reagents**

Chemical Reagent	Purity/Grade	Product No.	Manufacturer
Sodium sulfate cartridge	n/a	12131033	Agilent Technologies, Santa Clara, CA, USA
Sodium chloride	99.7%	S271-500	Fisher Scientific, Fair Lawn, NJ USA
Sodium chloride	99.0%	VW6430-1	VWR Scientific Products, West Chester, PA, USA
1 molar (M) Sodium phosphate buffer pH 7.0	n/a	P2070	Teknova, Hollister, CA, USA
Hexane*	ACS/HPLC†	H303-4	Fisher Scientific, Fair Lawn, NJ, USA
Dichloromethane	ACS/HPLC	300-4	Honeywell International Inc., Muskegon, MI, USA
IPA	ACS Plus	A416-4	Fisher Scientific, Fair Lawn, NJ, USA

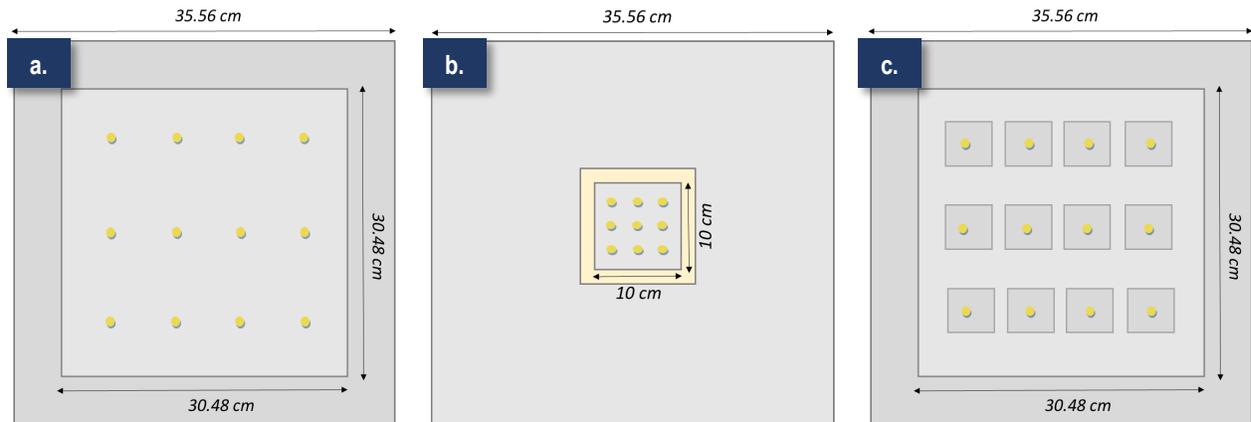
\*Mixture, as purchased, of *n*-hexane (45-60%), hexane (-isomers) (15-40%), and cyclohexane (3%).  
†HPLC - High performance liquid chromatography.

The target surface chemical concentrations varied in this study. Most wet-vacuum and wipe-based sampling tests were performed at a chemical surface concentration challenge level of approximately 300 milligrams (mg) per square meter (m<sup>2</sup>), which is equivalent to 26-29 mg per medium-sized coupon. For these tests, the central test area of each medium-sized coupon (30.48 cm × 30.48 cm, or 929 cm<sup>2</sup>) was contaminated with twelve uniformly distributed (2 µL) droplets of neat chemical. For low surface concentration and wipe-based (reference method) sampling tests, the coupons were contaminated at lower levels down to 1% of the default contamination level (26-29 mg/coupon of 929 cm<sup>2</sup>) using spiking solutions of the target chemical prepared by dissolving the neat chemical in an appropriate organic solvent. Each spiking solution was mixed using a vortex mixer and then via sonication for approximately 30 seconds. The accuracy and precision of the spiked test area was tested along with each experimental batch by analysis of control spike (CS) samples where the same amount of chemical that was applied to the surface was spiked directly into the extraction solvent.

### 3.3 Contamination of Coupons

Chemical solutions were applied to test coupons (TCs) using a discrete droplet (micro)application method. Prior to chemical application, each 35.56 cm × 35.56 cm TC was placed in a precleaned test box. Chemical solutions were then applied to the coupons under room temperature conditions within a chemical safety hood using a separate tip-programmable, electronic, repeatable pipette (Eppendorf Repeater Plus Single Channel Repeater Pipette, Eppendorf AG, Hamburg, Germany; product no. 22260201) and a precleaned stainless-steel spiking template placed over the coupon surface. For wet-vacuum testing, 12 droplets (2 microliter (µL) volume each) were applied within the 30.48 cm × 30.48 cm test area following the pattern shown in Figure 3-1a. For wipe-based sampling method evaluation, nine droplets of spiking solution were applied onto the 10 cm × 10 cm central part of the medium-sized coupon (Figure 3-1b). For positive

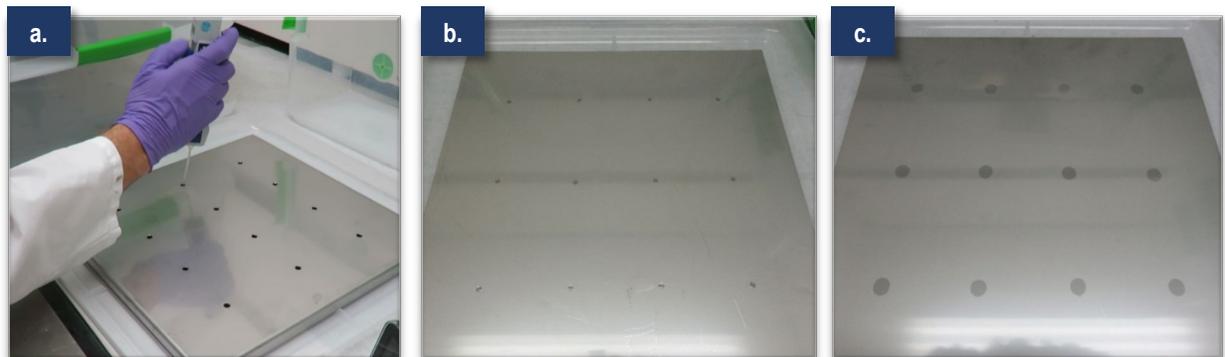
control testing, a composite sample comprising 12 sub-coupons of the same material was contaminated with a discrete droplet spike delivered onto each small coupon (Figure 3-1c).



**Figure 3-1. Patterns for discrete droplet application of chemicals onto the medium-sized test surfaces: (a) wet-vacuum sampling, (b) wipe-based sampling, and (c) bulk extraction of positive control sample**

After chemical application, the boxes were closed to allow a 30-minute simulated weathering or contact time; weathering was performed in the chemical hood under normal ambient laboratory conditions. Coupons were stored in the closed test boxes during the simulated weathering pre-decontamination phase to reduce possible evaporation of chemicals due to the high air-flow conditions inside the chemical safety hood.

Figure 3-2 shows (a) the spiking procedure, (b) examples of the chemical droplet pattern immediately after spiking, and (c) dried out (or post-weathering; contact time = 30 min) chemical prior to sampling. The example shown is 2-CEPS on stainless steel.

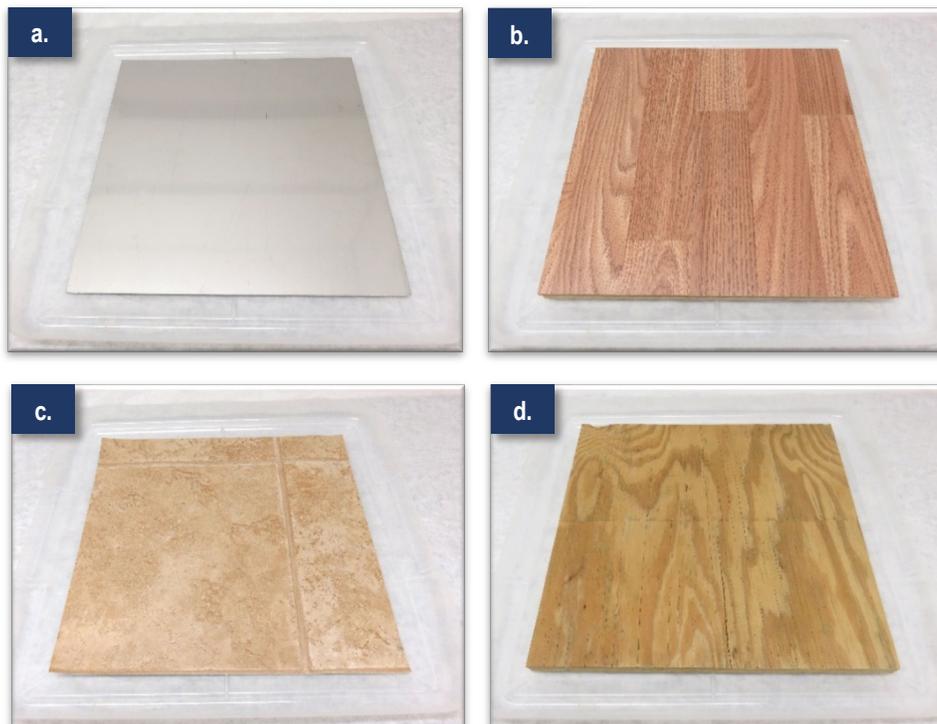


**Figure 3-2. Microdroplet application of chemical (a) and examples of wet (b) and dried out (or post-weathering) (c) chemical droplet pattern. Example shown is 2-CEPS on stainless steel**

The accuracy and precision of spiking solution preparation was assessed for each experimental batch by analysis of control spike samples.

### 3.4 Test Setup

All proof-of-concept and methodology optimization tests (Phases I and II, Sections 3.8.1 and 3.8.2) and simulated field sampling operational-scale experiments (Phase III, Section 3.8.3) were performed within a chemical safety hood. Each contaminated coupon was placed and handled in a precleaned test box. Figure 3-3 shows examples of clean stainless-steel, laminate flooring, vinyl flooring, and plywood coupons readied for testing.



**Figure 3-3. Clean test coupons prepared for testing: (a) stainless steel, (b) laminate flooring, (c) vinyl flooring, and (d) plywood**

Experimental details of the sampling approaches that were tested are given in Section 4.1.

### 3.5 Wet-Vacuum Test Equipment

Wet-vacuum sampling method laboratory-scale evaluations were performed using a custom-made wet-vacuum apparatus made from off-the-shelf components and two different commercial wet-dry vacuum cleaning units (hereafter referred to as wet-vacuums).

The custom-made sampling apparatus consisted of a Nalgene™ 500-milliliter (mL) wide-mouth polypropylene bottle (Thermo Scientific, Waltham, MA, USA) equipped with a wet-vacuum adapter. Samples were collected using a custom-made acrylic flat nozzle. The nozzle was connected to the wet-vacuum bottle via 1 meter (m) of latex tubing with a cord-grip fitting. Figure 3-4 shows the wet-vacuum apparatus readied for testing. The slit nozzle opening was approximately 1 mm (height) x 42 mm (width). A self-contained service vacuum (OmegaPlus Vacuum Cleaner [Atrix International Inc., Burnsville, MN USA]) pump was used to pull the liquid sample into the bottle. A high-efficiency particulate air filter (Atrix International Inc., Burnsville, MN USA) was included downstream from the bottle to protect the pump.



**Figure 3-4. Assembled wet-vacuum sampling apparatus consisting of connector, Nalgene bottle, tubing, and custom-made nozzle**

A new Nalgene bottle with new tubing was used to collect each sample to avoid cross-contamination. Grip fittings, wet-vacuum adapters, and nozzles were cleaned by soaking in an activated hydrogen peroxide-based decontamination solution (EasyDecon® DF200, Intelagard, Lafayette, CO, USA). After overnight soaking, all parts were triple-rinsed with deionized (DI) water. Following the rinse step, the reusable components were air dried.

After the method optimization using the wet-vacuum sampling prototype device (Phase I, Section 3.8.1) was completed, two types of commercial hand-held wet-vacuums were evaluated for sampling chemicals from different types of building surfaces. The selected units are readily available at hardware stores. Other models exist that have similar properties. The critical parameters were the need for a single unit that can dispense the wetting solvent and vacuum this solvent after contact with a surface with the ability to define the time between solvent application and vacuuming. The requirement for the minimal solvent / wetting agent collection volume was one liter. The following two models (price \$120-\$140 per unit) met these criteria:

- **Rug Doctor Portable Spot Cleaner** (model no. 93300, Rug Doctor, Inc., Plano, TX) (Figure 3-5a) is a high-suction power portable carpet cleaner equipped with a handheld motorized brush that moves 1200 times per minute and is specifically designed to deep-clean carpets and break down stains without the use of heated water or steam. The maximum capacity of both the clean

(wetting) liquid and dirty (cleaning waste) liquid tanks is 1890 mL. The unit weighs approximately 9.1 kg; dimensions are 45 (H) cm x 45 (W) cm x 32 (D) cm.

- **Bissell Little Green ProHeat Compact MultiPurpose Carpet Cleaner** (model no. 14259, Bissell Corp., Grand Rapids, MI) (Figure 3-5b) is a high-suction power portable carpet cleaner holding separate tanks for wetting liquid and cleaning waste, with an approximate capacity of 1420 mL each. The unit is equipped with a built-in (optional-use) water heater for enhanced cleaning of tough surface stains. The cleaning liquid heater option was not used during this study. The unit weighs approximately 6.4 kg; dimensions are 32 cm height (H) cm x 44 cm width (W) x 21 cm depth (D).



Figure 3-5. Commercial wet-vacuums used in this study: (a) Rug Doctor and (b) Bissell.

### 3.6 Surface Wetting Agents

The surface wetting liquid was used to wet the coupon surface and, after the appropriate elapsed time, was removed from the coupon surface using the vacuum technique. Surfaces were wetted via a spray nozzle (commercial units) or separate spray bottle (for prototype unit). The wetting liquids considered in this study represented two general classes: (1) a mild organic solvent, and (2) water-based agents. The organic solvent used for wet-vacuum sampling was IPA because it is a relatively nonvolatile solvent, typically not destructive to common building surfaces, and readily available in large quantities at low cost. Other wetting agents were selected based on previous research efforts [8] or manufacturers' information. The detailed information regarding composition and preparation of wetting agents is presented in Table 3-5.

**Table 3-5. Surface Wetting Agents for Wet-Vacuum Sampling**

Cleaning Agent	Composition	Manufacturer	Preparation	Utilized in Phase No.
<b>Aqueous Wetting Agents</b>				
Water	H <sub>2</sub> O	Dracor Water Systems, Durham, NC, USA	DI water; no preparation	I, II, III
Water-Tween® 20	Tween® 20 in water	Sigma Aldrich St. Louis, MO, USA	1-part Tween® 20 to 50 parts DI water	I
Water-Dawn Ultra®	Dawn Ultra® dishwashing liquid in water	P&G Cincinnati, OH, USA	1-part Dawn Ultra® dishwashing liquid to 50 parts DI water†	III
Water-SSDX-12 SuperSoap®	SSDX-12 SuperSoap® in water	Aerosafe Norcross, GA, USA	1-part SSDX-12 SuperSoap® to 128 parts DI water	III
<b>Organic Solvent Wetting Agents</b>				
IPA	CH <sub>3</sub> CHOHCH <sub>3</sub> , certified ACS	Fisher Chemical Waltham, MA, USA	No preparation	I, II, III

All aqueous solutions were mixed immediately prior to testing using the preparation procedures listed in Table 3-5. Wetting agents were prepared in quantities sufficient to perform sampling of all analytical batches planned for each testing day.

### 3.6.1 Wetting Agent Application Methods

During Phase I, wetting agents were applied by using a 240-mL spray bottle made of high-density polyethylene equipped with an adjustable polypropylene sprayer (McMaster-Carr, Elmhurst, IL, USA; product # 9864T51). Spray application was uniform across the surface in a left to right sweeping motion, with individual spray patterns overlapping by at least 50%.

Both commercial wet-vacuum units used during Phase II and III are equipped with a built-in spray tool that can be triggered to apply the solution from the wetting tank to the surface. No modifications were made to the spray pattern or spray flow rate of each unit. During method development tests only, one wet-vacuum unit was designated as the unit that sprayed the liquid while a second wet-vacuum unit was used to collect the sample. For each wet-vacuum sampling test, the initial and end mass of the wetting liquid holding tank was recorded to check for the accuracy and precision of wetting solvent application (nominal 50 mL).

## 3.7 Method Development Tests

### 3.7.1 Extraction of target chemicals from water and water-surfactant solutions

Methods for the liquid-liquid exchange (LLE) of target chemicals sampled using aqueous wetting agents (DI water and water-surfactant solutions) were optimized prior to testing. Liquid-based testing was used for each target chemical wetting agent combination. For this series of tests, one test was performed with a 1-hour analytical holding time (HT) and one test was performed with a 24-hour HT, where HT is the

period the chemical was in contact with the aqueous (surfactant-containing) solution prior to extraction. The HTs were designed to determine the ability to recover target chemicals from the surfactant-containing solutions and to determine the associated stability of target chemicals in surfactant–water solutions. The spiked sample concentration matched that of samples that originated from vacuum sampling (i.e., 24 µL of neat chemical was spiked into 50 mL of wetting solvent solution [100% target concentration]). Tests were performed in triplicate (n = 3) for each chemical–wetting solvent combination. One procedural blank of wetting solvent did not receive the chemical contamination but did undergo the extraction procedure to monitor for possible cross-contamination or quantitative interferences that might result from the LLE.

The extraction optimization test parameters are listed in Table 3-6. Optimized LLE procedures are detailed in Section 4.2. Recoveries of target chemicals for each LLE method and HT tested are described in Section 5.1. Additives shown in Table 3-6 were used to salt out the surfactant from the aqueous solutions.

**Table 3-6. Experimental Parameters for Liquid–Liquid Extraction Optimization Tests**

Wetting Agent Solution Chemical Combination	Chemical Conc. Target Tested	Extraction Solvent Type	Wetting Solvent to Extraction Solvent Ratio (v:v)	Additives	Other Steps
<b>TEP</b>					
Water	0–100%	DCM	1:1	NaCl, NaH <sub>2</sub> PO <sub>4</sub> /Na <sub>2</sub> HPO <sub>4</sub> buffer (pH 7.0)	Drying with Na <sub>2</sub> SO <sub>4</sub>
Water-Tween®	0–100%	DCM	1:1	NaCl, NaH <sub>2</sub> PO <sub>4</sub> /Na <sub>2</sub> HPO <sub>4</sub> buffer (pH 7.0)	Drying with Na <sub>2</sub> SO <sub>4</sub>
Water-Dawn Ultra®	0–100%	DCM	1:1	NaCl, NaH <sub>2</sub> PO <sub>4</sub> /Na <sub>2</sub> HPO <sub>4</sub> buffer (pH 7.0)	Drying with Na <sub>2</sub> SO <sub>4</sub>
Water-SuperSoap®	0–100%	DCM	1:1	NaCl, NaH <sub>2</sub> PO <sub>4</sub> /Na <sub>2</sub> HPO <sub>4</sub> buffer (pH 7.0)	Drying with Na <sub>2</sub> SO <sub>4</sub>
<b>2-CEPS</b>					
Water	0–100%	Hexane	1:1	None	Drying with Na <sub>2</sub> SO <sub>4</sub>
Water-Dawn Ultra®	0–100%	Hexane	1:1	NaCl	Drying with Na <sub>2</sub> SO <sub>4</sub>
Water-SuperSoap®	0–100%	DCM	1:1	NaCl	Drying with Na <sub>2</sub> SO <sub>4</sub>
<b>Nitrobenzene</b>					
Water	0–100%	Hexane	1:1	None	Drying with Na <sub>2</sub> SO <sub>4</sub>
Water-Tween®	0–100%	Hexane	1:1	None	Drying with Na <sub>2</sub> SO <sub>4</sub>

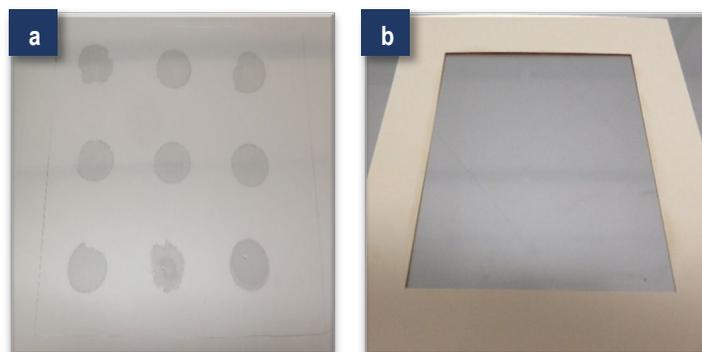
### 3.7.2 Wipe-based methods for surface sampling of target chemicals

The effectiveness of the optimized wet-vacuum method for medium- and large-area sampling, evaluated in experimental Phase III (Section 3.8.3), was compared to the chemical recovery achieved using the wipe-based reference method. In this study, modified SW-846 Method 3572 [9] for extraction of CWAs from wipe samples using micro extraction was considered for the surface sampling of CWA surrogates (TEP and 2-CEPS). During two rounds of method optimization testing (data not shown), the standard wipe sampling method was modified to include selected sampling strategies (extraction solvent and extraction solvent volume) outlined in the EPA technical report, Evaluation of Chemical Warfare Agent Wipe Sampling Collection Efficiencies on Porous, Permeable, or Uneven Surfaces [3]. The modifications are listed in Table 3-6.

**Table 3-6. Experimental Parameters of Original and Modified Reference Methods for Wipe Sampling and Extraction of Wipes**

Parameter/Method	EPA Method 3572 [3]	Modified Method 3572
Sampling area	10 cm x 10 cm	10 cm x 10 cm
Sampling wipe	5.08 cm. x 5.08 cm cotton gauze wipe	5.08 cm. x 5.08 cm cotton gauze wipe
Number of wipes per sampling area	1 per 100 cm <sup>2</sup>	2 per 100 cm <sup>2</sup>
Sampling (wetting) solvent	IPA	IPA
Sampling (wetting) solvent volume	1 mL per wipe	1.5 mL per wipe
Extraction solvent	10% IPA in DCM	DCM
Extraction solvent volume	4 mL	2 x 15 mL

For surface sampling efficiency tests, the reference surface (stainless steel) was contaminated with solutions of target chemicals using the procedure described in Section 3-3 and placed in the same type of precleaned test box that was used during decontamination testing. After the prescribed contact time of the chemical with the surface (30 min), wipe samples were collected and extracted using the procedures described in Section 4.1. Additional control samples included a wipe spike, a control spike sample, and procedural blank. Figure 3-6 shows a 2-CEPS-contaminated stainless-steel coupon before and after wipe sampling. The template was used to establish the 10 cm × 10 cm sampling area,



**Figure 3-6. Stainless-steel surface before (a) and after (b) wipe sampling.**

The results for the final round of the wipe-sampling method optimization are given in Table 5-1.

## 3.8 Surface Sampling Tests

### 3.8.1 Phase I: Selection of operational parameters for wet-vacuum sampling

Since the target chemicals were characterized by various degrees of solubility in water, the selection of the most appropriate, and ideally universal to all chemical–surface combinations wetting condition was considered first. Phase I, the proof of concept phase, focused on the fundamentals of a new wet-vacuum–based methodology being developed. In this phase of testing, a small custom-made apparatus (Section 3-5) was used to allow for cost-effective and environmentally responsible testing (the cost of equipment and amount of generated waste were minimal compared to Phase II and III operational-scale testing in which a commercial vacuum was used).

Phase I laboratory-scale testing included the following steps:

1. Selection of the wetting agent suitable for sampling chemicals with various degrees of solubility in water.
2. Selection of the optimal temporal duration between wetting agent application and start of wet-vacuum sampling (surface residence time, or lapse time), as a function of surface type.
3. Selection of the optimal wetting agent volume, defined as a minimal volume that offered good recovery of all target chemicals. Chemical recovery testing was limited to surfaces with low porosity.
4. Evaluation of an optimized method for all chemical–material combinations.

The selection of optimal experimental parameters was based on best sampling results (highest sampling efficiency) achieved in consecutive steps of testing. The test matrix for Phase I testing is provided in Table 3-7. The results are described in Section 5.2.

**Table 3-7. Experimental Parameters of Phase I Testing**

Test Parameter	Test Material <sup>†</sup>	Target Chemical	Wetting Agent <sup>†</sup>	Wetting Agent Volume (mL)	Lapse Time (s)	Test Matrix <sup>†</sup>	Area Sampled <sup>‡</sup> (cm <sup>2</sup> )
Selection of wetting agent	Stainless steel	Nitrobenzene Phenol TEP	Water Tween®-Water IPA	100	100	3 x TC 1 x PB 1 x CS	929
Selection of lapse time	Stainless steel Vinyl flooring Plywood	Nitrobenzene Phenol TEP	Water Tween®-Water IPA	100	1 10 100	3 x TC 1 x PB 1 x CS	929
Selection of wetting agent volume	Plywood	Phenol	IPA	10 50 100	10	3 x TC 1 x PB 1 x CS	929
Evaluation of optimized method on various surfaces	Stainless steel Vinyl flooring Plywood	Nitrobenzene Phenol TEP	IPA	50	10	3 x TC 1 x PB 1 x CS	929

<sup>†</sup>Detailed information on test materials and wetting agents in Section 3.0. <sup>‡</sup>Per each test condition. <sup>†</sup>Per replicate test sample; TC – Test coupon; PB – Procedural blank; CS – Control spike sample.

### 3.8.2 Phase II: Application of commercial wet-vacuum systems for surface sampling

The wet-vacuum sampling parameters developed during Phase I testing were applied to Phase II commercial wet-vacuum cleaner–based operational-scale testing. Phase II laboratory-scale testing included the following steps:

1. Initial evaluation of the selected commercial vacuum cleaner suitable for sampling chemicals with various degrees of solubility in water.
2. Selection and optimization of the commercial vacuum cleaner for operational-scale wet-vacuum sampling efficiency.

The overall effectiveness of wet-vacuum sampling was determined for two commercial devices and three wetting agents as a function of the material type (Table 3-8).

**Table 3-8. Experimental Parameters of Phase II Commercial Wet-Vacuum Cleaner Testing – Initial Evaluation of Rug Doctor and Bissell Vacuum Cleaners**

Wet Vacuum Cleaner*	Test Materials*	Target Chemical	Wetting Agents*	Wetting Agent Volume (mL)	Lapse Time (s)	Test Matrix†	Area Sampled‡ (cm <sup>2</sup> )
Rug Doctor	Stainless steel Laminate flooring	Nitrobenzene Phenol	Water IPA**	50	10	5 x TC 1 x PB 1 x CS	929
Bissell	Vinyl flooring	TEP	Water IPA**			5 x TC 1 x PB 1 x CS	929

\* Detailed information on test materials and wetting agents in Section 3.0. † Per each test condition. ‡ Per replicate test sample; TC – Test coupon; PB – Procedural blank; CS – Control spike sample; \*\*Tested in scoping tests only

Results from this initial evaluation of commercial vacuum cleaners (Section 5.3.1) showed that the Bissell unit offered better sampling efficiency for all chemical test material–wetting agent combinations tested. Due to large evaporative losses of IPA observed during the initial scoping tests, the organic solvent-based sampling procedure was further optimized to include a vacuum conditioning step (a 50-mL pre-rinse of the same wetting solvent). In addition, different volumes for the introduced post-sampling rinse of the vacuum cleaner (post-rinse), using clean IPA aspirated directly from a beaker, were tested for improved recovery of target chemicals (Table 3-9). The results of Phase II testing are summarized in Section 5.3.2.

**Table 3-9. Experimental Parameters of Phase II Commercial Vacuum Cleaner Testing for Improved Recovery of Organic Solvent-based Wetting Agent**

Wet Vacuum Cleaner	Test Material	Target Chemical	Wetting Agent*	Wetting Agent Volume			Lapse Time (s)	Test Matrix†	Area Sampled‡ (cm <sup>2</sup> )
				Pre-rinse (mL)	Surface Wetting (mL)	Post-rinse (mL)			
Bissell	Stainless steel	Phenol TEP	IPA	50	50	no post-rinse 100 150	10	5 x TC 1 x PB 1 x CS	929

\* Detailed information on test materials, wetting agents, and commercial wet-vacuum in Section 3.0. † Per each test condition. ‡ Per replicate sample.

### **3.8.3 Phase III: Multivariate characterization of wet-vacuum-based sampling methods**

In Phase III, the Bissell vacuum system was evaluated for collection efficiency of two selected chemicals (TEP and 2-CEPS) using the optimized method tested in Phase II that included a pre-rinse step (vacuum system conditioning step) and a post-collection rinse step. Phase III operational testing was conducted as follows:

1. Evaluation of one commercial device for wet-vacuum sampling of a medium-sized area, as a function of chemical surface loading, including a comparison to the wipe-based surface sampling method.
2. Evaluation of the commercial device for high-capacity composite-area wet-vacuum sampling.

The various test combinations are shown in Tables 3-10 and 3-11. Each Phase III test was performed in a sample configuration similar to the sample configuration in the Phase I and II experiments, three wet-vacuum sampling method replicates (test coupons) accompanied by a procedural blank and control spike. The same solvent (IPA) sampling and analysis sampling method was further tested for a discrete single medium-sized area (SA, 929 cm<sup>2</sup>) and a larger-sized area via multiarea composite (MAC) sampling of an area approximately 4,645 cm<sup>2</sup>. Because there were concerns about the feasibility of the IPA-based method for sampling of semiporous materials (vinyl flooring) and health and safety concerns (flammability) about the use of IPA in an operational setting, the SA methodology was also tested in the aqueous wetting solvent configuration. Application of the optimized methodology for low surface concentrations (1 to 20% of default-loading) sampling of nonporous material was performed using an SA and IPA as well as a selected water-based sampling agent. Other supplementary tests for the wet-vacuum system characterization are described in Section 3.9. The results from Phase III sampling tests are given in Section 5.4.

#### ***3.8.3.1 Operational assessment of commercial wet-vacuum cleaner-based sampling***

##### ***3.8.3.1.1 Medium-area sampling***

The medium-area sampling was designed as a systematic study of the effectiveness of the wet-vacuum method for sampling varying surface loadings of selected CWAs and TICs. The medium-area sampling was completed for TEP and 2-CEPS, which have noticeably different chemical solubility in water (see Table 3-4) on nonporous (stainless-steel) and semiporous (vinyl flooring) surfaces. The porous substrate, plywood, was not tested because the wet-vacuum method has very limited applicability for highly porous and permeable materials (as demonstrated during the proof of concept Phase I testing). Each coupon was sampled using a multi-pass vacuum procedure that was used for the medium-sized coupon sampling method (Appendix A, Section A-1). Both classes of wetting agents (organic solvent- and water-based, Table 3-10) were tested using this method. Testing at lower surface concentrations were limited to IPA wetting agent and only the best performing water-based wetting agent (Dawn Ultra®). Results from the optimized wet-vacuum sampling method were compared to results from the modified standard wipe-based CWA sampling method. The results from Phase III medium-area testing are provided in Sections 5.4.1 and 5.4.2, and the comparison to wipe method results are in Section 5.4.4.

**Table 3-10. Experimental Parameters of Phase III Multivariate Characterization of Commercial Vacuum Cleaner Testing – Single Medium-Area Sampling**

Wet Vacuum Cleaner	Test Material	Target Chemical	Surface Conc.*	Wetting Agents†	Wetting Agent Volume			Lapse Time (s)	Test Matrix‡	Area Sampled** (cm <sup>2</sup> )
					Pre-rinse (mL)	Surface Wetting (mL)	Post-rinse (mL)			
Bissell	Stainless steel	TEP 2-CEPS	1% 2% 10% 20% 100%	Isopropyl Alcohol	50	50	100	10	3 x TC 1 x PB 1 x CS	929
Bissell	Stainless steel	TEP 2-CEPS	100%	Water Dawn Ultra®-water SuperSoap®-water	no pre-rinse	50	100	10	3 x TC 1 x PB 1 x CS	929
Bissell	Vinyl flooring	TEP 2-CEPS	100%	Water Dawn Ultra®-water SuperSoap®-water	no pre-rinse	50	100	10	3 x TC 1 x PB 1 x CS	929
Bissell	Stainless steel	TEP 2-CEPS	2% 10% 20%	Dawn Ultra®-water	no pre-rinse	50	100	10	3 x TC 1 x PB 1 x CS	929

\*With respect to highest loading tested. †Detailed information on test materials, wetting agents, and commercial wet-vacuum is found in Section 3.0. ‡Per each test condition. \*\*Per replicate sample.

### 3.8.3.1.2 Large-area sampling

For large-area sampling, the organic solvent-based method was used to perform the MAC sampling, where the same vacuum cleaner was used for composite collection from five coupons (5 x 929 cm<sup>2</sup>; total area = 4645 cm<sup>2</sup>) (Table 3-11). Each composite-area coupon was sampled using the multi-pass procedure that was used for the medium-sized coupon sampling method (Appendix A, Section A-1). The vacuumed sampling liquids were collected and processed as one composite sample. The results from Phase III large-area testing are provided in Section 5.5.

**Table 3-11. Experimental Parameters of Phase III Multivariate Characterization of Commercial Vacuum Cleaner Testing – Large-Area Composite Sampling**

Wet Vacuum Cleaner	Test Material	Target Chemical	Surface Conc.	Wetting Agent*	Wetting Agent Volume			Lapse Time (s)	Test Matrix†	Area Sampled‡ (cm <sup>2</sup> )
					Pre-rinse (mL)	Surface Wetting (mL)	Post-rinse (mL)			
Bissell	Stainless steel	TEP 2-CEPS	100%	Isopropyl alcohol	50	5 x 50	100	10	3 x TC 1 x PB 1 x CS	4645

\*Detailed information on test materials, wetting agents, and commercial wet-vacuum is found in Section 3.0. †Per each test condition. ‡Per replicate composite sample.

## 3.9 Supplementary Surface Concentration Verification Tests

### 3.9.1 Verification of surface concentrations by direct extraction

The efficacy of wet-vacuum sampling is a combination of the affinity of the target chemical with the material, the wetting agent (either aqueous or organic solvent-based) and the mechanical cleaning action of the vacuum cleaner. During wet-vacuum sampling, both the applied vacuum and the vacuum cleaner nozzle/brush scrubbing action aid in the removal of the chemical from the contaminated surface. A series of supplementary tests was performed to investigate the ability of wetting solvents to extract selected chemicals from the reference material without mechanical scrubbing. These tests were performed to verify the potential of the wetting solvent for lifting and absorbing chemicals from the surface. Three composite-area stainless-steel coupon sets (12 [5.1 cm x 5.1 cm dimensions] coupons per set) were spiked individually with one discrete droplet (2-microliter ( $\mu\text{L}$ ) volume) with the chemical at the same spiking concentration as the wet-vacuum test coupons, i.e., at 100%, 20%, 10%, 2%, and 1% of the default surface concentration. The spiking approach was like the approach used to spike the medium size area (Figures 3-1 and 3-2) but now with 12 individual coupons arranged below the template. These small coupons did not undergo wet-vacuum sampling, but after the 30-min contact time, they were extracted using various aqueous and organic solvent wetting agents as well as directly in hexane. Extraction of each set of twelve coupons was performed in 150 mL of solvent using a custom-made extraction coupon holder made of stainless-steel wire and Teflon (Figure 3-7).



Figure 3-7. Surface spike controls ready for extraction.

After extraction, the resulting solutions underwent the same analytical process as the wetting liquid samples from the wet-vacuum sampling (Sections 4.1 and 4.2). Samples were processed for analysis as described in Section 4.2. Test results are provided in Section 5.4.3.

## 3.10 Supplementary Tests

### 3.10.1 Operational time estimates

The operational time estimates for wet-vacuum-based versus wipe-based sampling methods were prepared for each experimental step using laboratory data logs and laboratory notebooks. These estimates do not include the time needed to clean or contaminate materials for testing, as these steps would not be part of an actual field-sampling event. Therefore, only four procedural steps were included in the operational

time estimates: (1) Preparation of sampling kits; (2) surface sampling; (3) extraction process; and (4) preparation of samples for analysis. These time estimates are provided in Section 5.6.

### **3.10.2 Flow and temperature profiles of wet-vacuum system**

#### ***3.10.2.1 Measurement of vacuum airflow***

The airflow rate of the wet-vacuum system was measured at the EPA Metrology Laboratory in Research Triangle Park, NC. A flow meter (Roots Meter EM175, Dresser, Chagrin Falls, OH, USA) was placed in line with the wet-vacuum collection hose to record the volume of air moving through the wet-vacuum. The dispenser tubing was removed from the vacuum hose and penetrations through the vacuum hose were sealed. The vacuum nozzle was placed on the surface of a medium-sized stainless-steel TC and clamped in place at a 30-degree (approximate) angle to the coupon surface to simulate the sample collection procedure. Airflow was measured for 20 min to simulate the timeframe associated with sampling approximately 20 medium-sized coupons. Results are provided in Section 5.7.1.

#### ***3.10.2.2 Temperature measurement of wet-vacuum unit***

A series of tests was conducted to determine the maximum temperature that vacuum cleaner components reach after sampling a large-sized surface area. The tests were performed to locate any high-temperature areas (hot spots) within the wet-vacuum system during prolonged sampling. A medium-sized stainless-steel coupon was placed inside a plastic test box under a chemical hood. The vacuum unit holding tank was filled with DI water, and the coupon and vacuum cleaner were conditioned (pre-rinsed) with 50 mL of DI water. After the pre-rinse was completed, the surface was sampled using a standard wet-vacuum procedure (Appendix A, Section A-1). The spray and vacuum sampling were repeated from 4 to 20 times to mimic periods of various lengths of (composite) sampling. Following vacuum sampling, the system was rinsed with 100 mL of DI water aspirated from a 1000-mL beaker. The wet-vacuum system was filmed and photo-documented using a thermal imaging camera (FLIR E4, Wilsonville, OR, USA) during the entire sampling process. This infrared camera is equipped with an uncooled microbolometer detector and offers infrared (IR) resolution of 80 × 60 pixels combined with multispectral dynamic imaging (MSX®) enhancements for the spectral range of 7.5–13 μm. Results are reported in Section 5.7.2.

## 4.0 Sampling and Analysis

### 4.1 Surface Sampling Methods

#### 4.1.1 Medium-size area wet-vacuum sampling

The experimental design that was used for medium-sized discrete (1-point, 1 x 929 cm<sup>2</sup>) SA sampling is shown in Figure 4-1. A detailed description of the wet-vacuum sampling process is provided in Appendix A, Section A-1. Extraction methods are summarized in Section 4.2.

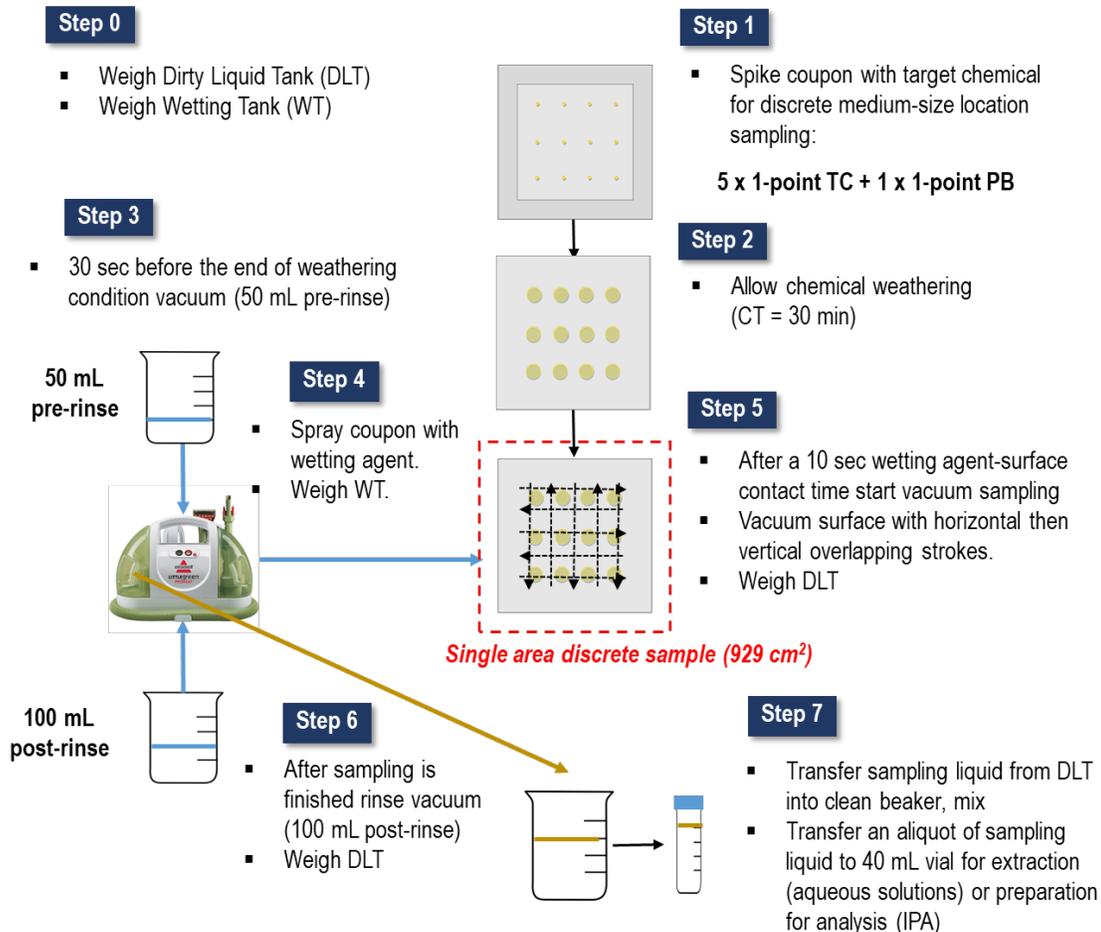


Figure 4-1. Experimental design and sample flow for SA sampling. TC - test coupon; PB – procedural blank; CT – contact time; WT – wetting tank; DLT – dirty liquid tank

#### 4.1.2 Large-area wet-vacuum sampling

Large-area sampling was designed to test the efficiency of wet-vacuum sampling for composite-area (5-point, 5 x 929 cm<sup>2</sup>) MAC sample collection. This sample compositing approach was developed for improved turnaround times and cost of analysis. The multistep experimental design that was used for MAC sampling is shown in Figure 4-2. A detailed description of the wet-vacuum sampling process is provided in Appendix A, Section A-1. Extraction methods are summarized in detail in Section 4.2.

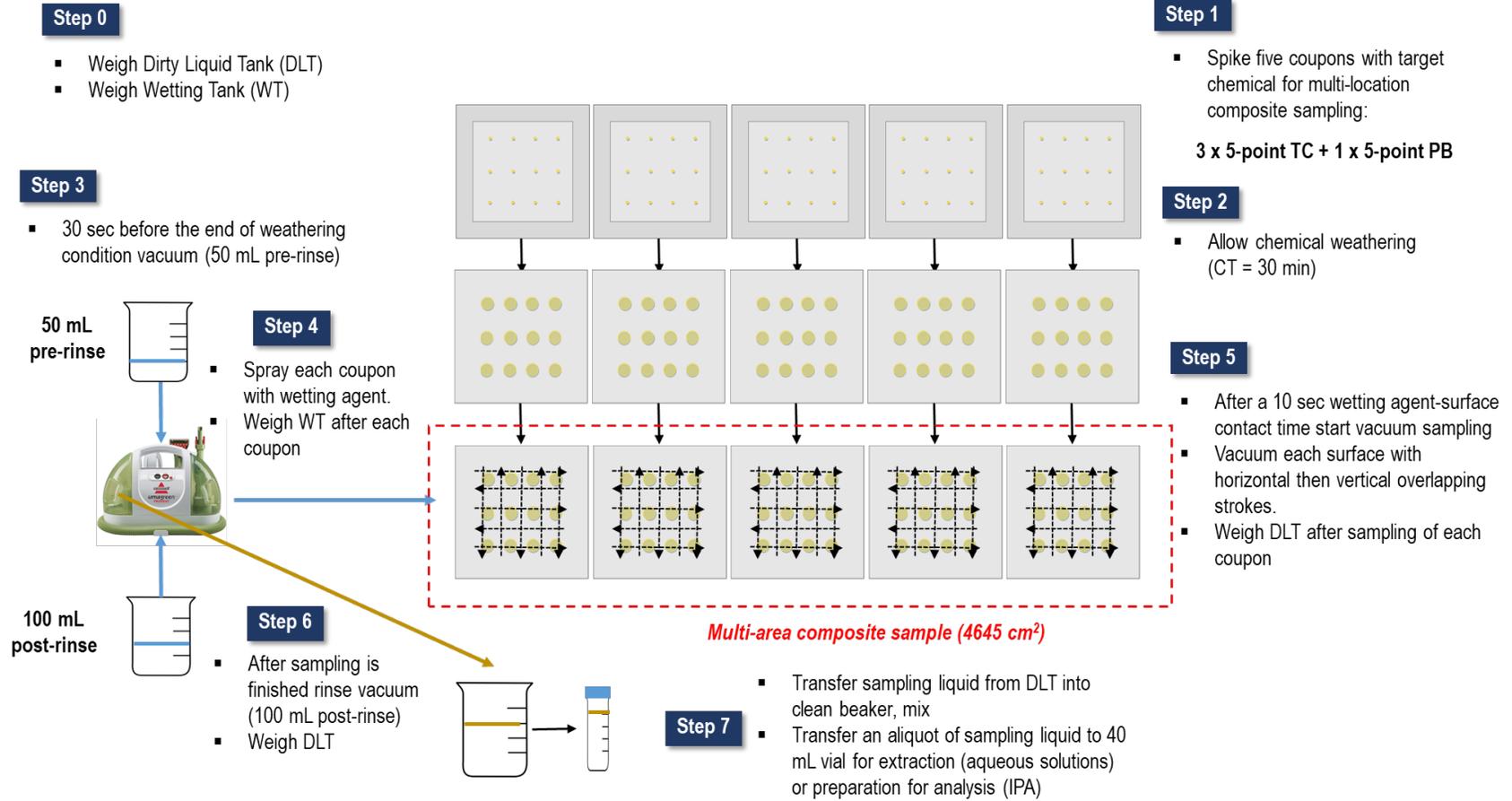


Figure 4-2. Experimental design and sample flow for MAC sampling. TC – test coupon; PB – procedural blank; CT – contact time; WT – wetting tank; DLT – dirty liquid tank

### 4.1.3 Small-area wipe-based sampling

Surface wipe sampling was performed on a small central area (1 point, 100 cm<sup>2</sup>; Figure 4-3) for comparison with wet-vacuum method performance and to sample for residual chemical on the surface post-sampling in mass balance tests. Each wipe was used following a four-step process. A series of horizontal to vertical strokes followed by diagonal strokes and then perimeter wiping strokes were used, where the wipe was folded after each step (with the contaminated side folded inward). Types of wiping media, wetting solvents, and amount of wetting solvent for all material–chemical combinations tested are given in Section 3.7.2. The wipe extraction methods are summarized in Section 4.2.

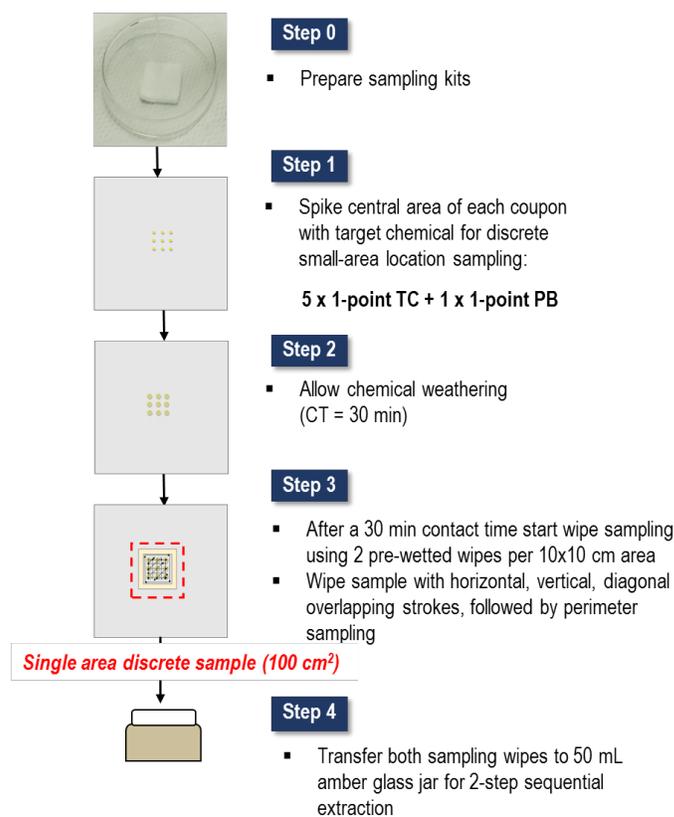


Figure 4-3. Experimental design and sample flow for small area sampling. TC – test coupon; PB – procedural blank; CT – contact time

## 4.2. Extraction Methods

This section summarizes the extraction procedures used for all surface-wipe and sample liquid-chemical combinations that resulted from the wipe-based and wet-vacuum-based sampling procedures. All wipe and wetting liquid extraction methods were described in Section 3.7.

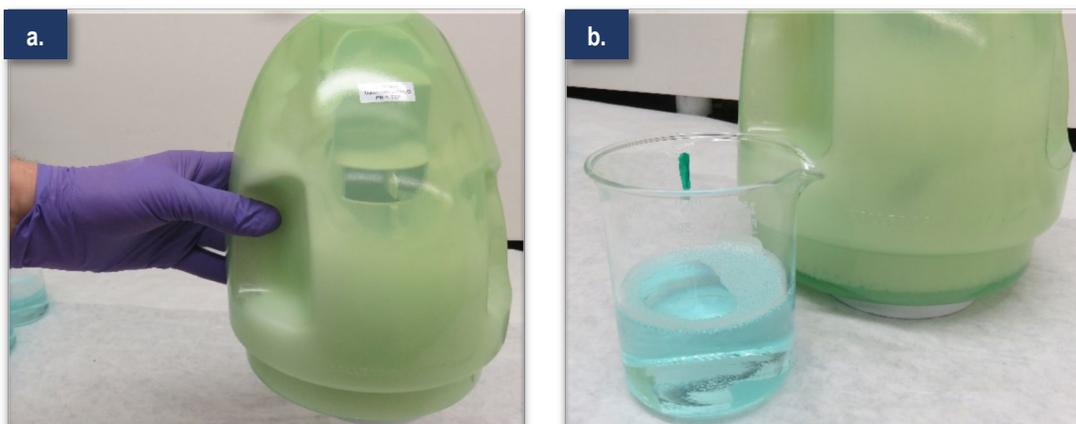
### 4.2.1 Extraction of surface wipes

After completion of wipe sampling (Section 4.1.3), the two wipes used for surface sampling were placed together in a precleaned 40-mL wide-mouth extraction vial with a polytetrafluoroethylene-lined lid for composite extraction. The extraction vial was filled with 15 mL of dichloromethane (DCM) (Optima™, high-performance liquid chromatography/spectrophotometry, GC/MS, and pesticide residue analysis grade Fisher Chemical, product no. H 303-4 UN1208), capped, and transferred to the sonicator for step 1 of the sequential extraction. Wipe samples were sonicated for 15 minutes. For step 2, the resulting liquid extract was transferred to a 40-mL glass vial and a second 15-mL aliquot of DCM was added to the two wipes. Then, the wipe samples were sonicated for another 15 minutes. For step 3, the step 2 extract was removed and combined with the extract from step 1 and refrigerated at  $4 \pm 2$  °C until further processing. Sample preparation for instrumental analysis is described in Section 4.3.

### 4.2.2 Extraction of water and water-based wetting agents

Target chemicals were extracted from the liquid samples generated during wet-vacuum procedures using a simplified LLE developed based on physicochemical properties of the target analytes, the sampling solvents, and existing standard methods [1]. All sampling liquids were extracted within one hour of sample collection because method development studies had shown that some chemicals have a limited stability in water and water-surfactant solutions (Section 5.1).

The recovered sampling liquid volume was measured through the weight measurement of the liquid waste collection tank before and after the sample collection. Sample liquids were transferred to a clean extraction beaker. For foaming wetting agents, the collected liquid was allowed to settle (10-15 min period) until the foam dissipated. This extract settling step was especially important for water-surfactant solutions with large expansion volumes for which large amounts of foam were observed (e.g., Dawn Ultra® water wetting agent). Figure 4-4 shows an example of the liquid waste in the collection tank immediately after sampling and during the settling phase as well as the settled liquid ready for LLE.



**Figure 4-4.** Dawn Ultra® water SL immediately after conclusion of sampling (a), during settling phase in the dirty liquid tank (b) and settled liquid (aliquot in beaker) ready for LLE (b).

The reagent volumes and amounts of additives to salt out the surfactants used for LLEs are listed in Table 4-1. For each LLE procedure, a 5- to 10-mL aliquot of sample liquid was transferred to a 40-mL extraction vial preloaded with the appropriate amount of additive(s) (Table 4-1). After addition of extraction solvent, each vial was capped, and the contents were manually shaken for 1 minute (min). After the aqueous and solvent layer separated, the entire extract layer was carefully collected using a Pasteur pipette and placed into a clean 40-mL vial with graduated markings. For TEP extractions, the procedure was performed twice, and the resulting extracts were combined. The total extract volume was recorded.

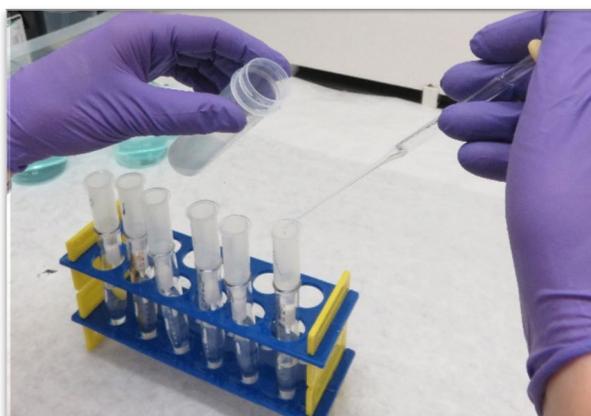
**Table 4-1. Chemical-Specific Experimental Conditions for Extractions of Aqueous SLs**

Wetting Agent	Extraction Solvent Type	Reagent Volumes		Additives	Other Steps
		Wetting Agent (mL)	Extraction Solvent (mL)		
<b>TEP</b>					
Water	DCM	10	2 x 5 mL	0.5 g NaCl; 10 $\mu$ L 1 M $\text{NaH}_2\text{PO}_4/\text{Na}_2\text{HPO}_4$ ; buffer pH 7.0	Extract dried with 1 g $\text{Na}_2\text{SO}_4$ cartridge
Water-Tween®	DCM	10	2 x 5 mL	5.0 g NaCl; 100 $\mu$ L of 1 M $\text{NaH}_2\text{PO}_4/\text{Na}_2\text{HPO}_4$ ; buffer pH 7.0	Extract dried with 1 g $\text{Na}_2\text{SO}_4$ cartridge
Water-Dawn Ultra®	DCM	10	2 x 5 mL	0.5 g NaCl; 10 $\mu$ L of 1 M $\text{NaH}_2\text{PO}_4/\text{Na}_2\text{HPO}_4$ ; buffer pH 7.0	Extract dried with 1 g $\text{Na}_2\text{SO}_4$ cartridge
Water- SuperSoap®	DCM	10	2 x 5 mL	0.5 g NaCl; 10 $\mu$ L 1 M $\text{NaH}_2\text{PO}_4/\text{Na}_2\text{HPO}_4$ ; buffer pH 7.0	Extract dried with 1 g $\text{Na}_2\text{SO}_4$ cartridge
<b>2-CEPS</b>					
Water	Hexane	10	10	None	Extract dried with 1 g $\text{Na}_2\text{SO}_4$ cartridge
Water-Dawn Ultra®	Hexane	10	10	5 g NaCl	Extract dried with 1 g $\text{Na}_2\text{SO}_4$ cartridge

Wetting Agent	Extraction Solvent Type	Reagent Volumes		Additives	Other Steps
		Wetting Agent (mL)	Extraction Solvent (mL)		
Water-SuperSoap®	DCM*	10	10	3 g NaCl	Extract dried with 1 g Na <sub>2</sub> SO <sub>4</sub> cartridge
Nitrobenzene					
Water	Hexane	10	10	None	Extract dried with 1 g Na <sub>2</sub> SO <sub>4</sub> cartridge
Water-Tween®	Hexane	10	10	None	Extract dried with 1 g Na <sub>2</sub> SO <sub>4</sub> cartridge

\*Unsuccessful with hexane

The extract was then passed through a short drying cartridge prepacked with granular anhydrous sodium sulfate (Bond Elut sodium sulfate drying cartridges, Agilent Technologies, Santa Clara, CA, USA; product no. 12131033) to remove residual surfactant water from the organic extract. The eluent was collected in a graduated centrifuge tube (Figure 4-5). After the drying step, the sodium sulfate cartridge was rinsed with solvent until the final sample volume was 10 mL.



**Figure 4-5. Drying of the SL extract**

After drying, extracts were transferred to a 12-mL vial and refrigerated at  $4 \pm 2$  °C until preparation for analysis (Section 4.3).

### 4.3 Preparation of Samples for Analysis

Extracts generated from sample liquids (Section 4.2.2), surface wipes (Section 4.2.1), and IPA wetting solvent were prepared for analysis in 1.8-mL amber glass gas chromatography (GC) vials. Depending on the type of sample, extracts underwent up to 100-fold dilution. Briefly, an aliquot of raw extract was drawn using an appropriate-sized micropipette and added to a GC vial filled with a premeasured amount of hexane or IPA. The control spike samples were also diluted up to 25-fold. If analytical results were outside the calibration range, the analytical laboratory performed necessary dilutions and reported dilution factors along with quality control (QC) data. The samples were refrigerated at  $4 \pm 2$  °C or below prior

to delivery to the local EPA laboratory or shipment to the external chemical analysis laboratory. All analytical batches were accompanied by a chain of custody form and inspected at the analytical laboratory upon receipt.

#### 4.4 Instrumental Analyses

Instrumental analyses were performed at an accredited external laboratory (EMSL Analytical, Inc., Cinnaminson, NJ) or by the EPA Organic Support Laboratory (OSL). The standard methods used for each analyte are listed in Table 4-2. The instrumental parameters and conditions for instrumental analyses are listed in Appendix A (Tables A-1 through A-6). Analyses by the EPA OSL were limited to Phase III samples associated with the wet-vacuum tests described in Section 3.8.3.

**Table 4-2. Instrumental Methods Used for Analysis of Target Analytes**

Target Analyte	Experimental Phase	Instrumental Method	Reference Method	Analytical Laboratory
Phenol	Phase I, Phase II	GC/FID*; Spectrophotometry†	NIOSH 2546‡ [10]; EPA 420.1 [11]	EMSL Analytical, Inc.
Nitrobenzene	Phase I, Phase II	GC/FID	NIOSH 2005‡ [12]	EMSL Analytical, Inc.
TEP	Phase I, Phase II	GC/FID	NIOSH 5034‡ [13], NIOSH 5038‡ [14]	EMSL Analytical, Inc.
	Phase III	GC/MS	Not available	EPA OSL
2-CEPS	Phase III	GC/MS	Not available	EPA OSL
*In IPA. †In aqueous samples; ‡Modified gas chromatography/flame ionization detector (GC/FID); GC/MS – gas chromatography/mass spectrometry.				

For EMSL Analytical, Inc. analyses, a calibration range of 1–100 µg/mL for all target analytes (7-point calibration curve; 1-10-20-40-60-80-100 µg/mL) was used for initial calibration (ICAL), with reporting limit verification (RLV) and initial calibration verification (ICV) analyses performed at the lowest and mid-calibration level, respectively, prior to each analytical run. In addition, prior to each analytical run, a laboratory control sample (LCS) and the laboratory control sample duplicate (LCSD) were analyzed. A continuous calibration (CC) standard at a mid-level concentration was analyzed for every 10 samples, with a calibration end check (EC) performed at the end of each analytical run. Additional QC samples included duplicate instrument injections (DUPS) of test samples and laboratory blanks. Samples with results below the lowest calibration point (i.e., 1 µg/mL) were reported as less than the limit of quantitation (<LOQ). Acceptance criteria for QC checks are listed in Table 4-3.

**Table 4-3. QC Checks for Instrumental Analyses Performed by External Laboratory**

QC Check	Acceptance Limits
Initial Calibration (ICAL)	7-point initial calibration prior to analysis*
Reporting limit verification at lowest point concentration (RLV)	60–140% of lowest ICAL
Initial calibration verification at midpoint concentration (ICV mid)	80–120% of midpoint ICAL
Laboratory control sample at midpoint concentration (LCS)	70–130% of expected concentration
Laboratory Control sample duplicate at midpoint concentration (LCSD)	<25% RPD
Continuous calibration (CC) at midpoint concentration	80–120% of midpoint ICAL
End check of calibration (EC) at midpoint concentration	80–120% of midpoint ICAL
Duplicate injections (DUP)	<25% RPD
Laboratory (solvent) blank (LB)	<LOQ
*Recalibrate when continuous calibration fails acceptance criteria and/or after system maintenance. RPD – relative percent difference; LOQ – limit of quantitation	

The EPA OSL calibration range was 100–10,000 ng/mL, with quantitation performed using two 5-point curves that were dependent on the sample concentration. The high-concentration curve (1,000-2,500-5,000-7,500-10,000 ng/mL) was used for analysis of sample materials that had high sampling efficiency and control spikes at a 100% target concentration. The low-concentration curve (100-250-500-750-1,000 ng/mL) was used for analysis of sample materials that had low sampling efficiency, control spikes at a 10% target concentration, and blanks. Each calibration standard included 1,000 ng/mL of the internal standard (IS), naphthalene- $d_8$  (from EPA 8270 semivolatile internal standard mix [CRM46955, MilliporeSigma, St. Louis, MO]); the IS was also present in all test and control spike samples at the same concentration level. Prior to sample analysis, a minimum 5-point ICAL was performed, and the coefficient of determination ( $R^2$ ) was determined (target  $R^2 \geq 0.995$ ). The continuous calibration was performed using a mid-concentration calibration standard, that is, approximately every 10 test samples and at the end of the analytical run, with an acceptance control limit of 80–120% of the ICAL concentration. If QC criteria were not met, the instrument was recalibrated, and any affected samples were reanalyzed. Additional QC samples included duplicate injections of test samples (one duplicate injection per analytical run; acceptance criteria: relative percent difference [RPD] <20%) and analysis of blanks (procedural blank and laboratory solvent blank [LB]).

Prior to testing, an initial laboratory proficiency evaluation was performed for both laboratories. Accuracy and precision were determined by analysis of multiple measurements of control spike solutions ( $n = 3-5$  for each concentration level; single analytical run). Control spike samples were generated by spiking the target chemical or target chemical solution used during testing directly into the injection solvent (e.g., hexane, IPA, water). All control spikes were sonicated for 10 minutes and then diluted as needed. Each control spike set was accompanied by one laboratory blank sample (1 mL of solvent used for preparation of samples for analysis). These control spike experiments were used as independent verifications of the results obtained from the outside chemical analysis laboratory. The initial and continuing laboratory proficiency results are listed in Table 4-4.

**Table 4-4. Initial and Continuing Laboratory Proficiency Results**

Target Chemical and Analytical Laboratory	Spike Control Sample Analysis Results		Solvent Blank
	Accuracy and Precision	Number of Samples Analyzed	
	(% of true value $\pm$ 1 SD; RSD [%])	(n)	
<b>EMSL Analytical, Inc.</b>			
Nitrobenzene (initial*)	81.6% $\pm$ 14.7% SD; RSD=18%	2	<LOQ
Nitrobenzene (continuing†)	98.2% $\pm$ 12.5% SD; RSD=13%	11	<LOQ
Phenol (initial‡)	97.0% $\pm$ 2.9 SD; RSD=2.8%	3	<LOQ
Phenol (continuing†‡)	97.0% $\pm$ 7.9% SD; RSD=8.2%	33	<LOQ
TEP (initial*)	85.5% $\pm$ 2.0% SD; RSD=2.3%	6	<LOQ
TEP (continuing†)	90.9% $\pm$ 14.1% SD; RSD=16%	21	<LOQ
<b>EPA OSL</b>			
TEP (initial*)	97.1% $\pm$ 3.5% SD; RSD=3.6%	12	<LOQ
TEP (continuing†)	96.6% $\pm$ 13.2% SD; RSD=13%	20	<LOQ
2-CEPS (initial*)	104.1% $\pm$ 2.6% SD; RSD=0.16%	2	<LOQ
2-CEPS (continuing‡)	95.5% $\pm$ 7.1% SD; RSD=7.1%	22	<LOQ
SD: Standard Deviation; RSD: Relative Standard Deviation *Direct spike into solvent; QC samples prepared for initial laboratory proficiency testing. †Direct spike into solvent; QC samples prepared for each analytical batch resulting from a test/tests performed. ‡One control spike that was elevated (374% of theoretical spiked value) was excluded from average laboratory proficiency calculations. The reported value was confirmed to be elevated due to spiking solution preparation error. The new spiking solution concentration was verified by analysis of a second source standard (Certified Reference Material [CRM]), phenol solution in methanol at 500 $\mu$ L/mL (Product No. Supelco 46688, Sigma Aldrich, St. Louis, MO, USA).			

The acceptance criteria for the initial laboratory proficiency tests were 80–120% for accuracy (as recovery compared to theoretical concentration [true value]) and <30% RSD precision for each concentration level for replicate analysis for each concentration target. The results from the initial and ongoing analyses of control spike samples – calculated as arithmetic mean ( $\pm$  1 SD, %RSD) from results of multiple injections of the initial proficiency evaluation control spikes, or single injections of multiple analytical batch-specific control spikes (Table 4-2) – were within the acceptance criteria described above. All solvent blanks were below LOQ.

## 4.5 Data Reduction Procedures

### 4.5.1 Chemical concentration in sampling liquid calculations

The final sample concentration results (in  $\mu\text{g/mL}$ ) for liquid samples that did not undergo extraction (IPA as the wetting liquid) were converted to total mass of chemical per sample (mg/sample or sample composite) by multiplying by the collected sample volume and dilution factor, if applicable:

$$M_{s(\text{IPA})} = C_S \times V_t \times D_F \times 1,000 \quad (\text{Eq. 1})$$

where:

$M_{s(\text{IPA})}$  = mass of chemical in sample (mg)

$C_S$  = concentration ( $\mu\text{g/mL}$ ) from an individual replicate sample of IPA sampling liquid (SL)

$V_t$  = volume of SL collected (mL)

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

For aqueous solutions that were extracted using liquid extraction, a volumetric fraction conversion factor ( $CF_v$ ), considering a fraction of the total SL collected ( $V_t$ ) to volume of SL extracted ( $V_e$ ), was applied to chemical mass calculations as follows:

$$M_{s(\text{aqueous})} = C_S \times V_t \times D_F \times 1,000 \times CF_v \quad (\text{Eq. 2})$$

where:

$M_{s(\text{aqueous})}$  = mass of chemical in sample (mg)

$C_S$  = concentration ( $\mu\text{g/mL}$ ) from an individual replicate sample of extracted aqueous SL

$V_t$  = volume of SL collected (mL)

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

$CF_v$  = volumetric fraction conversion factor calculated according to Eq. 3:

$$CF_v = 1/(V_e/V_t) \quad (\text{Eq. 3})$$

where:

$CF_v$  = volumetric fraction conversion factor

$V_e$  = volume of SL extracted (mL)

$V_t$  = volume of SL collected (mL)

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

$$\%R_{QC} = C_{QC}/(V_{SP} \times S_C/V_T/D_F) \times 100\% \quad (\text{Eq. 4})$$

where:

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

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

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

$S_C$  = concentration of chemical in spiking solution ( $\mu\text{g}/\text{mL}$ )

$V_T$  = total sample volume (mL)

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

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

#### 4.5.2 Sampling efficiency calculations

The sampling efficiency was calculated using the mean of the chemical mass recovered from the replicate samples compared to the theoretical amount spiked onto a coupon surface:

$$\chi SE = \chi(TC/TS)_n \times 100\% \quad (\text{Eq. 5})$$

where:

$\chi SE$  = mean sampling efficiency (%)

TC = mean chemical amount recovered from replicate TCs or coupon composites (mg)

TS = mean chemical amount spiked onto coupons or coupon composite surface (mg)

The mean sampling efficiency ( $\chi SE$ ) was calculated as the arithmetic mean for each set of three to five replicates ( $n= 3$  to  $5$ ), along with the associated standard deviation (SD) and coefficient of variation (relative standard deviation, %RSD). If the sample (or sample composite) concentration was found to be below the lowest point of the calibration curve, but the signal-to-noise (S/N) ratio was greater than 10, the results were reported and flagged according to National Functional Guidelines [15]. The samples where analytes were not detected (not found or  $S/N < 3$ ) were reported as ND (non-detect). The analysis of variance (ANOVA) was used to check if the observed differences in sampling efficiencies of various methods tested are statistically significant. The p-values are reported at significance level of 95% ( $\alpha=0.05$ ).

## 5.0 Results

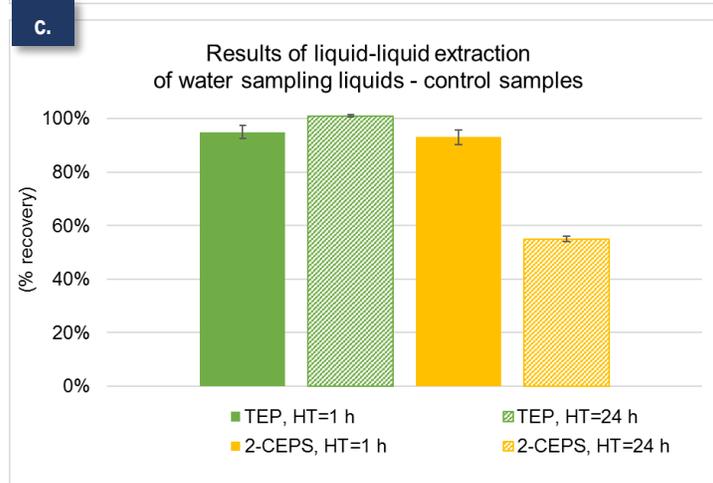
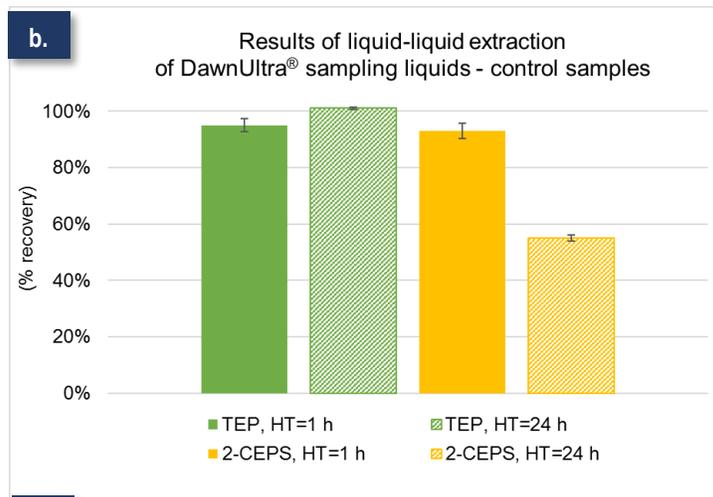
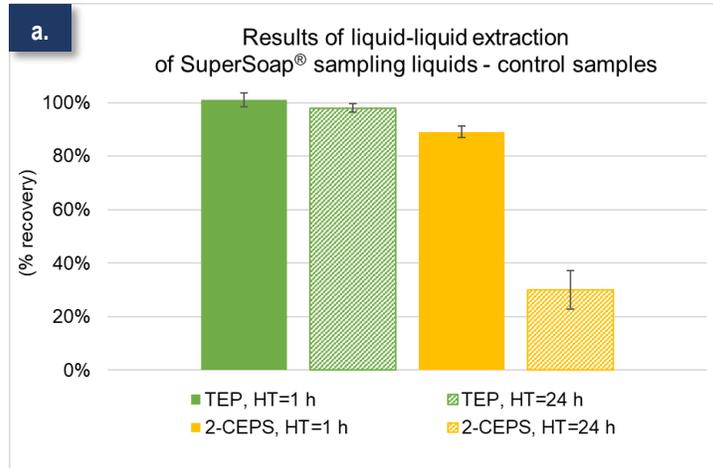
The wet-vacuum method assessment results are discussed in the order of the method development/proof of concept steps in Phase I, utilizing the prototype wet-vacuum system; Phase II, optimization of wet-vacuum method using two commercially available wet-vacuum systems and Phase III, the demonstration of the optimized commercial wet-vacuum system. Each phase has in common that the collected sample liquid should be analyzed based on validated analytical protocols. All analytical methods (Table 4-1) for the analysis of organic solvent-based and water-based sampling agents were verified prior to the collection of any wet-vacuum samples. These results are summarized in Section 5.1.

### 5.1 Analysis of Sampling Liquids

The wet-vacuum methodology was tested in two wetting solvent configurations: organic solvent (IPA) and aqueous solution. As discussed previously, the resulting sampling liquids were either directly analyzed using the appropriate analytical method (Section 4.4) or extracted using a straightforward LLE procedure (Section 4.2.2).

The ability to directly analyze the IPA sampling liquid was considered one of the main advantages of the wet-vacuum sampling method that was developed in this study, primarily due to the ease of downstream treatment post-sampling. There was no extraction step, and the sample preparation for analysis was limited to dilution of samples and addition of surrogate or IS compounds, when applicable (Section 4.3). The instrumental methods used for direct analysis of IPA (GC/MS, GC/FID; Table 4-1) were selected based on the general applicability/compatibility of target chemicals with IPA, accessibility, and the cost of sample analysis. All phenol-containing sampling liquids were directly analyzed using a spectrophotometric method that is used for drinking, surface, and saline waters, as well as domestic and industrial wastes [11]. The results of direct analysis of simulated liquids samples that were submitted to the analytical laboratories prior to testing were all above 90% of the amount of chemical spiked into the solvent. Target analyte-specific results are given in Appendix A (Tables A-8 through A-10).

Apart from the above-mentioned analysis of phenol, LLE procedures were necessary for preparation of water-based sample liquids for instrumental analysis. The LLE procedures were based on the fundamental phenomenon of the partition of the analyte between two immiscible (aqueous and organic) phases, one being the sample liquid and the other the organic extraction solvent. For TEP, the extraction was performed with addition of phosphate buffer to adjust the pH to a range where the analyte is nonionized and more easily migrates into the organic phase. Sodium chloride was added to salt out the surfactant from some of the aqueous SL solutions. Target analyte-specific recoveries of the optimized extraction method are provided in Appendix A (Table A-7). A summary of the results for the 1-hour and 24-hour holding times from control sample preparation (a simulated sample liquid) to extraction for these water-based samples is presented in Figure 5-1. A 24-hour HT was tested only for chemicals that were evaluated during operational-scale testing to obtain initial information on stability of the sample liquid from the wet-vacuum process. During field operations, it is likely that the sample handling time prior to analysis would be at least 1 day, equivalent to overnight shipping and a 1-day analytical turnaround time (meaning same-day extraction and analysis by the analytical laboratory). Results for nitrobenzene extractions from water and Tween®-20 (HT = 1 hour) are given in Table A-7 (Appendix A).



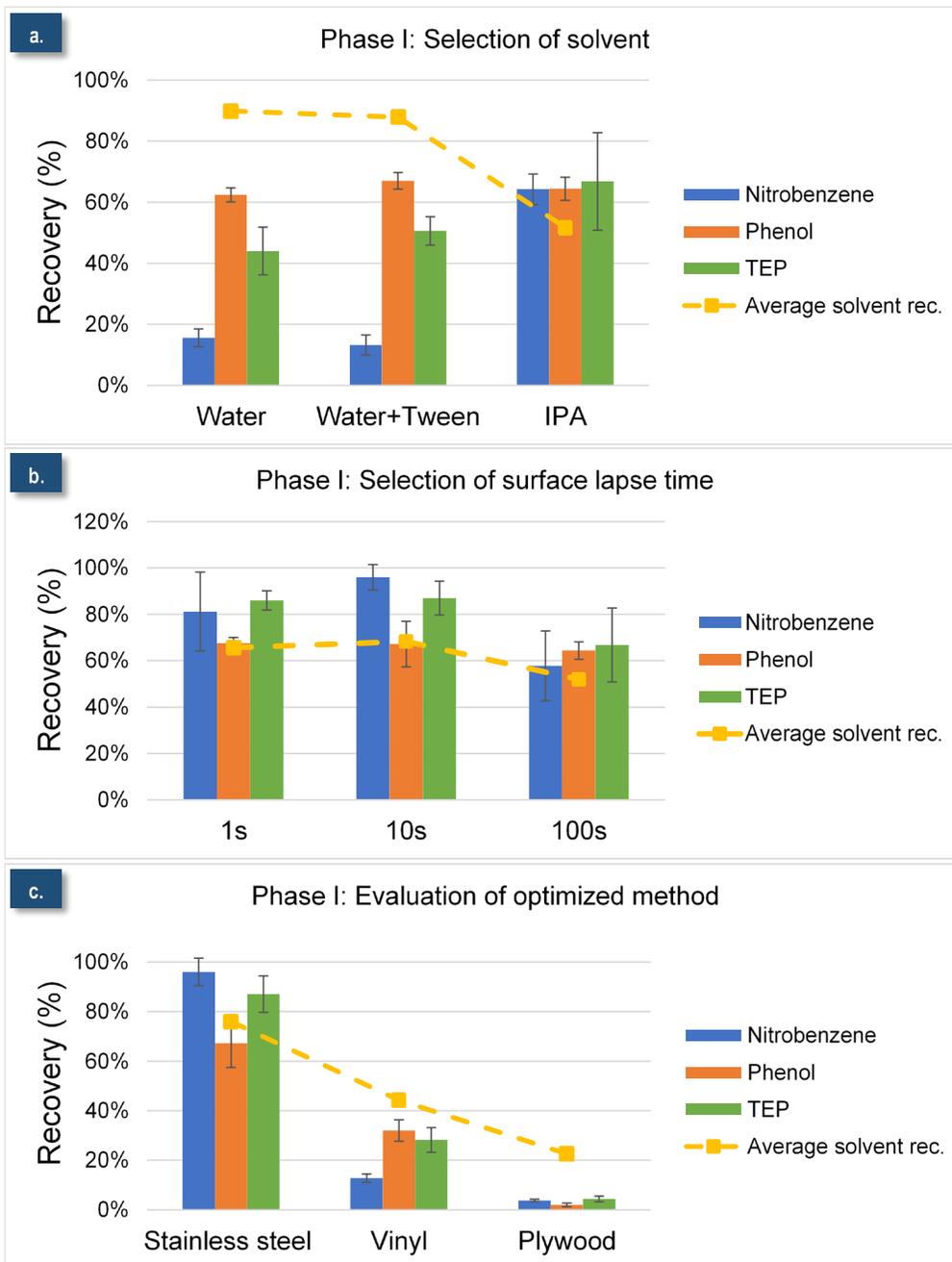
**Figure 5-1. Results from LLE of TEP and 2-CEPS in control samples of aqueous sample liquids; (a) SuperSoap®-water LLE, (b) Dawn Ultra®-water LLE, (c) water LLE; HT, analytical HT defined as from control sample preparation to extraction.**

The results show that LLE method extracted more than 90% of target analytes from all types of water-based wetting agents. The lower recoveries of 2-CEPS in all aqueous sampling liquids types analyzed with a 24-hour HT emphasizes the importance of liquid sample preservation immediately after collection to ensure stability of the target analyte prior to extraction. The LLE method is relatively simple and inexpensive. However, the LLE was also time-consuming and involved the use of relatively hazardous solvents (dichloromethane, hexane). Another drawback was that the solvent choice must be optimized for each analyte to be sampled using the wet-vacuum method, even for chemicals with similar physicochemical properties. Therefore, the LLE can never be specific to a particular sampling liquid, as the target chemical polarity will determine the ability to extract the solvent from the wetting agent matrix, making the procedure not easily expandable to possible further automation. Moreover, other chemicals co-collected during wet sampling may affect the effectiveness of the LLE procedure. In this laboratory study, all samples were extracted within 1-hour post-collection. Hence, the addition of preservatives to samples that contained 2-CEPS (e.g., glacial acetic acid and sodium chloride recommended for preservation of aqueous samples that contain CWAs [16]), was not considered.

## **5.2 Proof-of-Concept and Initial Optimization of Surface Sampling Method – Phase I**

The target chemicals were characterized by various degrees of solubility in water. Hence, the selection of the most appropriate, and ideally universal to all chemical–surface combinations, wetting condition was considered one of the first and most important steps of the method optimization. In the initial test, 100 mL was chosen as the wetting volume for a 926 cm<sup>2</sup> surface area, and 100 seconds was chosen as the initial wetting agent lapse time on the surface. Testing was performed on reference materials (stainless steel) using the wet-vacuum prototype system. The wetting solvent selection experiment results are summarized in Figure 5-2a, and test-specific results are given in Appendix A.

Recoveries of the wetting agent were the highest for the water-based agents (90%) while only 50% of the IPA was recovered in part due to evaporative IPA losses on the surface and within the prototype wet-vacuum system (Figure 5-2a). Nevertheless, the IPA wetting agent offered the highest recoveries of all target analytes, on average >60% of initial surface loading for chemicals that represented all solubility classes. Since it was also determined to be the easiest to handle because sample preparation was not needed prior to analysis, it was chosen to subsequently select the optimal lapse time (time between wetting and wet-vacuuming of the surface). Despite differences in average solvent recoveries between tests (Figure 5-2b), there was no statistically significant difference in chemical recovery ( $p=0.12$ ) for three lap times tested (1-100 sec). Then, an optimal amount of solvent for wetting the test surfaces was determined (using water) in a series of gravimetric tests; no significant difference was observed in recovery for 1 and 10 s lapse times (average 66-68%;  $p=0.35$ ), the average recovery for 100 s lapse time was lower at average 52% ( $\pm 2.7\%$  SD). The 10-sec long lapse time was considered a reasonable amount of time between wetting agent application and sampling while 50 mL of wetting solvent volume was considered to provide good surface coverage without excessive runoff (which occurred for the 100-mL wetting volume). This conservative amount of solvent was considered for the more volatile wetting agent such as IPA. Consequently, these parameters (lapse time = 10 sec; 50 mL wetting volume) were selected for evaluation of the laboratory-scale wet-vacuum method on three building materials of different porosities and permeabilities: stainless steel (SS), vinyl flooring (VF), and plywood (PW). The recoveries from different materials are shown in Figure 5-2c.



**Figure 5-2. Selection of operational parameters of laboratory-scale wet-vacuum sampling. (a) Selection of wetting solvent, (b) selection of lapse time for IPA, and (c) results of wet-vacuum sampling of various test surfaces using 50 mL IPA and LT = 10 s.**

These results clearly indicate that surface affinity, porosity and permeability are governing factors that affect recovery of both wetting solvent and target chemicals. An additional series of tests to improve

recovery of target chemicals from plywood was performed; IPA wetting solvent volumes of up to 100 mL per coupon were tested. Because no statistically significant improvement was observed in the sampling efficiency of plywood using larger volumes of IPA (data not shown), the permeable material was excluded from the next stages of experimental testing. A smooth and relatively nonporous laminate flooring material was used instead during the initial evaluation of commercial vacuum cleaners for wet-vacuum sampling (Phase II, section 5.3.1). Shorter lapse times (less than 1 s) would potentially have overcome some of the low wetting agent recoveries for plywood. However, this should be balanced against the specific time for a chemical to dissolve into the applied wetting solvent.

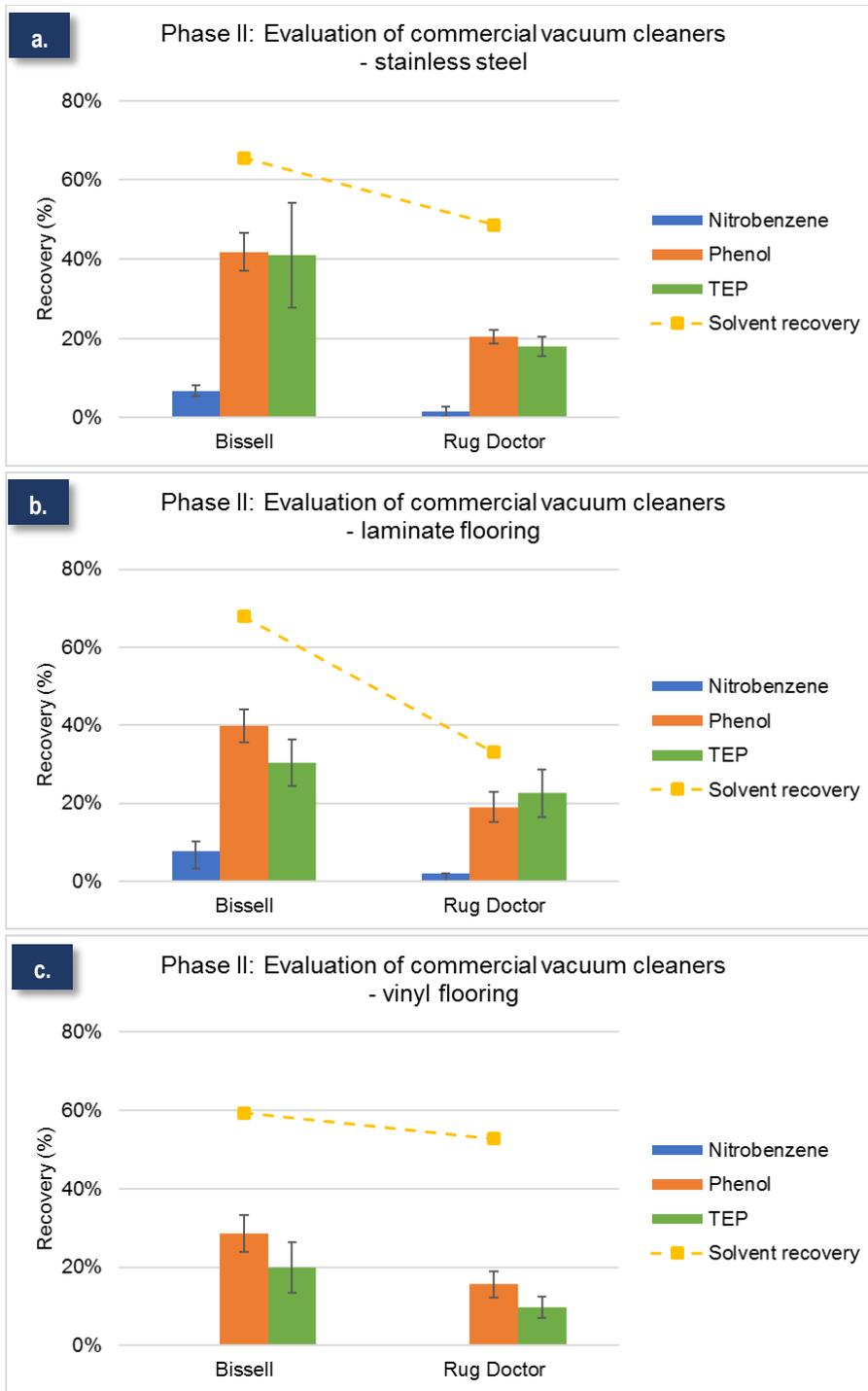
### **5.3 Initial Evaluation of Commercial Wet-Vacuum Cleaners – Phase II**

The commercial vacuum cleaners used in the initial evaluation of wet-vacuum sampling of chemicals were previously successfully deployed for environmental sampling of biological threats [17]. Both units are designed for cleaning tough surface stains and, hence, were expected to effectively lift contamination from surfaces and efficiently collect the wetting agent.

#### **5.3.1 Comparison of commercial wet-vacuum cleaners**

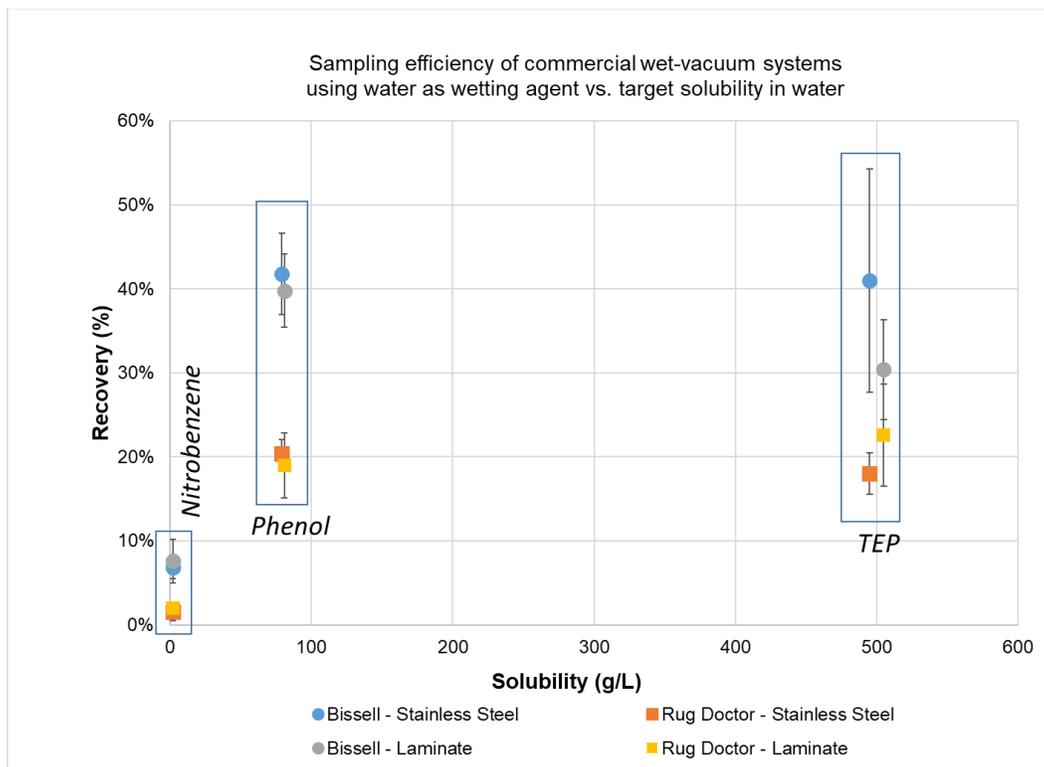
The initial wetting liquids selected for the commercial wet-vacuum systems testing were IPA and water. Due to large IPA losses observed during initial commercial wet-vacuum testing (data not shown), the sampling performance of these wet-vacuum units was initially tested using water only. The results for all chemical-material type commercial vacuum cleaners tested are shown in Figure 5.3. Sample-specific results are given in Appendix A (Tables A-20 through A-28).

The Bissell unit offered better sampling efficiency for all classes of chemicals and all types of surfaces tested (Figure 5-3). The unit was more efficient at collecting the wetting solvent, 59-68%, depending on the type of surface sampled, outperforming the Rug Doctor cleaner (33-53%) on the same type of surfaces.



**Figure 5-3. Results of the initial evaluation of commercial vacuum cleaners for wet-vacuum sampling of various test surfaces; sampling was performed using 50 mL of water, LT=10 s; material types: stainless steel (a); laminate flooring (b); vinyl flooring (c)**

Like the prototype testing, one of the most important factors influencing the sampling efficiency by commercial cleaners was the water solubility of the chemical. Figure 5-4 shows the chemical recovery achieved during water-based wet-vacuum sampling using Bissell and Rug Doctor cleaners as a function of the solubility of the chemical in water. The very low recovery of very slightly water-soluble nitrobenzene (Table 3-2) indicates that the solubility of the chemical in water is likely to be a limiting factor for collection efficiency during water-based wet-vacuum sampling. In the case of nitrobenzene, the mass delivered to the medium size coupon was 29 mg which, if recovery of nitrobenzene in water as the wetting solvent was 100%, would have yielded a 0.60 mg/mL sample concentration. This concentration is a factor of 3.2 below the 1.9 g/L solubility of nitrobenzene in water. Similar ratios of solubility over theoretical sample concentration are much larger for phenol and TEP, namely 160 and 914, respectively. The rate at which chemicals dissolve in water is expected to slow down when the solubility concentration is approached as is the case for nitrobenzene. Hence, a longer contact time of water as the wetting agent on the surface may have improved the recovery for nitrobenzene. One other approach to improve on recovery may have been a repeated application of the wetting agent on the same surface area. Neither was further investigated in this study considering the high recoveries using IPA as the wetting agent.



**Figure 5-4. Results of the initial evaluation of commercial vacuum cleaners for wet-vacuum sampling of various test surfaces as a function of the solubility of the chemical in water**

Based on solvent and chemical recovery results using water as the wetting solvent, the focus of the next experimental phase of testing was on improving the efficiency of IPA-based sampling, which offered better chemical recoveries across different levels of water solubility, as demonstrated in the prototype wet-vacuum testing (Figure 5.2a). Since the IPA solvent recovery appeared to be an important factor

contributing to overall sampling efficiency, the commercial unit-based IPA sampling was further optimized using the Bissell cleaner only, as described in Section 5.3.2.

### 5.3.2 Optimization of the commercial wet-vacuum cleaner-based sampling

The last step of the wet-vacuum sampling optimization using a commercially available unit included addition of a pre-sampling conditioning step of the wet-vacuum unit (pre-rinse) and a post-sampling rinse step of the wet-vacuum unit (post-rinse), both performed with IPA as the wetting solvent. Results of this optimization step are listed in Appendix A (Table A-29 and A-30). The pre-rinse step was designed to mitigate solvent evaporation related to losses of IPA; as observed during the initial stages of wet-vacuum sampling when the system was not pre-wetted (i.e., “primed”) with the IPA solvent. The optimal volume of solvent for the prewetting step was 50 mL of IPA for the test Bissell unit. A 100 mL prewetting volume provided similar improvement in absolute recovery of IPA (data not shown) but resulted in significant carryover of solvent from this pre-rinse step into the dirty liquid tank. The post-rinse volume selected for further testing was 100 mL, aspirated directly from a clean beaker containing IPA after sampling. A post-rinse volume of 100 mL was chosen over a 200-mL rinse to maintain a more conservative final sample volume. Additional chemical mass balance tests, performed during operational-scale testing (Phase III), demonstrated that the amount of chemical remaining in the vacuum cleaner system after application of the 100-mL rinse was relatively minimal, about 10% of the total mass of chemical recovered during wet-vacuum sampling (results from mass balance tests are discussed in Section 5.4.1.1).

### 5.3.3 Wipe sampling

The last test performed before the operational testing of the wet-vacuum method was related to the wipe sampling method development. The surface wipe sampling method was used in a direct comparison with the wet-vacuum method that had been developed. As discussed before, the wipe-based method was adopted from established methods used for sampling CWAs [9]. Results are given in Table 5-1.

**Table 5-1. Results for Wipe Sampling and Extraction Optimization Tests for 2-CEPS and TEP**

Target Chemical Concentration (mg)	Control Spike Recovery		Wipe Spike Recovery		Chemical Recovery from Wipe Sampling Method*				
	Mass (mg)	% Rec.	Mass (mg)	% Rec.	Mass (mg)	STD (mg)	% Rec.	STD (%)	RSD (%)
<b>2-CEPS/Stainless Steel</b>									
2.9	2.8	100	2.9	98	2.00	0.014	70	0.49	0.71
<b>TEP/Stainless Steel</b>									
2.7	2.4	89	1.9	71	0.55	0.54	21	20	96
*Per test area of 10 cm x 10 cm (100 cm <sup>2</sup> ), n = 3.									

The sampling of 2-CEPS from nonporous reference material (stainless steel) was characterized by good average recovery (70% ± 0.49% SD of the theoretical target) and reproducibility (%RSD <1%) (Table 5-1). However, the average recovery of TEP with wipe-based surface sampling was only 21% of the theoretical concentration delivered onto the stainless-steel surface and was characterized by large intra-test variation (RSD = 96%; n = 3) (Table 5-1). The high recovery of a control wipe sample that was spiked with

the target chemical prior to extraction (71% of target concentration) suggests that the wipe extraction method (as described in Section 4.2.1) was effective for extracting TEP from the gauze wipes used in sampling. As the focus of this study was development of novel wet-vacuum cleaner-based sampling approaches, the wipe-based method was not further improved for TEP recovery. The wipe sampling method may improve for TEP recovery when considering a different type of wipe and/or wetting solvent.

## 5.4 Operational-scale Testing of Wet-Vacuum Sampling Efficiency – Phase III; Medium-size Area

The operational-scale research addressed multiple aspects of discrete wet-vacuum sampling applications: the overall sampling effectiveness (Section 5.4.1) at the default surface concentration and at lower surface concentrations (Section 5.4.2); the direct comparison to the surface concentration via extraction of applied amounts (Section 5.4.3); and the wet-vacuum methods comparison to conventional wipe-based technique (Section 5.4.4)

### 5.4.1 Medium-size area sampling at default surface concentration

Medium-sized area (929 cm<sup>2</sup>) sampling focused on validation of the wet-vacuum method for single-location sampling. The sampling efficiency achieved for nonporous reference material (stainless steel) at 100% chemical surface loading (equivalent to approximately 26-28 mg/coupon or 0.28-0.30 g/m<sup>2</sup>) was considered the method performance baseline, compared to the wet-vacuum sampling effectiveness for one other material, several chemical challenge levels, and to the performance of the single-area wipe sampling method. The results for the medium-sized area sampling of a nonporous (stainless steel) and a more permeable, yet nonporous, flooring material (vinyl) are provided in Tables 5-2 and 5-3.

**Table 5-2. Test Results of Medium-Sized Area Wet-Vacuum Sampling of 2-CEPS from a Nonporous and Permeable Material at the Default Surface Concentration**

2-CEPS: Medium-Size Area Wet-Vacuum Sampling											
Material	Wetting Agent	Surface Loading*	Chemical Recovered			Wetting Liquid Recovered			Proc. Blank	Sampling Efficiency	
			Mean	±SD	%RSD	Mean	SD	%RSD		(mg)	%
			(mg)			%					
Stainless steel	IPA	100%	21.8	1.5	7.3%	71	3.7	5.2	<LOQ	77	5.1
	WAT	100%	0.85	0.22	26%	89	1.9	2.1%	<LOQ	3.0	0.8
	DUW	100%	12.6	0.58	4.6%	83	1.3	1.6%	<LOQ	45	2.1
	SSW	100%	8.86	1.01	11%	84	1.3	1.5%	<LOQ	31	3.6
Vinyl flooring	WAT	100%	0.22	0.10	43%	88	3.6	4.1	<LOQ	0.8	0.3
	DUW	100%	7.60	0.97	13%	84	4.5	5.4	<LOQ	27	3.4
	SSW	100%	3.66	0.20	5.5%	86	4.7	5.4	<LOQ	13	0.7

IPA – isopropyl alcohol, WAT – DI water, DUW – Dawn Ultra®-water, SSW – SuperSoap®-water;  
 \*Nominal surface loading (100%) = 28 mg/coupon for 2-CEPS

**Table 5-3. Test Results of Medium-Sized Area Wet-Vacuum Sampling of TEP from Nonporous and a Permeable Material**

TEP: Medium-Size Area Wet-Vacuum Sampling											
Material	Wetting Agent	Surface Loading*	Chemical Recovered			Wetting Liquid Recovered			Proc. Blank	Sampling Efficiency	
			Mean	±SD	%RSD	Mean	±SD	%RSD		(mg)	%
			(mg)			(%)					
Stainless steel	IPA	100%	18.7	0.93	4.7%	72	1.7	2.4%	<LOQ	73	3.6
	WAT	100%	21.6	1.1	5.2%	84	1.7	2.0%	<LOQ	84	4.3
	DUW	100%	21.6	1.3	6.1%	74	2.6	3.5%	<LOQ	84	5.1
	SSW	100%	23.0	0.52	2.3%	72	4.4	6.2%	<LOQ	89	2.0
Vinyl flooring	WAT	100%	12.8	1.21	9.5	75	12.6	16.9%	<LOQ	50	4.7
	DUW	100%	11.3	1.84	16.3	77	4.5	5.8%	<LOQ	44	7.2
	SSW	100%	13.9	1.22	8.7	71	3.3	4.6%	<LOQ	54	4.7

IPA – isopropyl alcohol, WAT – DI water; DUW – Dawn Ultra®-water; SSW – SuperSoap®-water;  
 \*Nominal surface loading (100%) = 26 mg/coupon for TEP

The optimized method performed well during the sampling of high surface concentration of chemicals. The sampling efficiency,  $\chi$ SE, for 2-CEPS (Table 5-2) varied depending on the wetting agent from 0.8% (DI-water, vinyl flooring) to 77% (IPA, stainless steel) and from 44% (DI-water with Dawn Ultra®, vinyl flooring) to 89% (DI-water, stainless steel) for TEP (Table 5-3). All default surface concentration (“100% surface loading”) tests were characterized by high reproducibility, with <30% RSD for intra-test variation. For 2-CEPS, wet-vacuum sampling efficiencies increased significantly in the presence of Dawn Ultra® or SuperSoap® additives (45 and 31%, respectively) compared to DI-water without additives (3.0%) but efficiencies never reached the IPA-based wet-vacuum sampling efficiency (77 %). For TEP, the presence of a surfactant in a water based wetting solution did not improve the wet-vacuum efficiency (84 and 89% for Dawn Ultra® and SuperSoap®, respectively, versus 84% for DI water). TEP is readily dissolved in water and may not require the additional surfactant to remove it from the surface. As also observed during the Phase I prototype wet-vacuum sampling effort, the efficiency for vinyl flooring material is noticeably lower than the efficiency compared to nonporous stainless steel.

**5.4.1.1 Residual chemical on wet-vacuumed surface and in wet-vacuums unit**

In supplementary tests, the target chemicals remaining on the surface after wet-vacuum sampling were confirmed to be ranging from non-detect to negligible (0.004% of total recovered amount) using conventional wipe sampling (Appendix A, Table A-31 and A-32). The ability to dissolve either chemical into IPA on the stainless-steel surface is highly efficient and occurs within the time (10 sec) between wetting agent application and wet-vacuums. However, a significant amount of both TEP and 2-CEPS chemical remained in the vacuum cleaners, as indicated by analysis of multistep rinses performed with clean IPA immediately after conclusion of sampling. The first additional 100-mL IPA rinse (#1) recovered an additional 12.7% of 2-CEPS and 6.2% of TEP, the second additional 100-mL IPA rinse (#2) recovered an additional 2.5% and 1.2%, respectively. These data suggest that these two-additional wet-vacuum unit rinses would improve the target chemical recovery by up to approximately 15% and 8% for 2-CEPS and TEP,

respectively. However, the final sample volume would also increase from 150 mL to 350 mL, i.e., by over 100%. Such an increase would worsen the analytical method detection limits.

#### 5.4.2 Medium-size area sampling at low (below default) surface concentration

The results for the medium-sized area sampling at lower surface loadings (1 to 20% of default surface concentration) are provided in Tables 5-4 (2-CEPS) and 5-5 (TEP). These tests were conducted using IPA and one water based wetting agent. Results from the default surface concentration tests (Tables 5-2 and 5-3) indicated that water without surfactant is less efficient than either of the two water-surfactant wetting agents. The Dawn Ultra® - water wetting agent was selected over the SuperSoap® - water wetting agent considering its better performance for sampling of 2-CEPS.

**Table 5-4. Test Results of Medium-Sized Area Wet-Vacuum Sampling of 2-CEPS at Low Concentrations**

2-CEPS: Medium-Size Area Wet-Vacuum Sampling - Low surface concentration threshold											
Material	Wetting Agent	Surface Loading*	Chemical Recovered			Wetting Liquid Recovered			Proc. Blank	Sampling Efficiency	
			Mean	±SD	%RSD	Mean	±SD	%RSD		(mg)	%
			(mg)			(%)					
Stainless steel	IPA	20%	3.5	0.07	2.1%	72	2.4	3.4%	<LOQ	60	1.2
		10%	1.3	0.06	4.3%	71	0.35	0.50%	<LOQ	45	2.0
		2%	0.0015 <sup>(J)</sup>	0.00010	46%	69	7.7	11%	<LOQ	0.26	0.12
		1%	0.00010 <sup>(J,B)</sup>	0.00003	33%	71	0.80	1.1%	<LOQ	0.32	0.11
	DUW	20%	1.69	0.27	16%	83	6.4	7.7%	<LOQ	29	4.8
		10%	0.59	0.070	12%	74	2.02	2.7%	<LOQ	20	2.4
		2%	0.00090	0.0004	42%	74	1.67	2.3%	<LOQ	0.15	0.06

IPA – isopropyl alcohol, WAT – DI water, DUW – Dawn Ultra®-water, J – estimated value, data reported were below lowest point of the calibration curve, B – target compound detected in associated procedural blank  
\*With respect to default nominal surface loading (100%) = 28 mg/coupon for 2-CEPS

**Table 5-5. Test Results of Medium-Sized Area Wet-Vacuum Sampling of TEP at Low Concentrations**

TEP: Medium-Size Area Wet-Vacuum Sampling - Low surface concentration threshold											
Material	Wetting Agent	Surface Loading*	Chemical Recovered			Wetting Liquid Recovered			Proc. Blank	Sampling Efficiency	
			Mean	±SD	%RSD	Mean	±SD	%RSD		(mg)	%
			(mg)			(%)			(mg)		%
Stainless steel	IPA	20%	0.66	0.36	53%	74	2.8	3.8%	<LOQ	13	7.0
		10%	0.027	0.0098	36%	75	4.0	5.8%	<LOQ	1.1	0.39
		2%	0.0060 <sup>(J)</sup>	0.0028 <sup>(J,B)</sup>	47%	75	2.0	2.6%	<LOQ	1.2	0.55
		1%	0.0015 <sup>(J)</sup>	0.0014 <sup>(J,B)</sup>	94%	74	2.8	3.8%	<LOQ	0.56	0.53
	DUW	20%	1.20	0.30	25%	85	4.8	5.7%	<LOQ	24	5.88
		10%	0.0053	0.0012	22%	86	5.2	6.0%	<LOQ	0.21	0.05
		2%	0.0022	0.00097	43%	85	2.4	2.9%	<LOQ	0.44	0.19

IPA – isopropyl alcohol, WAT – DI water; DUW – Dawn Ultra®-water; J – estimated value, data reported were below lowest point of the calibration curve; B – target compound detected in associated procedural blank  
 \*With respect to default nominal surface loading (100%) = 26 mg/coupon for TEP

The analysis of average chemical recovery results from 1 to 20% of default surface loading tests suggests a limited applicability of wet-vacuum sampling for a low surface concentration of chemical, regardless of type of wetting solvent used or surface sampled (Tables 5-4 and 5-5). Sampling recoveries for 2-CEPS when using IPA as the wetting solvent dropped from 73% (100% default surface concentration, Table 5-2) to 45% at 10% of the surface concentration to less than 1% recovered at 1 or 2% surface concentrations. For 2-CEPS with water plus Dawn Ultra®, recoveries dropped from 45% (100% default surface concentration, Table 5-2) down to 20% recovered at 10% of the default surface concentration to less than 1% recovered at 2% of the default surface concentration. For TEP, the trend is similar with sampling efficiencies going down with lower surface concentrations sampled, namely, for the IPA wetting agent wet-vacuum sampling, recoveries decline from 75% (100% default surface concentration, Table 5.3) to 13% (at 20% of default surface concentration) to less than 1% recovered at all lower surface concentrations while with water plus Dawn Ultra® as the wetting agent, recoveries dropped from 84% (100% default surface concentration, Table 5-3) down to 24% at 20% of the default surface concentration to less than 1% at the lowest tested surface concentration (2% of default surface concentration). Further, the low-surface concentration tests had coefficients of variation reaching 100%, depending on analyte and surface concentration. Test-specific coefficients of variation were generally highest for experiments with lowest surface concentrations (1% and 2% of default surface loading), suggesting that further method optimization for a robust and reproducible wet-vacuum sampling concentration may be required. However, it is important to emphasize that low recoveries observed for the wet-vacuum sampling tests could also be a result of unanticipated evaporative losses of the chemicals from test surfaces for these low surface concentration tests. This concern is valid, considering the use of a diluted chemical solution application onto the surfaces using a volatile solvent (here, IPA) that may facilitate the evaporation of the targeted chemical. Section 5.4.3 describes the outcome of supplementary tests that alludes to such an enhanced evaporation process.

### 5.4.3 Surface concentration verification by direct extraction

Additional supplemental tests were conducted to confirm the surface concentration via direct extraction, allowing for verification of whether the observed losses in wet-vacuum sampling efficiencies at lower surface concentrations (Section 5.4.2) could be attributed solely to the loss in sampling efficiency of the wet-vacuum sampling method or to an inherent lower recovery of the targeted chemical when spiked onto a surface in a diluted solution due to accelerated evaporation of the chemical in the presence of a volatile solvent or a combination of both processes. Table 5-6 and 5-7 provide the data on the amount recovered of 2-CEPS and TEP, respectively, by direct extraction in the identified extraction agent as described in Section 3.9.1 and the associated control spike recovery efficiency.

**Table 5-6. Test Results for Direct Extraction of 2-CEPS from Stainless Steel**

2-CEPS: Medium-Size Area Direct Extraction Sampling										
Material	Extraction Agent	Surface Loading (mg) [% of default]	Chemical Recovered		Extraction Efficiency			Control Spike Recovered	Control Spike Recovery Efficiency	
			Mean	±SD	Mean	SD	%RSD		(mg)	%
			(mg)		(%)					
Stainless steel	Hexane	28.2, [100]	33.1	0.5	117	1.7	1.5			
	Hexane	5.64, [20]	4.6	N/A	82	N/A	N/A	0.90	94	N/A
	Hexane	2.82, [10]	1.9	N/A	66	N/A	N/A	0.45	94	N/A
	Hexane	0.56, [2]	0.002 <sup>(J)</sup>	N/A	0.4	N/A	N/A	0.09	90	N/A

WAT – DI water; DUW – Dawn Ultra®-water; SSW – SuperSoap®-water; J – estimated value, data reported was below lowest point of the calibration curve. N/A: Not applicable (single test)

**Table 5-7. Test Results for Direct Extraction of TEP from Stainless steel**

TEP: Medium-Size Area Direct Extraction Sampling										
Material	Extraction Agent	Surface Loading (mg) [% of default]	Chemical Recovered		Extraction Efficiency			Control Spike Recovered	Control Spike Recovery Efficiency	
			Mean	±SD	Mean	SD	%RSD		(mg)	%
			(mg)		(%)					
Stainless steel	Hexane	25.7, [100]	24.3	0.4	94	1.5	1.6			
	Hexane	5.15, [20]	0.08036 <sup>(J)</sup>	N/A	1.6	N/A	N/A	0.94	112	N/A
	Hexane	2.57, [10]	0.00385 <sup>(J)</sup>	N/A	0.16	N/A	N/A	0.50	117	N/A
	Hexane	0.51, [2]	0.00095 <sup>(J)</sup>	N/A	0.19	N/A	N/A	0.06	72	N/A

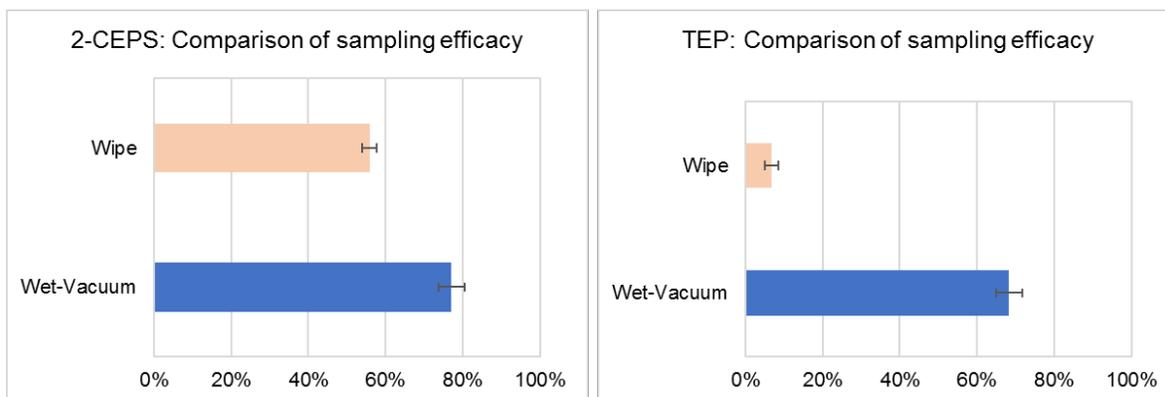
WAT – DI water; DUW – Dawn Ultra®-water; SSW – SuperSoap®-water; J – estimated value, data reported was below lowest point of the calibration curve. N/A: Not applicable (single test)

Control spike recoveries for both 2-CEPS and TEP were high (90-94% for 2-CEPS and 72-117% for TEP), indicating that extraction of these two chemicals from the hexane solvent is feasible and that prepared spiked diluted solutions are correct in absolute concentration. However, extraction efficiencies of these two diluted chemicals when applied to stainless-steel surfaces followed by a short contact time of 30

min were strongly dependent on the solution concentration, with the lowest efficiency noted for the lowest applied concentration. This dependence on concentration can be caused by a much poorer extraction efficiency for these chemicals from a surface at lower concentrations and/or by a lower delivery of the chemical due to an enhanced evaporation from surfaces of less volatile chemicals in the presence in a more volatile solvent. Without further research, neither of the phenomena can be verified. Nevertheless, the recovered amount is negatively biased, which will translate into lower recoveries when other sampling methods such as wipe sampling or wet-vacuum sampling are considered. The observed poorer wet-vacuum efficiency when sampling lower surface concentrations may be linked to this negative bias and would not reflect an inherently poor quality of the proposed sampling method.

#### 5.4.4 Comparison of wet-vacuum sampling to wipe-based sampling methods

As discussed previously, surface wipe sampling is one of the most common techniques used for assessing environmental surface contamination with chemicals. The wipe sampling method used in this study was a modification to an existing EPA reference method [9] and included selected modifications from a large wipe-sampling evaluation study performed by EPA NHRSC [3]. The method was compared to efficiency of the single coupon IPA-based wet-vacuum sampling of a smooth nonporous material (stainless steel), which was considered the performance baseline for the new method developed in the current study. Both sampling methods were deployed against the same chemical surface loadings (280-300 mg per square meter, or 100% default surface concentration target), but were deployed at different surface areas, as described in detail in Section 4.1. The sampling efficiency results for medium-size area wipe sampling against the results of the wet-vacuum sampling using IPA are summarized below in Figure 5-5.



**Figure 5-5. Comparison of sampling efficiency of SA wet-vacuum and wipe-based method for 2-CEPS (a) and TEP (b); WS – wipe sampling, WV – wet-vacuum sampling**

The wipe sampling method was outperformed by the wet-vacuum sampling method, offering a statistically significant improvement in sampling efficiency ( $p=0.00067$  and  $p=0.000012$  for 2-CEPS and TEP tests, respectively). As mentioned previously, the wipe sampling method was adopted from CWA sampling methods and, therefore, had a limited applicability for sampling of TEP from surfaces. At the same time, the IPA-based wet-vacuuming method was deployed in an identical (nonchemical-specific) configuration, suggesting a much broader applicability to different classes of chemical compounds without chemical-specific optimization requirements.

## 5.5 Operational-scale Testing of Wet-Vacuum Sampling Efficiency – Phase III; Large-size (Composite) Area

Due to relatively low recovery of target chemicals at low surface concentration as described in Section 5.4.2, the large area evaluation was performed only at the 100% default contamination level (26-28 mg/coupon) on the nonporous stainless-steel material. The theoretical sample volume for five coupons was expected to be approximately double the total sample liquid volume collected during a single coupon sampling, namely, 5 x 50 mL of wetting solvent + 100-mL rinse (equals 350 mL) versus 1 x 50 mL of wetting solvent and 100-mL rinse (equals 150 mL). Wetting liquid recoveries and chemical specific recoveries are provided in Table 5-8.

**Table 5-8. Test Results of Large-Sized Area Wet-Vacuum Sampling of 2-CEPS and TEP from Stainless-steel Material (n=3)**

Test Material	Large Size Area Wet-Vacuum Sampling												
	High surface concentration threshold												
	Chemical	Wetting Agent	Surface Loading	Chemical Recovered			Wetting Liquid Recovered			Proc. Blank	Sampling Efficiency		
				Mean	±SD	%RSD	Mean	SD	%RSD		(mg)	%	SD
				(mg)			(%)						
Stainless steel	2-CEPS	IPA	100%	95.7	1.1	1.1%	47	3.3	7.1%	<LOQ	68	0.8	
	TEP	IPA	100%	99.8	1.0	1.0%	52	2.5	4.7%	<LOQ	78	1.0	

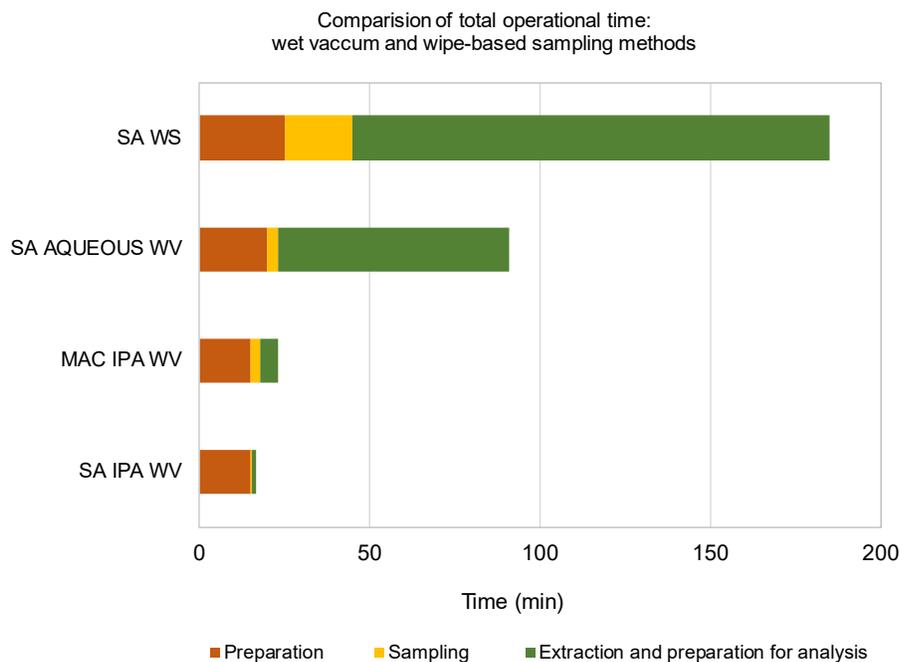
The losses of IPA as observed in the dirty liquid tank during composite sample collection of 2-CEPS and TEP caused a much lower solvent recovery, average  $170 \pm 20$  mL or approximately 50% of empirically predicted volume. The losses in recovered IPA were associated with the evaporation of IPA during the prolonged sampling. However, this evaporation of IPA solvent in the dirty liquid tank did not significantly affect the method performance with the overall sampling efficiency of 68% for 2-CEPS and 78% for TEP, with excellent method reproducibility (RSD <1%; n=3) between sample-composites collected. These results indicate that composite wet-vacuum sampling of larger areas is feasible with equal recoveries expected in comparison to a single medium-size coupon wet-vacuuming recovery.

### 5.5.1 Operational considerations for large area composite sampling

The wet-vacuum sampling method was expedient to perform. The sampling of a 5-point composite – including spraying of IPA onto the surface – took approximately 5 minutes, with the average sampling time per 929 cm<sup>2</sup> square area estimated as  $68 \pm 0.3$  s. The only disadvantage of this composite sampling method was a moderate heating of the wet-vacuum cleaner unit as observed during sampling, which would cause a possible volatilization of the flammable IPA solvent (IPA flash point [open cup] is 12 °C). The temperature of the wet-vacuum cleaner exhaust, identified as the unit temperature ‘hot spot’, reached approximately 53.4 °C after five minutes of the vacuum cleaner use, but the temperature of the dirty liquid tank, which contains a large amount of IPA, remained at < 20 °C, likely due to the evaporative cooling effect during large-area sampling and release of the liquid into the dirty liquid tank. A more detailed characterization of the vacuum temperature profile (performed prior to the large scale-testing) is discussed in Section 5.7.2.

## 5.6 Operational time comparisons

As part of this project, it was important to assess not only the test method feasibility for hazard mapping and identification of various levels of contamination zones but also the time and cost-effectiveness of proposed wet-vacuum approaches. The comparative analysis of the total operational time (TOT) performed for each method included all major operational steps of the complete sampling procedure, including the preparation for sampling step (consisting of preparation of sampling kits for wipe sampling methodology, and preparation of cleaners and wetting agents for wet-vacuum based methodologies). The TOT estimates were normalized for a surface area of 929 cm<sup>2</sup>, a minimum area evaluated for sampling efficiency using wet-vacuum sampling methodologies. Collection of 10 SA wipe samples would be necessary and the MAC method would allow for one composite sample characterization of five medium-size (929 cm<sup>2</sup>) areas. Results of TOT comparison between wipe and wet-vacuum-based sampling methods are shown in Figure 5-6 and suggest a large TOT reduction for the wet-vacuum methodology versus the wipe-based methodology. A noticeable 8- to 10-fold reduction in TOT observed for IPA-based wet-vacuum methodologies indicated that the same-solvent sampling and analysis should be considered as a critical factor for improving analytical turnaround times, followed by sample-compositing during sampling.

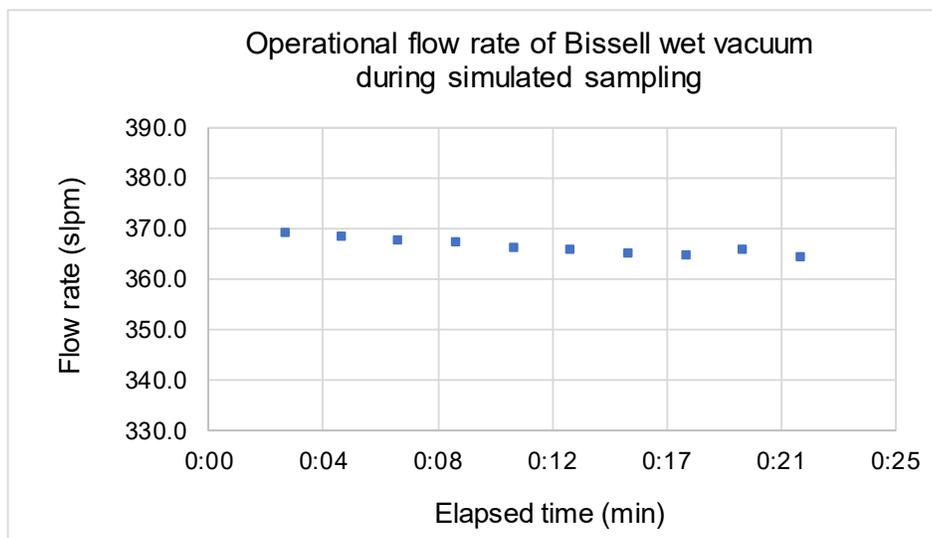


**Figure 5-6. Comparison of total operational time for wet-vacuum and wipe-based sampling methods; WS – wipe sampling; WV – wet-vacuum sampling; SA – single area; MAC - multiarea composite.**

## 5.7 Supplementary Characterization Tests

### 5.7.1 Bissell wet-vacuum airflow

The wet-vacuum flow rate of the Bissell wet-vacuum system that was tested was measured by installing a reference flow meter in the vacuum hose between the operational [sampling]-position nozzle and the body of the wet-vacuum (details in Section 3.10.2.1). The results of the operational flow rate measurements – collected at approximately two-minute intervals throughout the duration of continuous, 20-min long, wet-vacuum operation – are found in Figure 5-7.

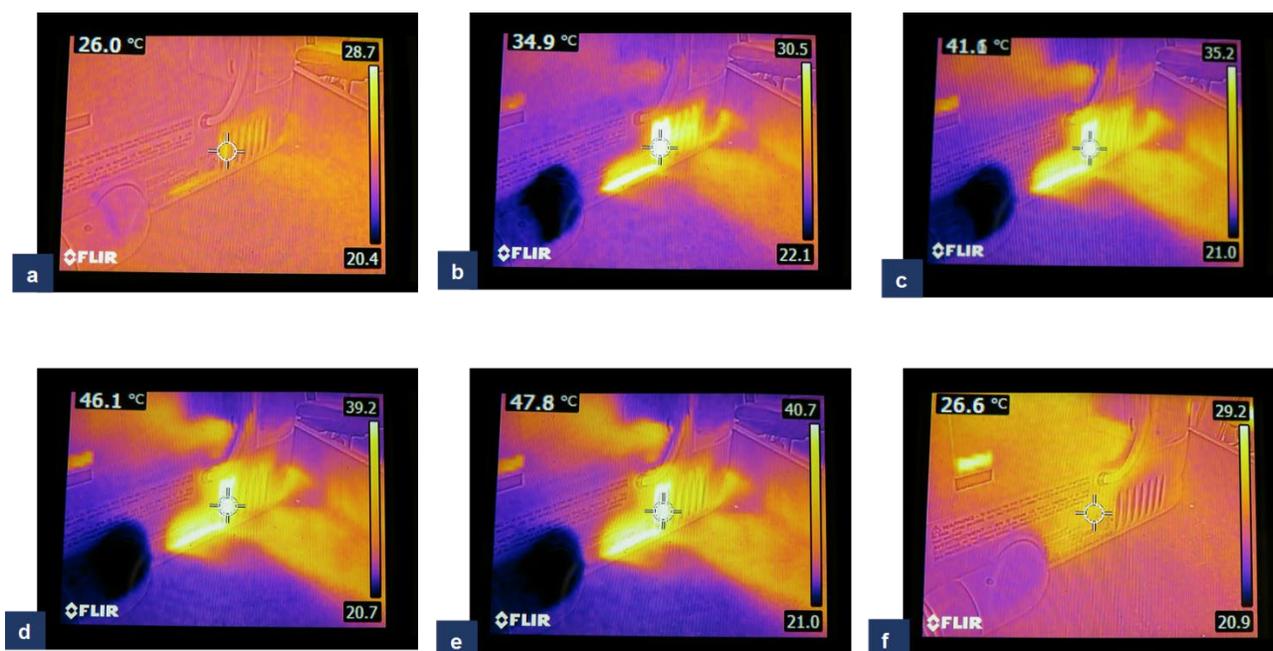


**Figure 5-7. Airflow monitoring of Bissell unit simulated large-area sampling; flow rate is reported at US EPA standard ambient sampling conditions of 101.325 kPa and 25 °C; slpm – standard liters per minute.**

The average flow rate was recorded at 366.3 standard liters per minute (slpm) with an SD of 1.6 slpm, with a slight (<2%) flow rate drop observed throughout the 20-min long operation, which suggests that robust and stable wet-vacuum airflow conditions were attainable for both SA and MAC methods.

### 5.7.2 Wet-vacuum temperature profile

During preliminary testing, the Bissell exhaust port, which is located near the vacuum motor, was identified as a possible “hot spot” during large-area sampling. The temperature of the Bissell unit exhaust port was thermally imaged throughout the duration of a simulated sampling test (with and without sampling of surface). Figure 5-8 shows the heating of the wet-vacuum system during the surface sampling with water as the wetting solvent. At the start of the test, the exhaust port temperature was approximately 26 °C (Figure 5-8a). After one minute of vacuum sampling, the temperature had risen to 34.9 °C (Figure 5-8b), and after two or three minutes the temperature was above 41 °C (Figure 5-8c and 5-8d, respectively). At the end of the sampling event, at approximately four minutes, the vacuum exhaust port temperature had reached 47.8 °C (Figure 5-8e); 15 minutes later, the vacuum port had cooled to 26.6 °C or close to the starting temperature). Internal components of the vacuum (pump and motor) were still at approximately 30 °C following the 15-minute cooling period (thermal imaging data not shown in Figure 5-8).



**Figure 5-8. Temperature monitoring of Bissell exhaust during simulated sampling. Sampling performed under the chemical hood. (a) start of vacuum sampling (t = 0 min); (b) after 1 min of vacuum sampling (t = 1 min); (c) after 2 min of sampling (t = 2 min); (d) after 3 min of sampling (t = 3 min); (e) at the end of sampling (t = 3 min 24 s); f, 15 min after sampling ended (t = 19 min 24 s).**

The simulated sampling described above was performed using only four medium-size coupons, the number of coupons that can be accommodated in the chemical hood during operational-scale testing and indicated that multiarea sampling quickly increases the temperature of the vacuum cleaner exhaust port. From an operational safety perspective, the most critical consideration was to determine if the wetting liquid holding tank or liquid waste collection tank temperatures were rising substantially during sampling, especially while working with a flammable solvent like IPA. The temperature of major vacuum cleaner components was monitored continuously for 20 min, with and without wetting solvent (water). Thermal images of all general vacuum components during this test indicated the wetting liquid holding tank and liquid waste collection tank were cooler than the exhaust port and the central vacuum system (Figure 5-9a). Remarkably, the liquid waste collection tank temperature fell below 20 °C during 20-min long sampling (Figure 5-9b). As the effect of the evaporative cooling was going to be even greater during IPA testing, the Bissell unit was deemed usable for IPA-based SA and MAC wet-vacuum sampling.

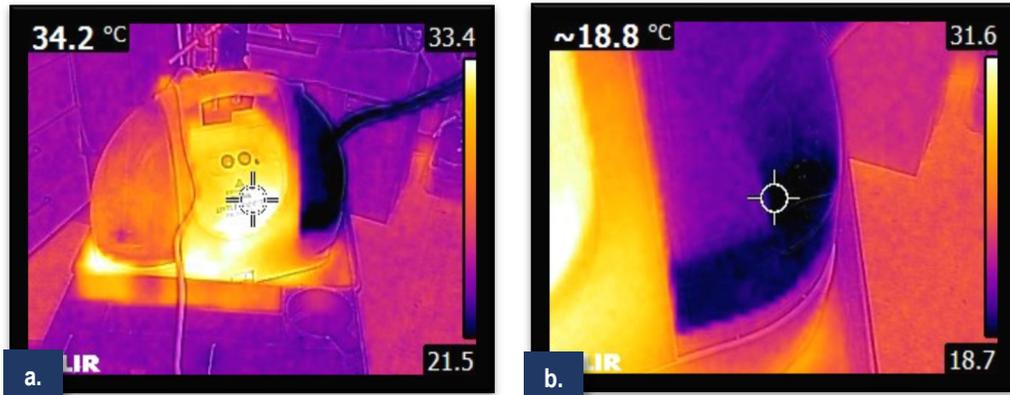


Figure 5-9. Temperature in various zones of Bissell vacuum cleaner after 20 min sampling using water. (a) Central system temperature near motor; (b) Liquid waste collection tank temperature.

## 6.0 Quality Assurance and Quality Control

### 6.1 Test Equipment Calibration

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

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

Equipment	Calibration/Certification	Expected Tolerance	Results
Thermometer	Compare to independent NIST thermometer (a thermometer that is recertified annually by either NIST or an ISO-17025 facility) value once per quarter.	$\pm 1$ °C	100%
Stopwatch	Compare to official U.S. time at time.gov every 30 days.	$\pm 1$ min/30 days	100%
Micropipettes	Certified as calibrated at time of use; recalibrated by gravimetric evaluation of performance to manufacturer's specifications every year.	$\pm 5\%$	100%
Scale	Certified as calibrated at time of use; calibration verified yearly by the AEMD Metrology Laboratory.	$\pm 1$ g	100%
Graduated cylinder	Certified by manufacturer at the time of use.	$\pm 1$ mL	100%
Solvent dispenser	Certified by manufacturer at the time of use; rechecked volume delivered using graduated cylinder prior to use.	$\pm 1$ mL	100%
Gas chromatography/mass spectrometer	5-point calibration prior to analysis; continuous calibration prior to each analytical run; recalibrate when continuous calibration fails acceptance criteria and/or after system maintenance; details in Section 4.4.	$\pm 20\%$ at mid-point	100%
Gas chromatography/flame ionization detector.	6-point calibration prior to analysis; continuous calibration prior to each analytical run; recalibrate when continuous calibration fails acceptance criteria and/or after system maintenance; details in Section 4.4.	$\pm 20\%$ at mid-point	100%

NIST – National Institute of Standards and Technology; ISO – International Organization for Standardization;

### 6.2 Data Quality Results for Critical Measurements

The following measurements were deemed critical to accomplishing project objectives:

- Surface concentration of target chemicals as determined by instrumental analysis
- Chemical concentration in the SL
- Chemical concentration in the SL or wipe extracts
- Contact time
- Lapse time
- Volume of wetting agent applied onto coupon surfaces

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

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

Critical Measurement	Target Value and Acceptance Criteria	Results
Contact/weathering time	30 min ± 1 min	All contact times within 30 min ± 1 min from spiking
Lapse time	10 min ± 30 s	All lapse times within 10 min ± 30 s from application of wetting solvent to sampling
Delivery of target surface concentration of chemical*	80–120% of target	All mean spike controls for decontamination tests were within acceptance criteria with coefficients of variation ≤30% between tests (all target chemicals); results are in Table 4-6.
Recovery of chemical from surface samples†	<30% coefficient of variation for identical test set	Selected low-concentration tests had >30% coefficient of variation for surface samples resulting from identical test set; test-specific results are in Tables 5-2 and 5-3.
Procedural blank	<5% of the analyte amount recovered from the positive control.	All procedural blank samples within acceptance criteria; all reported <LOQ; test-specific results are in Tables 5-2 and 5-3.
Solvent blank	<LOQ	All solvent blanks reported <LOQ
Volume of wetting solvent applied‡	± 20% of target volume	All wetting solvent volumes within acceptance criteria; results are summarized in Appendix A.
<p>*As determined by analysis of control spikes; criteria for recovery of chemical from surface.            †Optimized method used for operational-scale testing.            ‡Volumes that were delivered on the surface as determined by gravimetric measurements of wetting tank.</p>		

## 7.0. Summary

A novel wet-vacuum-based chemical sampling methodology was evaluated for laboratory-scale and operational scale application to sample four types of building materials (stainless steel, vinyl flooring, laminate flooring, and plywood). A first step of method development involved the evaluation of a wetting agent suitable for wet-vacuum sampling of chemicals with varied solubility in water, followed by measurement of the amount of organic solvent or water-based wetting agent to be dispensed to a surface, and selection of the wetting agent surface residence time (or lapse time). After wetting solvent and lapse time evaluation, IPA- and water-based sampling methods were evaluated for sampling of target chemicals using two types of commercially available cleaners. The cleaner that offered better efficiency of solvent collection (Bissell Multi-Purpose Cleaner) and better overall chemical sampling efficiency was further optimized for improved recovery of IPA and chemical recovery. The optimized method was evaluated in operation-scale testing for multivariate (chemical and surface type, surface contamination level, and wetting agent type) sampling of medium-size (<1000 cm<sup>2</sup>) and large-size (approximately 5000 cm<sup>2</sup>) areas and compared to existing wipe-based sampling methods and/or modifications thereof.

The main findings of this study are:

- Wet-vacuum-based methods, utilizing a commercially available cleaner and IPA wetting solvent for sample collection, can be considered for sampling of various classes of chemicals with varied solubility in water, but the methodology is prone to the evaporation-related losses of IPA from the surface and – when larger-volume composite samples are collected – also in the liquid waste collection tank.
- Wet-vacuum method performance is lower for collection of target chemicals from semiporous materials (wood, vinyl), and for sampling of surfaces contaminated with lower ( $\mu\text{g}/\text{m}^2$ ) levels of chemicals.
- The efficiency of aqueous wetting agent-based wet-vacuum sampling is affected by the solubility of the chemical in water. The addition of surfactant improves recovery of selected water-insoluble chemicals but generally does not improve the sampling efficiency when lower surface concentrations of chemicals are targeted.
- The newly developed methodology used with IPA as wetting solvent offers eight- to tenfold improvement in turnaround time needed to collect and prepare surface sample for analysis. The aqueous wetting agent methods – that include the step of liquid-liquid extraction of sampling liquid– offers an approximately twofold reduction of the TOT, as compared to wipe-based methodology deployed in a discrete-area configuration.
- Both variations of wet-vacuum methods (single area and multiarea composite) used for collection of  $\text{mg}/\text{m}^2$  surface concentrations of chemicals, offer a similar (or better) sampling efficiency when compared to wipe-based methods.
- The vacuum cleaner components were confirmed to contain the chemical after use suggesting the wet-vacuum units should not be considered for reuse and should be handled as contaminated waste.

The novel sampling method developed in this study can be considered as a promising analytical tool for the medium- to large-area chemical contamination sampling. However, a careful selection of the wetting solvent that is appropriate for target chemical-surface characteristics combinations is needed for obtaining

reliable results. Further testing should be considered to find the maximum area the method can be deployed for while using composite-sampling techniques and water-based wetting solvent(s). Such optimization would improve the applicability of this method to field sampling of CWAs and other toxic chemicals of interest.

Future work to improve a wet-vacuum sampling method should consider use of more inert materials which may eliminate the use of a pre-sampling conditioning step and may allow for reuse of the same unit resulting in a lower sampling cost and less waste generated.

## 8.0. References

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## Appendix A: Supporting Information

### A-1 Wet-Vacuum Sampling Procedure

This procedure describes vacuum sampling using the Bissell SpotClean Portable Carpet Cleaner, model 5207A device. This procedure was optimized for sampling of areas from 929 cm<sup>2</sup> to 4,645 cm<sup>2</sup>.

#### Preparation of the vacuum

To assemble the vacuum cleaner, slide the flex hose clip into the opening on the front of the cleaner until you hear it click into place. Then snap the hose grip bracket into the back of machine on the clean tank side. Wrap the flex hose around the unit and snap the hose grip into the bracket. Attach the cord wrap by snapping it into place on the dirty water tank (referred to as dirty liquid tank below) side of the machine. Wrap the power cord around the cord wrap. The machine is now assembled.

#### Sampling

**Step 1** Connect the cleaning tool/nozzle and peripheral equipment per manufacturer instructions and secure. Tool easily snaps onto hose. For this study, the 3-in. Tough Stain Brush Tool was used.

**Step 2** Remove tank from tool side of unit. Pull on the black tab to remove rubber stopper and reveal tank opening.

**Step 3** Tank is marked with lines for a large stain or a small stain. For this project, fill the tank with IPA to the small stain fill line.

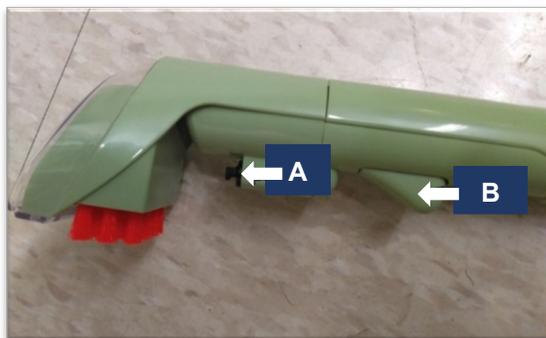
**Step 4** Replace rubber stopper, then replace the tank on the machine.

**Step 5** Twist the quick-release cord-wrap to release the cord. The vacuum is now ready to clean with the spot-clean option.

**Step 6** Prime the sprayer by compressing the trigger and discharging the IPA into a waste beaker for approximately 5 seconds. Weigh and record the wetting solvent tank mass. Weigh the liquid waste collection tank prior to the start of vacuum sampling.

**Step 7** If using IPA wetting agent for sampling, condition the vacuum by vacuuming 50 mL of IPA from a 1-L beaker. No conditioning is needed for any aqueous wetting agents (e.g., water, water-surfactant solutions). Immediately proceed to wetting the surface with the wetting agent.

**Step 8** To wet the sampling area of 929 cm<sup>2</sup>, spray the coupon surface for 8 seconds with the wet-vacuum sprayer. Hold the spray nozzle approximately 6 in. above the coupon surface. Press the spray trigger and spray the test surface of the coupon for eight seconds in an S-pattern covering the test area. Let IPA dwell on the coupon surface for 10 seconds. Figure A-1 illustrates the locations of the spray nozzle and spray trigger.



**Figure A-1 Spray nozzle (A) and spray trigger (B) locations**

**Step 9** Vacuum each TC starting in the top left corner the coupon. Position the nozzle at a 45-degree angle. Pull the nozzle toward the right side of the coupon. When the edge of the coupon is reached, start the next vacuuming pass on the left side overlapping approximately 50% of the previous pass. Continue this vacuuming pattern until the bottom of the coupon is reached. Next, vacuum the coupon starting in the top left corner. Position the nozzle at a 45-degree angle. Pull the nozzle toward the bottom edge of the coupon. When the bottom edge of the coupon is reached, start the next vacuuming pass on the top side overlapping approximately 50% of the previous pass. Continue this vacuuming pattern until the right edge of the coupon is reached. For composite-area sampling, repeat the procedure for all coupons.

**Step 10** After surface sampling is completed, rinse the wet-vacuum with 100 mL of IPA from a clean beaker.

**Step 11** Weigh the liquid waste collection tank and then transfer the entire contents of the dirty liquid tank to a clean Nalgene or equivalent archival bottle and store the sample at  $4 \pm 2$  °C until preparation for analysis.

## A-2 Parameters and Conditions for Instrumental analysis

**Table A-1. GC/FID Analyses of Nitrobenzene with Modified NIOSH\* 2005 (EMSL Analytical, Inc.)**

Parameter	Description/Conditions
Instrument	Agilent 7890A with dual FID (Agilent Technologies, Santa Clara, CA)
Autosampler	Agilent 7693 automatic sampler (Agilent Technologies, Santa Clara, CA)
Column	RTX 624sil Restek #13870 S/N 1208834 30 m, × 0.32 mm ID, 1.8 µm df (Restek Corporation, Bellefonte, PA)
GC column program	40 °C initial temperature, hold 0 min, 10 °C/min to 190 °C, hold 2 min
Carrier gas flow rate	3.3 mL/min
Injection volume/type	1.0 µL/splitless
Inlet temperature	225 °C
FID heater temperature	250 °C
Carrier	Helium
df: film thickness; *National Institute for Occupational Safety and Health	

**Table A-2. GC/FID Analyses of Phenol Modified NIOSH Method 2546 (EMSL Analytical, Inc.)**

Parameter	Description/Conditions
Instrument	Agilent 7980A gas chromatograph equipped with FID (Agilent Technologies, Santa Clara, CA)
Autosampler	7693 Automatic Sampler (Agilent Technologies, Santa Clara, CA)
Column	RTX™-VGC capillary column, 60 m × 0.530 µm ID, 0.3 µm df, part no. 19488 (Restek Corporation, Bellefonte, PA)
GC column program	70 °C initial temperature, hold 0 min, 15 °C/min to 250 °C, hold 2 min
Carrier gas flow rate	11.258 mL/min
Injection volume/type	1.0 µL/splitless
Inlet temperature	225 °C
FID heater temperature	250 °C
Hydrogen flow rate	40 mL/min
Air flow rate	400 mL/min
df: film thickness	

**Table A-3. GC/FID Analyses of TEP Using Modified NIOSH 5034 (EMSL Analytical, Inc.)**

Parameter	Description/Conditions
Instrument	Agilent 7980 Gas Chromatograph equipped with FID (Agilent Technologies, Santa Clara, CA)
Autosampler	7693 Automatic Sampler (Agilent Technologies, Santa Clara, CA)
Column	RTX™-VGC Capillary Columns, 60 m × 0.530 μm ID, 0.3 μm df; part no. 19488 (Restek Corporation, Bellefonte, PA)
GC column program	70 °C initial temperature, hold 0 min, 15 °C/min to 250 °C, hold 2 min
Carrier gas flow rate	11.258 mL/min
Injection volume/type	1.0 μL/splitless
Inlet temperature	225 °C
FID heater temperature	225 °C
Hydrogen flow rate	40 mL/min
Air flow rate	400 mL/min
df: film thickness	

**Table A-4. GC/FID Analyses of TEP Modified NIOSH 5038 (EMSL Analytical, Inc.)**

Parameter	Description/Conditions
Instrument	Agilent 7890A with dual FID (Agilent Technologies, Santa Clara, CA)
Autosampler	Agilent 7693 Automatic Sampler (Agilent Technologies, Santa Clara, CA)
Column	RTX 624 sil Restek #13870 S/N 1208834 30 m × 0.32 mm ID, 1.8 μm df (Restek Corporation, Bellefonte, PA)
GC column program	40 °C initial temperature, hold 0 min, 10 °C/min to 190 °C, hold 2 min
Carrier gas flow rate	3.3 mL/min
Injection volume/type	1.0 μL/splitless
Inlet temperature	225 °C
FID heater temperature	250 °C
Carrier	Helium
df: film thickness	

**Table A-5. GC/MS Analyses of TEP and 2-CEPS Samples in IPA (EPA OSL)**

Parameter	Description/Conditions
Instrument	Thermo Trace 1300 GC ISQ™ Mass Spectrometer (Thermo Fisher Scientific, Inc., Waltham, MA)
Autosampler	AS/AI 1310 Autosampler (Thermo Fisher Scientific, Inc., Waltham, MA)
Column	DB-5, 20 m × 0.25 mm ID, 0.25 µm df (Agilent, Santa Clara, CA)
GC column program	60 °C initial temperature, hold 0 min, 8 °C/min to 260 °C, hold 8 min
Carrier gas flow rate	1.3 mL/min
Injection volume/type	1.0 µL/splitless
Inlet temperature	150 °C
MS source temperature	155 °C
MS transfer line	150 °C
df: film thickness	

**Table A-6. GC/MS Analyses of TEP and 2-CEPS in Hexane (EPA OSL)**

Parameter	Description/Conditions
Instrument	Thermo Trace 1300 Gas Chromatograph GC ISQ™ Mass Spectrometer (Thermo Fisher Scientific, Inc., Waltham, MA)
Autosampler	AS/AI 1310 Autosampler (Thermo Fisher Scientific, Inc., Waltham, MA)
Column	DB-5, 20 m × 0.25 mm ID, 0.25 µm df (Agilent, Santa Clara, CA)
GC column program	60 °C initial temperature, hold 0 min, 8 °C/min to 260 °C, hold 8 min
Carrier gas flow rate	1.3 mL/min
Injection volume/type	1.0 µL/splitless
Inlet temperature	250 °C
MS source temperature	250 °C
MS transfer line	250 °C
df: film thickness	

## A-3 Sample-Specific Test Results

Table A-7. Liquid-Liquid Extraction Efficiency of Selected Chemicals from Wetting Agents

Chemical:	2-CEPS				TEP				Nitrobenzene	
HT tested:	1 h		24 h		1 h		24 h		1 h	
Amount spiked:	5.6 mg								2.4 mg	
Sample recovery:	Mass (mg)	Recovery (%)	Mass (mg)	Recovery (%)	Mass (mg)	Recovery (%)	Mass (mg)	Recovery (%)	Mass (mg)	Recovery (%)
Extraction from SSDX-12®-water solution*										
PB-1	ND	NA	ND	NA	ND	NA	ND	NA	Not tested	
TL-1	4.82	86%	1.76	31%	4.98	97%	4.92	96%		
TL-2	5.10	90%	1.66	29%	5.28	103%	5.04	98%		
TL-3	5.10	90%	1.67	30%	5.25	102%	5.11	100%		
Average	4.99	89%	1.70	30%	5.17	101%	5.02	98%		
Std Dev	0.12	2.2%	0.045	0.79%	0.13	2.6%	0.081	1.6%		
% RSD	2.4%		2.6%		2.6%		1.6%			
Extraction from Dawn Ultra®-water solution*										
PB-1	ND	NA	ND	NA	ND	NA	ND	NA	Not tested	
TL-1	5.04	90%	3.06	54%	4.81	94%	5.21	101%		
TL-2	5.35	95%	3.01	53%	4.77	93%	5.18	101%		
TL-3	5.40	96%	3.15	56%	5.05	98%	5.16	100%		
Average	5.27	93%	3.07	55%	4.87	95%	5.18	101%		
Std Dev	0.16	2.8%	0.060	1.1%	0.12	2.4%	0.020	0.4%		
% RSD	3.0%		1.9%		2.53%		0.38%			
Extraction from water*										
PB-1	ND	NA	ND	NA	ND	NA	ND	NA	ND	NA
TL-1	4.86	86%	2.48	44%	4.80	93%	4.98	97%	2.10	85%
TL-2	4.92	87%	2.78	49%	5.16	100%	5.03	98%	2.70	109%
TL-3	4.94	88%	2.80	50%	4.99	97%	5.05	98%	2.20	89%
Average	4.91	87%	2.69	48%	4.98	97%	5.02	98%	2.33	94%
Std Dev	0.034	0.61%	0.15	2.6%	0.15	2.8%	0.029	0.56%	0.32	13%
% RSD	0.70%		5.4%		2.9%		0.58%		14%	
Extraction from Tween® 20-water solution*										
PB-1	Not tested				Not tested				NA	NA
TL-1									4.3	75%
TL-2									4.4	76%
TL-3									1.2 (E)	21% (E)
Average									4.4	76%
Std Dev									0.05	1%
% RSD									1%	
*1:1 (v:v) extraction with DCM or hexane, chemical target-wetting agent combination-specific additives are listed in Table 3-6; E- excluded from arithmetic mean (average) calculations due to suspected reporting error identified during data validation; TL- test liquid sample (wetting liquid spiked with target chemical); PB – procedural blank sample (not spiked wetting liquid that underwent the entire analytical procedure)										

**Table A-8. Results of Direct Analysis of Phenol in Various Wetting Agents**

Chemical:	Phenol	
HT tested:	1 h	
Sample recovery:	Mass (mg)	Recovery (%)
Wetting Agent		
Water	2.0	94%
2% Tween-water	2.1	99%
IPA	2.1	99%
Average	2.1	97%
Std Dev	0.06	2.9%
% RSD	2.8%	

**Table A-9. Results of Direct Analysis of TEP in IPA**

Chemical:	TEP	
HT tested:	1 h	
Sample recovery:	Recovery (%)	Standard Deviation (%)
Phase		
Phase I, phase II	105.3	4.7
Phase III	96.1	7.14
Average recovery (all phases)	100.7%	
Std Dev	4.6%	
% RSD	4.6%	

**Table A-10. Results of Direct Analysis of 2-CEPS in IPA**

Chemical:	2-CEPS	
HT tested:	1 h	
Sample recovery:	Recovery (%)	Standard Deviation (%)
Phase		
Phase II	92.9	7.2
Average	92.9%	
Std Dev	7.2%	
% RSD	7.7%	

**Table A-11. Phase I Operational Parameter Optimization – Selection of Wetting Agent for Sampling of Phenol from Nonporous Reference Material**

Phenol					
Material Type and Wetting Agent Type	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
<b>Stainless Steel – Water</b>					
TC-1	26	37.8	87%	15.5	60%
TC-2	26	43.0	86%	15.9	62%
TC-3	26	40.4	87%	15.7	61%
TC-4	26	41.7	87%	16.2	63%
TC-5	26	42.3	88%	16.9	66%
PB	Not spiked	44.9	89%	ND	NA
Average		41.7	87%	16.0	62%
Std Dev		2.42	1.0%	0.55	2.3%
% RSD		5.8%	1.2%	3.4%	3.7%
<b>Stainless Steel – Tween®-water</b>					
TC-1	26	41.0	87%	17.6	69%
TC-2	26	41.1	89%	17.7	69%
TC-3	26	42.4	89%	16.5	64%
TC-4	26	42.0	88%	16.4	64%
TC-5	26	41.5	86%	17.8	69%
PB	Not-spiked	42.4	89%	ND	NA
Average		41.7	88%	17.2	67%
Std Dev		0.63	1.3%	0.69	2.7%
% RSD		1.5%	1.4%	4.0%	4.1%
<b>Stainless Steel – IPA</b>					
TC-1	26	26.1	43%	16.5	64%
TC-2	26	27.8	56%	15.6	61%
TC-3	26	25.0	46%	17.0	66%
TC-4	26	30.4	63%	17.9	70%
TC-5	26	23.2	42%	15.8	61%
PB	not-spiked	35.3	58%	ND	NA
Average		28.0	51%	16.6	64%
Std Dev		4.35	8.8%	0.93	3.8%
% RSD		15.6%	17.1%	5.6%	5.9%
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB – SL collected using wet-vacuum apparatus from noncontaminated (procedural blank) stainless-steel coupon; ND – Not detected; NA – Not applicable.					

**Table A-12. Phase I Operational Parameters Optimization – Selection of Wetting Agent for Sampling of Nitrobenzene from Nonporous Reference Material**

Nitrobenzene					
Material Type and Wetting Agent Type	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
<b>Stainless Steel – Water</b>					
TC-1	29	44.4	92%	4.0	14%
TC-2	29	46.8	91%	5.6	20%
TC-3	29	41.8	92%	4.1	14%
TC-4	29	45.4	93%	3.9	13%
TC-5	29	48.3	91%	4.8	17%
PB	Not spiked	44.4	91%	ND	NA
Average		45.2	92%	4.5	16%
Std Dev		2.24	0.8%	0.72	2.9%
% RSD		5.0%	0.9%	16.0%	18.5%
<b>Stainless Steel – Tween®-water</b>					
TC-1	29	35.0	88%	4.2	15%
TC-2	29	25.3	85%	2.8	10%
TC-3	29	33.8	89%	3.1	11%
TC-4	29	44.6	87%	4.9	17%
TC-5	29	37.3	88%	22.0 (S,E)	77% (S,E)
PB	Not spiked	32.2	84%	ND	NA
Average		34.7	87%	3.8	13%
Std Dev		6.33	1.9%	0.97	3.3%
% RSD		18.2%	2.2%	26.0%	24.9%
<b>Stainless Steel – IPA</b>					
TC-1	29	28.2	51%	17.5	61%
TC-2	29	13.6 (L)	28% (L)	9.2 (E)	32% (E)
TC-3	29	28.9	57%	18.8	65%
TC-4	29	21.6	46%	17.3	60%
TC-5	29	33.0	62%	20.5	71%
PB	Not spiked	26.2	50%	ND	NA
Average		27.6	53%	18.5	64%
Std Dev		4.16	6.3%	1.48	5.0%
% RSD		15.1%	11.8%	8.0%	7.8%
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB – SL collected using wet-vacuum apparatus from noncontaminated (procedural blank) stainless-steel coupon; S – Possible sample preparation error identified; L – low solvent collection efficiency observed; E – value excluded from statistical calculations; ND – Not detected; NA – Not applicable.					

**Table A-13. Phase I Operational Parameters Optimization – Selection of Wetting Agent for Sampling of TEP from Nonporous Reference Material**

TEP					
Material Type and Wetting Agent Type	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
<b>Stainless Steel – Water</b>					
TC-1	26	45.4	89%	11.1	43%
TC-2	26	46.7	89%	11.1	43%
TC-3	26	48.6	92%	13.0	51%
TC-4	26	47.9	90%	13.1	51%
TC-5	26	52.9	93%	8.2	32%
PB	Not spiked	46.2	91%	ND	NA
Average		48.0	91%	11.3	44%
Std Dev		2.69	1.6%	1.99	7.8%
% RSD		5.6%	1.8%	17.6%	17.8%
<b>Stainless Steel – Tween®-water</b>					
TC-1	29	41.0	88%	12.4	48%
TC-2	29	44.3	90%	13.3	52%
TC-3	29	44.1	90%	13.5	53%
TC-4	29	43.7	88%	11.2	44%
TC-5	29	46.8	88%	14.5	56%
PB	Not spiked	45.3	88%	ND	NA
Average		44.2	89%	13.0	51%
Std Dev		1.92	1.0%	1.24	4.7%
% RSD		4.3%	1.2%	9.6%	9.2%
<b>Stainless Steel – IPA</b>					
TC-1	26	30.4	53%	17.6	69%
TC-2	26	25.5	49%	16.1	63%
TC-3	26	27.8	51%	22.3	87%
TC-4	26	22.0	42%	11.0	43%
TC-5	26	38.3	76%	18.4	72%
PB	Not spiked	31.2	55%	ND	NA
Average		29.2	54%	17.1	67%
Std Dev		5.58	11.5%	4.10	16.0%
% RSD		19.1%	21.2%	24.0%	23.9%
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB – SL collected using wet-vacuum apparatus from non-contaminated (procedural blank) stainless-steel coupon; ND – Not detected; NA – Not applicable.					

**Table A-14. Phase I Operational Parameters Optimization – Selection of the Wetting Agent Surface Contact Time (Lapse Time) for IPA Sampling of Phenol from Nonporous Reference Material**

Phenol					
Material Type– Wetting Agent – Surface Lapse Time	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
<b>Stainless Steel – IPA – LT = 1 s</b>					
TC-1	26	31.2	59%	17.8	69%
TC-2	26	31.4	60%	16.6	65%
TC-3	26	42.8	72%	18.0	70%
TC-4	26	36.8	70%	17.7	69%
TC-5	26	43.0	76%	16.8	65%
PB	Not spiked	33.4	65%	ND	NA
Average		36.4	67%	17.4	68%
Std Dev		5.40	6.8%	0.63	2.4%
% RSD		14.8%	10.2%	3.6%	3.6%
<b>Stainless Steel – IPA – LT = 10 s</b>					
TC-1	26	34.8	68%	18.5	72%
TC-2	26	34.6	65%	19.7	77%
TC-3	26	32.9	59%	13.2	51%
TC-4	26	31.7	64%	17.1	67%
TC-5	26	42.9	73%	17.6	69%
PB	Not spiked	34.4	65%	ND	NA
Average		35.2	66%	17.2	67%
Std Dev		3.95	4.6%	2.45	9.8%
% RSD		11.2%	7.1%	14.3%	14.6%
<b>Stainless Steel – IPA – LT = 100 s</b>					
TC-1	26	26.1	43%	16.5	64%
TC-2	26	27.8	56%	15.6	61%
TC-3	26	25.0	46%	17.0	66%
TC-4	26	30.4	63%	17.9	70%
TC-5	26	23.2	42%	15.8	61%
PB	Not spiked	35.3	65%	ND	NA
Average		28.0	53%	16.6	64%
Std Dev		4.35	10.2%	0.93	3.8%
% RSD		15.6%	19.5%	5.6%	5.9%
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB – SL collected using wet-vacuum apparatus from noncontaminated (procedural blank) stainless-steel coupon; ND – Not detected; NA – Not applicable; LT – Surface lapse time (wetting agent surface contact time).					

**Table A-15. Phase I Operational Parameters Optimization – Selection of the Wetting Agent Surface Contact Time (Lapse Time) for IPA Sampling of Nitrobenzene from Nonporous Reference Material**

Nitrobenzene					
Material Type– Wetting Agent – Surface Lapse Time	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
<b>Stainless Steel – IPA – LT = 1 s</b>					
TC-1	29	17.1	34%	15.2	53%
TC-2	29	39.7	75%	27.8	97%
TC-3	29	30.1	63%	24.4	85%
TC-4	29	34.4	65%	26.2	91%
TC-5	29	36.0	70%	23.0	80%
PB	Not spiked	36.4	62%	ND	NA
Average		32.3	62%	23.3	81%
Std Dev		8.07	14.3%	4.89	17.0%
% RSD		25.0%	23.3%	21.0%	20.9%
<b>Stainless Steel – IPA – LT = 10 s</b>					
TC-1	29	26.6	61%	29.2	102%
TC-2	29	34.1	66%	29.0	101%
TC-3	29	30.9	65%	25.9	90%
TC-4	29	37.8	74%	26.1	91%
TC-5	29	32.4	70%	27.5	96%
PB	Not spiked	41.5	77%	ND	NA
Average		33.9	69%	27.5	96%
Std Dev		5.24	6.0%	1.55	5.5%
% RSD		15.5%	8.7%	5.6%	5.8%
<b>Stainless Steel – IPA – LT = 100 s</b>					
TC-1	29	28.2	51%	17.5	61%
TC-2	29	13.6	28%	9.2	32%
TC-3	29	28.9	57%	18.8	65%
TC-4	29	21.6	46%	17.3	60%
TC-5	29	33.0	62%	20.5	71%
PB	Not spiked	26.2	50%	ND	NA
Average		25.3	49%	16.7	58%
Std Dev		6.81	11.7%	4.36	15.1%
% RSD		27.0%	23.9%	26.2%	26.0%
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB – SL collected using wet-vacuum apparatus from non-contaminated (procedural blank) stainless-steel coupon; ND – Not detected; NA – Not applicable; LT – Surface lapse time (wetting agent surface contact time).					

**Table A-16. Phase I Operational Parameters Optimization – Selection of the Wetting Agent Surface Contact Time (Lapse Time) for IPA Sampling of TEP from Nonporous Reference Material**

TEP					
Material Type– Wetting Agent – Surface Lapse Time	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
<b>Stainless Steel – IPA – LT = 1 s</b>					
TC-1	26	36.7	72%	20.9	81%
TC-2	26	35.4	68%	22.0	86%
TC-3	26	39.0	71%	23.4	91%
TC-4	26	40.2	70%	22.9	89%
TC-5	26	35.5	65%	21.3	83%
PB	Not spiked	37.9	64%	ND	NA
Average		37.5	68%	22.1	86%
Std Dev		1.94	3.3%	1.05	4.1%
% RSD		5.2%	4.8%	4.8%	4.8%
<b>Stainless Steel – IPA – LT = 10 s</b>					
TC-1	26	37.0	69%	20.0	78%
TC-2	26	42.4	73%	21.6	84%
TC-3	26	35.5	68%	23.8	93%
TC-4	26	41.3	73%	24.8	96%
TC-5	26	38.7	70%	21.7	84%
PB	Not spiked	38.7	69%	ND	NA
Average		38.9	70%	22.4	87%
Std Dev		2.58	2.2%	1.91	7.3%
% RSD		6.6%	3.1%	8.5%	8.4%
<b>Stainless Steel – IPA – LT = 100 s</b>					
TC-1	26	30.4	53%	17.6	69%
TC-2	26	25.5	49%	16.1	63%
TC-3	26	27.8	51%	22.3	87%
TC-4	26	22.0	42%	11.0	43%
TC-5	26	38.3	76%	18.4	72%
PB	Not spiked	31.2	55%	ND	NA
Average		29.2	54%	17.1	67%
Std Dev		5.58	11.5%	4.10	16.0%
% RSD		19.1%	21.2%	24.0%	23.9%
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB – SL collected using wet-vacuum apparatus from non-contaminated (procedural blank) stainless-steel coupon; ND – Not detected; NA – Not applicable; LT – Surface lapse time (wetting agent surface contact time).					

**Table A-17. Phase I Operational Parameters Optimization – Evaluation of IPA Wetting Agent for Sampling of Phenol from Semiporous and Porous Materials**

Phenol					
Material Type– Wetting Agent – Surface Lapse Time	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
Vinyl flooring – IPA – LT = 10 s					
TC-1	26	19.2	42%	9.4	37%
TC-2	26	20.3	40%	7.7	30%
TC-3	26	23.9	48%	9.1	35%
TC-4	26	20.0	40%	8.2	32%
TC-5	26	17.8	38%	6.8	26%
PB	Not spiked	22.3	44%	ND	NA
Average		20.6	42%	8.2	32%
Std Dev		2.19	3.6%	1.05	4.3%
% RSD		10.7%	8.5%	12.8%	13.4%
Plywood – IPA – LT = 10 s					
TC-1	26	14.6	25%	0.8	3%
TC-2	26	10.6	20%	0.6	2%
TC-3	26	11.2	19%	0.4	2%
TC-4	26	11.2	19%	0.4	1%
TC-5	26	11.3	21%	0.6	2%
PB	Not spiked	11.6	23%	ND	NA
Average		11.8	21%	0.6	2%
Std Dev		1.43	2.4%	0.17	0.7%
% RSD		12.2%	11.3%	29.9%	35.4%
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB – SL collected using wet-vacuum apparatus from non-contaminated (procedural blank) stainless-steel coupon; ND – Not detected; NA – Not applicable; LT – Surface lapse time (wetting agent surface contact time).					

**Table A-18. Phase I Operational Parameters Optimization – Evaluation of IPA Wetting Agent for Sampling of Nitrobenzene from Semiporous and Porous Materials**

Nitrobenzene					
Material Type– Wetting Agent – Surface Lapse Time	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
<b>Vinyl flooring – IPA– LT = 10 s</b>					
TC-1	29	22.0	48%	3.7	13%
TC-2	29	19.5	53%	2.7	10%
TC-3	29	29.2	39%	4.1	14%
TC-4	29	20.0	59%	3.6	13%
TC-5	29	26.7	40%	4.0	14%
PB	Not spiked	22.8	48%	ND	NA
Average		23.4	48%	3.6	13%
Std Dev		3.84	7.6%	0.55	1.6%
% RSD		16.4%	15.9%	15.3%	12.8%
<b>Plywood – IPA – LT = 10 s</b>					
TC-1	29	10.7	20%	1.2	4%
TC-2	29	8.7	17%	0.9	3%
TC-3	29	9.6	19%	1.1	4%
TC-4	29	19.7	33%	1.1	4%
TC-5	29	14.1	26%	1.2	4%
PB	Not spiked	12.6	22%	ND	NA
Average		12.6	23%	1.1	4%
Std Dev		4.01	5.8%	0.12	0.4%
% RSD		31.9%	25.6%	11.1%	11.8%
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB – SL collected using wet-vacuum apparatus from non-contaminated (procedural blank) stainless-steel coupon; ND – Not detected; NA – Not applicable; LT – Surface lapse time (wetting agent surface contact time).					

**Table A-19. Phase I Operational Parameters Optimization – Evaluation of IPA Wetting Agent for Sampling of TEP from Semiporous and Porous Materials**

TEP					
Material Type– Wetting Agent – Surface Lapse Time	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
<b>Vinyl flooring – IPA – LT = 10 s</b>					
TC-1	26	24.7	48%	6.4	25%
TC-2	26	31.0	61%	9.3	36%
TC-3	26	20.7	42%	5.8	23%
TC-4	26	27.6	53%	7.2	28%
TC-5	26	26.0	50%	7.5	29%
PB	Not spiked	23.6	46%	ND	NA
Average		25.6	50%	7.2	28%
Std Dev		3.53	6.5%	1.33	5.0%
% RSD		13.8%	13.1%	18.4%	17.6%
<b>Plywood – IPA – LT = 10 s</b>					
TC-1	26	11.7	22%	1.3	5%
TC-2	26	18.6	31%	1.0	4%
TC-3	26	17.3	31%	0.9	4%
TC-4	26	16.1	28%	1.5	6%
TC-5	26	10.4	20%	0.8	3%
PB	Not spiked	15.9	33%	ND	NA
Average		15.0	28%	1.1	4%
Std Dev		3.23	5.3%	0.29	1.1%
% RSD		21.6%	19.3%	26.5%	25.9%
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB – SL collected using wet-vacuum apparatus from non-contaminated (procedural blank) stainless-steel coupon; ND- Not detected; NA – Not applicable; LT – Surface lapse time (wetting agent surface contact time).					

**Table A-20. Phase II Evaluation of Commercial Wet-Vacuums for Sampling of Phenol from Stainless Steel**

Phenol					
Material and Vacuum Type – Wetting Agent – Surface Lapse Time	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
<b>Stainless Steel – Bissell Little Green ProHeat – IPA – LT = 10 s</b>					
TC-1	26	26.6	59%	9.8	38%
TC-2	26	40.1	69%	12.8	50%
TC-3	26	34.6	67%	10.7	42%
TC-4	26	29.4	62%	10.3	40%
TC-5	26	27.5	58%	9.9	39%
PB	Not spiked	36.1	66%	ND	NA
Average		32.4	64%	10.7	42%
Std Dev		5.38	4.5%	1.23	4.8%
% RSD		16.6%	7.1%	11.5%	11.5%
<b>Stainless Steel -Rug Doctor Portable Spot Cleaner - IPA– LT = 10 s</b>					
TC-1	26	17.1	32%	4.8	19%
TC-2	26	15.2	70%	4.9	19%
TC-3	26	19.8	39%	5.1	20%
TC-4	26	24.1	42%	5.8	23%
TC-5	26	22.5	43%	5.4	21%
PB	Not spiked	23.5	41%	ND	NA
Average		20.4	45%	5.2	20%
Std Dev		3.63	13.1%	0.41	1.7%
% RSD		17.8%	29.4%	7.8%	8.2%
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB – SL collected using wet-vacuum apparatus from non-contaminated (procedural blank) stainless-steel coupon; ND – Not detected; NA – Not applicable; LT – Surface lapse time (wetting agent surface contact time).					

**Table A-21. Phase II Evaluation of Commercial Wet-Vacuums for Sampling of Phenol from Laminate Flooring**

Phenol					
Material and Vacuum Type – Wetting Agent – Surface Lapse Time	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
<b>Laminate flooring –Bissell Little Green ProHeat - IPA – LT = 10 s</b>					
TC-1	26	31.2	64%	10.9	43%
TC-2	26	34.7	64%	11.5	45%
TC-3	26	20.2	51%	9.1	35%
TC-4	26	21.0	53%	9.2	36%
TC-5	26	23.1	57%	10.2	40%
PB	Not spiked	29.7	61%	ND	NA
Average		26.7	58%	10.2	40%
Std Dev		6.02	5.6%	1.05	4.3%
% RSD		22.6%	9.6%	10.3%	10.9%
<b>Laminate flooring – Rug Doctor Portable Spot Cleaner – IPA – LT = 10 s</b>					
TC-1	26	19.8	33%	5.9	23%
TC-2	26	18.3	35%	4.0	16%
TC-3	26	16.7	32%	3.7	14%
TC-4	26	19.6	36%	5.7	22%
TC-5	26	16.9	28%	5.2	20%
PB	Not spiked	12.8	26%	ND	NA
Average		17.4	32%	4.9	19%
Std Dev		2.58	3.9%	1.00	3.9%
% RSD		14.9%	12.4%	20.4%	20.4%
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB – SL collected using wet-vacuum apparatus from non-contaminated (procedural blank) stainless-steel coupon; ND – Not detected; NA – Not applicable; LT – Surface lapse time (wetting agent surface contact time).					

**Table A-22. Phase II Evaluation of Commercial Wet-Vacuums for Sampling of Phenol from Vinyl Flooring**

Phenol					
Material and Vacuum Type – Wetting Agent – Surface Lapse Time	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
<b>Vinyl flooring – Bissell Little Green ProHeat – IPA – LT = 10 s</b>					
TC-1	26	19.0	51%	8.2	32%
TC-2	26	15.6	48%	6.9	27%
TC-3	26	27.1	60%	8.7	34%
TC-4	26	22.9	39%	5.7	22%
TC-5	26	25.8	51%	7.2	28%
PB	Not spiked	22.4	43%	ND	NA
Average		22.1	49%	7.3	29%
Std Dev		4.27	7.3%	1.17	4.7%
% RSD		19.3%	15.0%	16.0%	16.3%
<b>Vinyl flooring – Rug Doctor Portable Spot Cleaner – IPA – LT = 10 s</b>					
TC-1	26	17.1	32%	3.1	12%
TC-2	26	19.4	38%	4.7	18%
TC-3	26	21.3	38%	4.7	18%
TC-4	26	12.0	25%	3.0	12%
TC-5	26	20.7	40%	4.6	18%
PB	Not spiked	18.6	38%	ND	NA
Average		18.2	35%	4.0	16%
Std Dev		3.38	5.7%	0.89	3.3%
% RSD		18.6%	16.1%	22.1%	21.1%
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB – SL collected using wet-vacuum apparatus from non-contaminated (procedural blank) stainless-steel coupon; ND – Not detected; NA – Not applicable; LT – Surface lapse time (wetting agent surface contact time).					

**Table A-23. Phase II Evaluation of Commercial Wet-Vacuums for Sampling of Nitrobenzene from Stainless Steel**

Nitrobenzene					
Material and Vacuum Type – Wetting Agent – Surface Lapse Time	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
<b>Stainless Steel – Bissell Little Green ProHeat – IPA – LT = 10 s</b>					
TC-1	29	31.4	59%	1.9	6%
TC-2	29	30.5	58%	2.0	7%
TC-3	29	34.7	68%	2.3	8%
TC-4	29	28.2	59%	1.5	5%
TC-5	29	38.5	66%	2.3	8%
PB	Not spiked	24.0	54%	ND	NA
Average		31.2	61%	2.00	7%
Std Dev		5.04	5.3%	0.33	1.3%
% RSD		16.1%	8.7%	16.6%	19.2%
<b>Stainless Steel – Rug Doctor Portable Spot Cleaner – IPA – LT = 10 s</b>					
TC-1	29	10.0	25%	0.0	0%
TC-2	29	21.0	39%	0.5	2%
TC-3	29	19.0	36%	0.4	1%
TC-4	29	25.3	44%	0.7	2%
TC-5	29	25.8	46%	0.8	3%
PB	Not spiked	23.9	40%	ND	NA
Average		20.8	38%	0.48	2%
Std Dev		5.91	7.4%	0.31	1.1%
% RSD		28.4%	19.4%	64.9%	71.3%
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB –SL collected using wet-vacuum apparatus from non-contaminated (procedural blank) stainless-steel coupon; ND – Not detected; NA – Not applicable; LT – Surface lapse time (wetting agent surface contact time).					

**Table A-24. Phase II Evaluation of Commercial Wet-Vacuums for Sampling of Nitrobenzene from Laminate Flooring**

Nitrobenzene					
Material and Vacuum Type – Wetting Agent – Surface Lapse Time	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
<b>Laminate flooring – Bissell Little Green ProHeat – IPA – LT = 10 s</b>					
TC-1	29	34.5	72%	1.4	5%
TC-2	29	22.4	82%	1.5	5%
TC-3	29	41.9	80%	2.7	9%
TC-4	29	49.0	82%	3.2	11%
TC-5	29	37.9	79%	2.3	8%
PB	Not spiked	33.5	80%	ND	NA
Average		36.5	79%	2.22	8%
Std Dev		8.94	3.7%	0.77	2.6%
% RSD		24.5%	4.7%	34.8%	34.3%
<b>Laminate flooring – Rug Doctor Portable Spot Cleaner – IPA – LT = 10 s</b>					
TC-1	29	21.1	42%	0.5	2%
TC-2	29	22.6	41%	0.6	2%
TC-3	29	22.1	14%	0.6	2%
TC-4	29	21.5	40%	0.5	2%
TC-5	29	17.9	34%	0.6	2%
PB	Not spiked	13.3	30%	ND	NA
Average		19.8	34%	0.56	2%
Std Dev		3.57	10.6%	0.05	0.0%
% RSD		18.1%	31.7%	9.8%	0.0%
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB – SL collected using wet-vacuum apparatus from non-contaminated (procedural blank) stainless-steel coupon; ND – Not detected; NA – Not applicable; LT – Surface lapse time (wetting agent surface contact time).					

**Table A-25. Phase II Evaluation of Commercial Wet-Vacuums for Sampling of Nitrobenzene from Vinyl Flooring**

Nitrobenzene					
Material and Vacuum Type – Wetting Agent – Surface Lapse Time	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
<b>Vinyl flooring – Bissell Little Green ProHeat –IPA – LT = 10 s</b>					
TC-1	29	24.4	59%	ND	NA
TC-2	29	35.6	64%	ND	NA
TC-3	29	41.7	69%	ND	NA
TC-4	29	28.7	64%	ND	NA
TC-5	29	31.7	65%	ND	NA
PB	Not spiked	31.9	62%	ND	NA
Average		32.3	64%	ND	NA
Std Dev		5.92	3.3%	ND	NA
% RSD		18.3%	5.2%	ND	NA
<b>Vinyl flooring – Rug Doctor Portable Spot Cleaner – IPA – LT = 10 s</b>					
TC-1	29	25.7	52%	ND	NA
TC-2	29	32.1	65%	ND	NA
TC-3	29	25.9	94%	ND	NA
TC-4	29	34.1	67%	ND	NA
TC-5	29	36.1	74%	ND	NA
PB	Not spiked	42.2	74%	ND	NA
Average		32.7	71%	ND	NA
Std Dev		6.31	13.9%	ND	NA
% RSD		19.3%	19.5%	ND	NA
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB – SL collected using wet-vacuum apparatus from non-contaminated (procedural blank) stainless-steel coupon; ND – Not detected; NA – Not applicable; LT – Surface lapse time (wetting agent surface contact time).					

**Table A.26 Phase II Evaluation of Commercial Wet-Vacuums for Sampling of TEP from Stainless Steel Coupon**

TEP					
Material and Vacuum Type – Wetting Agent – Surface Lapse Time	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
<b>Stainless-steel coupon – Bissell Little Green ProHeat – IPA – LT = 10 s</b>					
TC-1	26	26.0	69%	10.7	42%
TC-2	26	38.7	70%	5.8	23%
TC-3	26	37.1	72%	15.2	59%
TC-4	26	34.4	66%	8.9	35%
TC-5	26	40.6	80%	11.8	46%
PB	Not spiked	40.0	78%	ND	NA
Average		36.1	73%	10.48	41%
Std Dev		5.44	5.4%	3.48	13.3%
% RSD		15.1%	7.5%	33.2%	32.5%
<b>Stainless-steel coupon – Rug Doctor Portable Spot Cleaner – IPA – LT = 10 s</b>					
TC-1	26	25.2	52%	3.5	14%
TC-2	26	36.7	62%	5.1	20%
TC-3	26	29.7	64%	5.0	20%
TC-4	26	58.0	69%	4.6	18%
TC-5	26	34.7	63%	4.5	18%
PB	Not spiked	39.1	68%	ND	NA
Average		37.2	63%	4.54	18%
Std Dev		11.34	6.1%	0.63	2.4%
% RSD		30.5%	9.6%	14.0%	13.6%
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB – SL collected using wet-vacuum apparatus from non-contaminated (procedural blank) stainless-steel coupon; ND – Not detected; NA – Not applicable; LT – Surface lapse time (wetting agent surface contact time).					

**Table A-27. Phase II Evaluation of Commercial Wet-Vacuums for Sampling of TEP from Laminate Flooring**

TEP					
Material and Vacuum Type – Wetting Agent – Surface Lapse Time	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
<b>Laminate flooring – Bissell Little Green ProHeat – IPA – LT = 10 s</b>					
TC-1	26	33.0	60%	8.3	32%
TC-2	26	37.8	72%	8.7	34%
TC-3	26	27.2	68%	9.5	37%
TC-4	26	30.9	64%	5.6	22%
TC-5	26	26.9	66%	7.0	27%
PB	Not spiked	34.3	69%	ND	NA
Average		31.7	67%	7.82	30%
Std Dev		4.23	4.2%	1.54	5.9%
% RSD		13.4%	6.3%	19.6%	19.5%
<b>Laminate flooring-Rug Doctor Portable Spot Cleaner - IPA– LT = 10 s</b>					
TC-1	26	23.3	42%	7.0	27%
TC-2	26	23.9	41%	4.1	16%
TC-3	26	18.9	28%	7.7	30%
TC-4	26	20.3	35%	4.3	17%
TC-5	26	20.8	37%	6.0	23%
PB	Not spiked	8.3	18%	ND	NA
Average		19.3	34%	5.82	23%
Std Dev		5.68	9.1%	1.60	6.1%
% RSD		29.5%	27.1%	27.5%	27.0%
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB – SL collected using wet-vacuum apparatus from non-contaminated (procedural blank) stainless-steel coupon; ND – Not detected; NA – Not applicable; LT – Surface lapse time (wetting agent surface contact time).					

**Table A-28. Phase II Evaluation of Commercial Wet-Vacuums for Sampling of TEP from Vinyl Flooring**

TEP					
Material and Vacuum Type – Wetting Agent – Surface Lapse Time	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
<b>Vinyl flooring – Bissell Little Green ProHeat – IPA – LT = 10 s</b>					
TC-1	26	20.1	60%	5.0	20%
TC-2	26	30.4	59%	5.8	22%
TC-3	26	31.8	63%	2.3	9%
TC-4	26	36.3	66%	6.5	25%
TC-5	26	44.4	73%	6.2	24%
PB	Not spiked	31.7	73%	ND	NA
Average		32.5	66%	5.16	20%
Std Dev		7.94	6.2%	1.69	6.4%
% RSD		24.5%	9.4%	32.8%	32.2%
<b>Vinyl flooring – Rug Doctor Portable Spot Cleaner – IPA – LT = 10 s</b>					
TC-1	26	30.2	62%	2.0	8%
TC-2	26	28.8	52%	2.2	9%
TC-3	26	23.8	47%	1.7	7%
TC-4	26	31.5	61%	2.7	11%
TC-5	26	36.7	70%	3.7	14%
PB	Not spiked	10.1	22%	ND	NA
Average		26.9	52%	2.46	10%
Std Dev		9.20	16.9%	0.78	2.8%
% RSD		34.3%	32.3%	31.8%	28.3%
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB – SL collected using wet-vacuum apparatus from non-contaminated (procedural blank) stainless-steel coupon; ND – Not detected; NA – Not applicable; LT – Surface lapse time (wetting agent surface contact time)					

**Table A-29. Phase II Optimization of Wetting Solvent and Chemical Recovery Using Bissell Little Green ProHeat Wet-Vacuum – Phenol on Stainless-steel Coupon**

Phenol						
Surface loading: 85 mg						
Wetting agent: IPA						
Procedure: Vacuum conditioning – surface wetting- vacuum rinse (volumes, mL)	Chemical Recovered		Wetting Solvent Recovered (%)		Recovery of Rinse (%)	
	Mass (mg)	Recovery (%)	Volume (mL)	Recovery (%)	Volume (mL)	Recovery (%)
50 mL + 50 mL + no rinse	20.8	24%	27	57%	NA	no rinse
	19.7	23%	26	50%	NA	no rinse
	35.1	41%	33	68%	NA	no rinse
Average	25.2	30%	28.7	59%	NA	NA
SD	8.60	10%	3.63	9.1%	NA	NA
%RSD	34%		13%	16%	NA	NA
50 mL + 50 mL +100 mL	67	79%	32	65%	76	76%
	72	84%	30	62%	85	85%
	66	77%	27	54%	79	79%
Average	68.1	80%	29.6	61%	80.3	80%
SD	3.10	3.6%	2.49	5.5%	4.68	4.7%
%RSD	4.5%		8.4%	9.1%	5.8%	
50 mL + 50 mL +200 mL	59	69%	30	62%	133	66%
	60	70%	31	67%	135	67%
	56	66%	29	63%	126	63%
Average	58.1	68%	30.1	64%	131	66%
SD	2.02	2.4%	1.27	2.2%	4.39	2.2%
%RSD	3.5%		4.2%	3.5%	3.3%	

**Table A-30. Phase II Optimization of Wetting Solvent and Chemical Recovery Using IPA and Bissell Little Green ProHeat Wet-Vacuum – TEP on Stainless-steel Coupon**

TEP						
Surface loading: 27 mg						
Wetting agent: IPA						
Procedure: vacuum conditioning – surface wetting- vacuum rinse (volumes, mL)	Chemical Recovered		Wetting Solvent Recovered (%)		Recovery of Rinse (%)	
	Mass (mg)	Recovery (%)	Volume (mL)	Recovery (%)	Volume (mL)	Recovery (%)
50 mL + 50 mL + no rinse	8.94	34%	24.2	54%	NA	no rinse
	5.40	20%	24.6	53%	NA	no rinse
	8.87	26%	28.6	58%	NA	no rinse
Average	7.07	27%	25.8	55%	NA	NA
SD	1.78	6.7%	2.47	2.9%	NA	NA
%RSD	25%		10%	5.2%	NA	NA
50mL + 50 mL +100 mL	11.3	43%	27.4	60%	75.8	76%
	13.1	50%	30.2	58%	79.4	79%
Average	11.2	46%	28.8	59%	77.6	78%
SD	1.27	4.8%	1.98	0.8%	2.52	2.5%
%RSD	10%		6.9%	1.4%	3.2%	
50mL + 50 mL + 100 mL	38	143%	27.1	56%	173	86%
	20	74%	29.9	65%	167	84%
Average	28.8	109%	28.5	60%	170	85%
SD	12.9	45%	1.98	6.1%	3.6	1.8%
%RSD	45%		6.9%	10.2%	2.1%	

**Table A-31. Phase III Optimized IPA Wet-Vacuum Method Performance for 100% Surface Concentration Reference Material Sampling Baseline of 2-CEPS, including Mass Balance Tests**

2-CEPS					
Material and Vacuum Type – Wetting Agent – Vol-Surface Lapse Time – Rinse Vol	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
Stainless-steel coupon – Bissell Little Green ProHeat – 50 mL IPA ( LT = 10 s) – 100 mL post rinse					
Target concentration: 100%					
TC-1	28	100	66%	21.4	76%
TC-2	28	118	73%	23.5	83%
TC-3	28	111	73%	20.6	73%
PB	not-spiked	111	73%	ND	NA
Average		110	71%	21.8	77%
Std Dev		7.6	3.7%	1.5	5.1%
% RSD		6.9%	5.2%	6.9%	6.9%
Mass balance samples					
TC- 1-VW		NA	NA	0.0011 ( J )	0.004%
TC- 2-VW		NA	NA	0.0011 ( J )	0.004%
TC- 3-VW		NA	NA	0.0010 ( J )	0.004%
TC- 1-AR1		89.7	89%	3.58	13%
TC- 1- AR2		89.9	90%	0.70	2.5%
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB – SL collected using wet-vacuum apparatus from non-contaminated (procedural blank) stainless-steel coupon; ND – Not detected; NA – Not applicable; LT – Surface lapse time (wetting agent surface contact time); VW - Post-vacuum sampling surface wipe; AR – Additional post-rinse; (J) – Estimated, reported value below lowest point of calibration curve.					

**Table A-32. Phase III Optimized TEP Wet-Vacuum Method Performance for 100% Surface Concentration Reference Material Sampling Baseline of TEP, including Mass Balance Tests**

TEP					
Material and Vacuum Type – Wetting Agent – Vol-Surface Lapse Time – Rinse Vol	Surface Loading (mg)	Volume of Wetting Solvent Collected (mL)	Wetting Liquid Recovered (%)	Mass of Chemical Collected (mg)	Chemical Recovery (%)
Stainless-steel coupon – Bissell Little Green ProHeat – 50 mL IPA ( LT = 10 s) – 100 mL post-rinse					
Target concentration: 100%					
TC-1	26	109	70%	18.5	72%
TC-2	26	111	74%	17.9	70%
TC-3	26	115	73%	19.7	77%
PB	not-spiked	109	73%	ND	NA
Average		111	72%	18.7	73%
Std Dev		2.9	1.7%	0.93	3.6%
% RSD		2.7%	2.4%	4.7%	4.7%
Mass balance samples					
TC- 1-VW		NA	NA	ND	NA
TC- 2-VW		NA	NA	ND	NA
TC- 3-VW		NA	NA	ND	NA
TC- 1-AR1		89.7	90%	1.73	6.7%
TC- 1- AR2		89.9	90%	0.32	1.2%
TC – SL collected using wet-vacuum apparatus from contaminated stainless-steel coupon; PB –, SL collected using wet-vacuum apparatus from non-contaminated (procedural blank) stainless-steel coupon; ND- Not detected; NA – Not applicable; LT – Surface lapse time (wetting agent surface contact time); VW - Post-vacuum sampling surface wipe; AR - Additional post-rinse; (J) – Estimated, reported value below lowest point of calibration curve					



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