EPA Public Access

Author manuscript

Sci Total Environ. Author manuscript; available in PMC 2020 March 10.

About author manuscripts

Submit a manuscript

Published in final edited form as:

Sci Total Environ. 2019 March 10; 655: 539–546. doi:10.1016/j.scitotenv.2018.11.128.

The impact of wipe sampling variables on method performance associated with indoor pesticide misuse and highly contaminated areas

Stuart A. Willison^a, Daniel Stout^a, Amy Mysz^c, James Starr^b, Dennis Tabor^d, Barbara Wyrzykowska-Ceradini^e, Josh Nardin^e, Eric Morris^e, Emily Gibbs^a

^aU.S. Environmental Protection Agency, Office of Research and Development, National Homeland Security Research Center, 26 Martin Luther King Dr., Cincinnati, OH 45268, United States of America

^bU.S. EPA, National Exposure Research Laboratory, 109 T.W. Alexander Drive, Durham, NC 27709, United States of America

^cU.S. EPA, Region 5, 77 W. Jackson Blvd., Chicago, IL 60604, United States of America

^dNational Risk Management Research Laboratory, 109 T.W. Alexander Drive, Durham, NC 27709, United States of America

^eJacobs Technology, Inc., 600 William Northern Blvd., Tullahoma, TN 37388, United States of America

Abstract

Pesticide misuse incidents in residential indoor areas are typically associated with misapplications that are inconsistent with the label directions of the product. Surface wipe sampling and analysis procedures are relied upon to evaluate the extent of indoor contamination and the remediation efforts successfully. In general, surface wipe sampling procedures are widely varied, which can complicate the comparison of the results and data interpretation. Wipe sampling parameters were studied for the insecticides malathion and carbaryl. The parameters evaluated include wipe media, wetting solvents, composite sampling, surface concentration, and the influence of differing product formulations. Porous and nonporous surfaces tested include vinyl tile, plywood and painted drywall (porous/permeable) and stainless steel and glass (nonporous/impermeable). Specific wipe materials included pre-packaged sterile-cotton gauze, pre-cleaned cotton twill, cotton balls, and a pre-packaged, pre-wetted wipe. Commercially available insecticide formulations were tested, and the results were compared to surfaces fortified with neat analytes to determine surface recovery results (efficiency). A sampling procedure to measure pesticide residues was developed, and variables associated with the sampling methods were evaluated to clarify how estimations of surface residues are impacted. Malathion recoveries were 73–86% for twill and pre-wetted, pre-packaged isopropanol wipes on nonporous materials. Malathion formulations ranged from 78 to 124% for pre-wetted, pre-packaged isopropanol wipes and cotton gauze wipes on nonporous materials. Carbaryl and carbaryl formulation recoveries were 82–115% and 77–110%, respectively, on nonporous surfaces for all tested wipe materials. While not every wipe sampling variable could be tested, the collected information from this study may be useful and applied to sampling plans for classes of chemicals with similar physicochemical properties.

Graphical abstract

Keywords

Pesticides; Misuse; Indoor areas; Wipe sampling; Sampling strategies

1. Introduction

Pesticides are commonly used in the United States to suppress peridomestic insect infestations in residential dwellings and commercial facilities. There is an increase in the number of pesticide misuse incidents in the U.S. due to noncompliance with the product's label directions, specifically the exceedance of label rates for application or the use of the product in a manner or location outside its registered use (MMWR, 2011). Moderate pesticide exposure can result in various human health effects, including neurological symptoms such as cognitive and psychomotor dysfunction (Kamel and Hoppin, 2004; Tinoco-Ojanguren and Halperin, 1998; Rubin et al., 2002; Ruckart et al., 2004). High concentrations of organophosphate or carbamate pesticides can depress cholinesterase activity in the blood and result in death (Kamel and Hoppin, 2004; Tinoco-Ojanguren and Halperin, 1998; Rubin et al., 2002; Ruckart et al., 2004; Roberts and Reigart, 2013). Pesticide misapplications occurring in human dwellings can result in an increased potential for human exposure and require remediation efforts to reduce or eliminate residues prior to reoccupation (Clark et al., 2002). Limited tools are available to evaluate pesticide residues on indoor surfaces, assess the potential risk to occupants and evaluate the effectiveness of cleaning procedures. Federal, State and local agencies responding to pesticide incidents may use different surface sampling procedures for organic chemicals on surfaces. These agencies seek guidance regarding best practices and the interpretation of surface sampling results. Current practices for collecting, processing, and analyzing surface wipe samples vary in these procedures (Clark et al., 2002; ASTM International, 2011; EPA/600/R-07/004, 2007). Examples of varied sampling parameters include wipe material and size, wipe solvent selection and volume, sampled surface area, and wiping technique.

Different wipe sampling procedures suggest there is no consensus on surface sampling methodology for collection of pesticide residues, particularly for pesticide indoor misuse situations (EPA/600/R-07/004, 2007; Mercier et al., 2011; Stout II et al., 2009; Carr and Hill, 1989; Deziel et al., 2011). Well-validated sampling procedures can delineate and quantify contaminated areas and contribute to the determination of the effectiveness of

decontamination efforts. Surface sampling findings can also be used to establish action thresholds and serve as a valuable tool for risk assessors. However, unless different surface sampling methods are similarly validated, their comparability may be diminished, and consequently, the results will be difficult to compare and interpret. Hence, a validated sampling guideline broadly designed and mutually applied to the various surface wipe approaches is needed to provide characterized methods for those responding to incidents of pesticide misuse. Many variables associated with surface sampling processes affect removal efficiency and the precision and accuracy of the results (ASTM International, 2011; Mercier et al., 2011; Stout II et al., 2009). Often, very little is known about how these variables affect method performance and if the variables are incorporated into sampling guidelines.

Many wipe sampling methods investigate target analytes at low concentration levels because that is where clearance decisions regarding civilian reoccupation are typically made. Surface sampling studies typically do not investigate the impact of high surface concentrations on method performance. Understanding the impact of high concentrations will ensure that dedicated wipe sampling and analysis procedures work as expected when used during the characterization and decontamination phases of an incident, avoiding underestimation of exposure at a range of concentrations. The composition of commercial pesticide formulations might impact surface sampling efficacies. Formulations are composed of technical grade pesticides (active ingredients) and inert ingredients that enhance solubility, handling, efficacy, safety, storage and UV stability, etc. that might cause the pesticide to interact with the wipe and impact the efficient removal of the pesticide from the surface. The performance of wipe sampling methods for neat solutions of pesticides may not directly correlate with the performance of these methods for solutions derived from commercial formulations. Surface sampling methods are needed that have considered factors that influence sampling variables and may affect data comparability.

To address the gaps outlined above, the following parameters were evaluated: wipe material type, wetting solvent (solvent used to wet a wipe material), surface materials (varied by composition and porosity), pesticide concentrations, commercially available pesticide formulations, and the number of sampling wipes used per surface (to inform total number of wipes required for composite sampling). The information and investigations described herein focus on understanding how the impact of specific variables associated with a wipe sampling procedure can affect target pesticide recoveries. The information can be used to assist decision-makers during pesticide misuse incidents and remediation of contaminated areas. Operational factors that inform sample collection procedures (e.g., material compatibility, health and safety considerations) were also considered. This information expands the existing knowledge of wipe sample collection and processing procedures, discusses the impacts of specific wipe sampling variables, and initiates a path towards developing more standard methods and guidelines for wipe sampling.

2. Materials and methods

2.1. Materials

Carbaryl and malathion standards were purchased from MilliporeSigma (St. Louis, MO). Carbaryl-D₇ and malathion-D₇ (pre-extraction standards) were purchased from CDN

Isotopes (Quebec, Canada). Carbaryl- $^{13}\text{C}_6$ and malathion- D_{10} (internal standards) were purchased from Cambridge Isotope Laboratories (Andover, MA). Acetone, dichloromethane, hexane, methanol, water, and acetonitrile were purchased from Fisher Scientific (Pittsburgh, PA) and were of pesticide analysis grade quality. Tested surfaces were cut to appropriate size and pre-cleaned prior to use. Tested surfaces included stainless metal (McMaster-Carr, Elmhurst, IL), vinyl tile (Lowe's, Mooresville, NC), glass, painted drywall (white, flat, latex-based paint) and plywood (Lowe's, Mooresville, NC). Commercial formulations of malathion (Ortho® Max Malathion, 50% malathion by weight, The Ortho Group, Marysville, OH) and carbaryl (Sevin®, 43% carbaryl by weight, Bayer, Research Triangle Park, NC) were purchased from the manufacturer or local hardware store. Multiple wipes were tested, including cotton gauze (CG) (Dukal™, 4 in. × 4 in. – 12-ply sterile cotton gauze pads, individually packaged, Fisher Scientific, Pittsburgh, PA), twill wipe (TW) (4 in. × 4 in., M. G. Chemicals, Toronto, Ontario, Canada), cotton ball (CB) (Swisspers® organic triple size, Cleveland, OH), filter paper (FP) (Whatman® Grade 1 Cellulose Qualitative, GE Healthcare, Little Chalfont, United Kingdom), and a pre-wetted isopropyl alcohol (IPA-PW) wipe [Cleantex™ TexPad Wipes; no residue, pre-saturated, 100% polyester wipe (91% IPA and 9% deionized water), Jacksonville, FL]. Nitrile gloves (Fisher Scientific, Pittsburgh, PA) were used to handle wipe materials while wiping the tested surface (except when working with acetone, latex gloves were used).

2.2. Wipe preparation

CG, FP, CB and IPA-PW wipes were used as received. TW wipes were pre-cleaned as described in the Supporting Information. Forceps were used to remove one clean wipe from its respective package or storage container and to place that wipe in a clean Petri dish. Calibrated pipettes were used to dispense 3 ml of solvent (acetone or IPA) onto the center of the wipe, and the Petri dish was covered to allow the solvent to disperse into the wipe material and reduce solvent loss. No additional solvent was added to the pre-packaged, pre-wetted IPA-PW wipe.

2.3. Solution preparation

Malathion spiking concentrations were prepared by adding the neat malathion standard (Malathion PESTANAL®, analytical standard, PN: 36143, MilliporeSigma, MO, USA) into hexane to achieve a concentration of 4 mg/ml. Carbaryl spiking solutions were prepared by adding the neat carbaryl standard (Carbaryl PESTANAL®, analytical standard, PN: 32055, MilliporeSigma, MO, USA) into dichloromethane to achieve a concentration of 24 mg/ml. Malathion and carbaryl technical formulations were made by diluting 2 ml and 30 ml of Ortho Max® Malathion and Sevin®, respectively, in water to achieve a final spiking concentration of 1 mg/ml and 6 mg/ml. The formulations were sonicated and heated to 60 °C to ensure sample homogeneity prior to sample spiking.

2.4. Sample spiking, collection, and extraction

A clean stainless steel template was placed over the sampling coupon to define the sampling area boundary and location of the spiking solution (see Supporting information). A clean template was used for each sampling coupon during the wipe sampling procedure to minimize the potential for cross-contamination between individual coupons. Twenty 50-μl

droplets of the 4 mg/ml malathion solution or 24 mg/ml carbaryl solution were deposited onto a 12 in. × 12 in. test surface using a pre-cleaned template to evenly distribute the drops across the surface (total mass of malathion and carbaryl deposited was 4 mg or 24 mg, respectively). Technical formulations were pipetted onto the test surfaces using twenty 200 µl aliquots of the pesticide solution for a final concentration of 4 mg/coupon and 24 mg/coupon of malathion and carbaryl, respectively, and allowed to dry at room temperature for approximately 45–60 min.

A systematic wiping procedure was utilized to wipe each test surface (see Supporting Information). The wetted wipe medium was folded in half between horizontal, vertical, and diagonal wiping directions. Finally, the wipe was folded and the perimeter of the test surface was wiped. Upon completion, the wipe was placed into a clean wide-mouth jar capped with a polytetrafluoroethylene (PTFE)-lined screw cap. The wipe was spiked with the pre-extraction standard to evaluate extraction efficiency (malathion-D₇ for malathion analysis or carbaryl-D₇ for carbaryl analysis for a final concentration of the isotopically-labeled analyte of 1000 µg per wipe). Hexane, for malathion samples, or 1:10 (v/v) acetone:hexane, for carbaryl samples, was added to the extraction jar, and the extraction jar was capped and sonicated for 15 min at room temperature. Depending on the concentration of the target analyte in the raw extract, different dilution factors were used for each analyte (see Supporting Information).

For malathion analysis, 20 to 980 µl of raw extract (depending on the concentration) was added to an amber glass autosampler vial pre-loaded with hexane and 20 µl of internal standard (IS)/surrogate standard mix (noted below) for a total volume of 1000 µl (1 ml) and vortexed for 30 s. A 10 ml aliquot of the raw hexane extract was placed in a 12-ml glass vial and archived for storage. The archived extract and prepared samples were stored at 4 °C until ready for analysis. For carbaryl analysis, 10 to 500 µl of raw extract (depending on concentration tested) was added to an amber glass autosampler vial pre-loaded with hexane and 20 µl of internal standard (IS)/surrogate standard mix (noted below) for a total sample volume of 1000 µl (1 ml) and vortexed for 30 s. A 10 ml aliquot of the raw acetone:hexane extract was placed in a 12 ml glass vial and archived for storage. The archived extract and prepared samples were stored at 4 °C until ready for analysis.

2.5. GC/MS analysis

Malathion and carbaryl sample extracts were analyzed by gas chromatography/mass spectrometry (GC/MS). The GC/MS, was operated in selected ion monitoring (SIM) mode, and GC/MS details are listed in the Supporting Information. The GC was equipped with a 60-meter DB-5 (0.25 mm × 0.25 µm) column (J&W Scientific, Folsom, CA, USA). The extract (1 µl) was injected using splitless mode. The GC ramp was started at 110 °C and ramped to 250 °C at 25 °/min with 5 min hold, followed by ramping to 325 °C and hold for 2.25 min). The injector was set at 150 °C with a 0.8 min splitless injection, then purge. The ion source and transfer line were set to 250 °C. Column flow was set for 1.3 ml/min.

Calibration standards were prepared and injected onto the GC/MS system prior to malathion and carbaryl extract analysis. The standards ranged from 500 to 5000 ng/ml for carbaryl and 500 to 10,000 ng/ml for malathion (see Supporting information). Acceptable relative

standard deviation of the response factor across the calibration region was less than or equal to 30%. Each standard included 1000 ng/ml of the IS and pre-injection spike (ERS-091 Internal Standards Mix and malathion-D₁₀ or carbaryl-¹³C₆, respectively). The labeled standards were added to diluted sample extracts prior to the GC/MS analysis (as described above). Overall target analyte validity was determined by the presence of the target ion, plus molecular ion/qualifier ions (Supporting information). The isotopic ratios and retention times of the samples were compared with those of the daily calibration standards. The NIST mass spectral library (NIST/EPA/NIH Mass Spectral Library (NIST 05), n.d.) was used to confirm the presence of malathion and carbaryl.

3. Results and discussion

3.1. Evaluation of wipe material and wetting solvent

Wipe material and wipe wetting solvent can impact method performance and wipe sampling results. Five different wipe types and two different wetting solvents were evaluated for pesticide transfer and extraction efficiency from the surface to a single wipe. Surface recoveries were evaluated on a nonporous, stainless steel metal surface (Table 1). General guidance for benchmark recovery levels (SW-846 Compendium, n.d.) were identified as 70–130% recovery with low variability

(RSD < 20%). Acetone was chosen as a wetting solvent because malathion and carbaryl are soluble in acetone, and the use of acetone may allow for easier extraction from the surface. Isopropyl alcohol (IPA) was selected because it is less destructive to surfaces, is easier to handle in a field setting, has been used in previous studies, and is commonly used by state pesticide regulatory agencies (Bernard et al., 2008; Tulse et al., 2006; Geno et al., 1996). Deziel et al. (2011) also note that wipe saturation and composition may affect surface recoveries. Optimal wipe saturation was evaluated for the wipe media (data not shown), and determined that wipe saturation was achieved with three milliliters of solvent.

3.1.1. Wetting solvent effects on malathion recovery from stainless steel—

Malathion recoveries for a single acetone-wetted wipe were 87(4), 98(5), 74(11) and 45(19) % (RSD) for TW, CG, CB and FP wipes, respectively (Table 1). Considering the size of the acetone-wetted CB wipe (1 in. × 1 in.), high transfer efficiencies from the metal surface were not anticipated. However, the results suggest that CB wipes may be an acceptable alternative wipe material for malathion recovery. Filter paper did not perform as well as the other tested wipe materials (45% recovery and higher variability with a RSD of 19%). Filter paper was anticipated to be less absorbent and retain less wetting solvent than the other tested materials. The physical characteristics of the filter paper allowed malathion to visibly remain and spread across the tested surface rather than absorb into the wipe (see Supporting information). Filter paper wipe materials produced a lower transfer efficiency from the metal to the wipe and resulted in larger variability among the collected data.

IPA-wetted wipes resulted in lower malathion recoveries for a single wipe (48(19), 64(18), 42(8) and 26(23) % (RSD) for TW, CG, CB and FP, respectively). Standard deviations for the tested wipe materials were also greater for IPA-wetted wipes than acetone-wetted wipes. IPA-PW wipes exhibited the highest malathion recoveries (118%) of all of the tested IPA-

wetted wipe materials. Since IPA-PW wipes were manufactured as pre-packaged, pre-wetted wipes, they contained a pre-determined amount of IPA and water. IPA-PW wipe materials may be of considerably different composition (more densely packed) than the other tested wipes. These factors may contribute to greater malathion recoveries than previously observed with the other IPA-wetted test wipes (Stout II et al., 2009; Carr and Hill, 1989).

An analysis of variance (ANOVA) compared wipe wetting solvents (IPA versus acetone) to determine if there were statistically significant differences for each wipe material. TW, CG, CB, and FP wipes exhibited a statistically significant ($p < 0.05$) difference, at the 95% confidence level, between acetone and IPA wetting solvents. The significance suggests that wetting solvent does affect analyte recovery. IPA-wetted wipe data represented higher variability and mostly lower recoveries. Acetone-wetted wipe data suggest that acetone was a better wetting solvent on the nonporous metal surface. This trend was also observed by Carr and Hill and may be associated with solubility of the target analytes (Carr and Hill, 1989). However, only a single wipe was used to collect the target analytes on the surface. Poor extraction efficiency with alternative wipe wetting solvents (e.g., IPA) may be overcome by using multiple wipes on each surface. Findings suggest acetone-wetted wipes were more efficient at recovering malathion from the metal surface, but using acetone could result in complications associated with wipe sample collection (e.g., surface destruction, leaching). Acceptable recoveries (>70%) can be attained with IPA wetting solvent as well (Bernard et al., 2008; Tulve et al., 2006). IPA-PW wipe recovery data are an example of achieving acceptable malathion recoveries with IPA as a wipe wetting solvent. Furthermore, IPA, rather than acetone, may be a more appropriate choice of wetting agent because of worker health and safety issues associated with the transporting and handling of acetone under field conditions and is widely embraced as a wipe solvent among those who conduct field sampling.

3.1.2. Wetting solvent effects on carbaryl recovery from stainless steel—

Carbaryl recoveries from a single acetone-wetted wipe were 85(7), 56(9), 86(4) and 62(18) % (RSD) for TW, CG, CB and FP wipes, respectively (Table 1). Carbaryl standard deviations for average acetone-wetted wipes were low (<10), except for FP wipes. As previously mentioned, the absorptive properties of the FP wipe likely played a role in diminished recovery results. Similar to the malathion results, the composition of the wipe media may again play a role in carbaryl extraction efficiencies from the surface to the wipe material. For example, TW wipes are more tightly woven, and CB wipes can be described as a denser material than the CG wipes. Denser materials may allow for better solvent retention and enhanced analyte surface recovery. Polar pesticides such as carbamates might be easily solubilized in IPA and allow for greater uptake into wipe media (ASTM International, 2011; Carr and Hill, 1989). Carbaryl transfer efficiencies for IPA-wetted TW, CG, CB, and IPA-PW wipe materials were [68(5), 73(6), 63(16) and 81(7) % (RSD), respectively]. IPA-wetted FP wipe recoveries were lower ($57 \pm 6\%$) from the metal surface. The relative standard deviations were <10 for all tested wipes except IPA-wetted CB wipes and acetone-wetted FP wipes.

An ANOVA statistical analysis compared wipe wetting solvents (IPA versus acetone) to determine if there were statistically significant differences for each wipe material. TW, CG

and CB wipes all exhibited statistically significant differences ($p < 0.05$), at the 95% confidence level, between acetone and IPA wetting solvent. The data suggest that wetting solvent does play a role in analyte recovery. FP wipes did not exhibit a statistically significant difference between the two wetting solvents; however, the large variance in the FP recovery results may have caused the lack of statistical significance. Furthermore, FP wipe carbaryl recoveries for both wetting solvents were below acceptable levels (70% recovery). It is unlikely that the FP wipe would have been able to achieve recovery benchmark levels (70% recovery and RSD < 20%) regardless of wetting solvent.

3.2. Selecting an appropriate wetting solvent and wipe

Selecting an appropriate wetting solvent was difficult. Acetone-wetted wipes exhibited high extraction efficiencies for malathion and carbaryl across many of the tested wipe materials. However, the number of IPA-wetted wipes used to wipe each surface could be increased to improve wipe recovery results. Since wipe media composition may also play an important role, composite sampling should improve surface sampling recoveries. Acetone-associated matrix effects (e.g., leaching, surface degradation, etc.), worker health and safety concerns, and overall acceptance of IPA as a wipe solvent in the community that conducts sampling are factors that would need to be considered if acetone were used. Acetone might impact sampling recoveries because it is suspected to facilitate the penetration of the target analytes into porous/permeable surfaces, be destructive to tested surfaces (e.g., plastics and paint), and cause paints or colors to percolate from tested materials (EPA/600/R-07/004, 2007; Carr and Hill, 1989). These complications may create matrix interferences and potentially highly variable data. Therefore, IPA wetting solvent was selected for all remaining experiments instead of acetone.

Four potential wipe materials were selected to use in conjunction with IPA solvent; TW, CG, CB and IPA-PW. Commercial-off-the-shelf products (CG, CB, and IPA-PW) were appealing because they are commercially available and the tested wipes did not require additional preparation (e.g., pre-cleaning or processing steps). TW wipes are commercially available, but typically require pre-cleaning prior to use (Deziel et al., 2011); however, further evaluation was warranted because the wipe performed well during initial testing. Additional wipes that are similar to those tested in this investigation may also perform well as a surface wiping material. Wipe preparation and processing of any additional wipe materials should be evaluated before use to determine if pre-cleaning is required. The selected wipe materials were able to recover malathion and carbaryl on the surface with relatively low variability (RSD < 20%) using a single wipe. All four wipe materials (TW, CG, CB and IPA-PW) were subjected to further testing in to obtain a clearer representation of wipe performance.

3.3. Evaluation of concentration and composite sampling effects

Surfaces spiked with malathion and carbaryl were sampled using multiple IPA-wetted TW, CG, CB and IPA-PW wipes. Different wipe materials were studied to determine a potential optimal wipe. High and low concentration levels of each analyte were also investigated on a metal surface and are presented in Table 2, Table 3. While higher variability was associated with IPA wetting solvent, using multiple wipes on the same surface may reduce the overall variability if the samples are composited. Multiple wipes (two or three) were used for each

tested surface and analyzed separately to determine the analyte quantities obtained from each wipe. Separate analysis of each wipe also determined the total number of wipes needed to recover the target analytes in sufficient quantities (70%) from each surface. Concentration effects may also play a role on extraction efficiency and variability (Stout II et al., 2009). High concentration levels were selected based on the highest reported values identified by local and state authorities investigating pesticide misuse cases in urban settings in the United States.

3.3.1. Concentration and composite sampling effects on malathion recovery

—High concentrations (4 mg/coupon) of malathion were spiked onto a metal surface, the appropriate wipe material was wetted with IPA solvent, and the wipe was used to sample the surface. Malathion recoveries were considerably higher for TW wipes (~75%) than CG wipes (~30%) (Table 2). Concentration effects were previously studied by Deziel and Bernard (Deziel et al., 2011; Bernard et al., 2008), but the variance in the data between the two studies may have been attributed to the amount of wetting solvent used for the wipe media rather than effects related to pesticide concentration.

Although intended to be representative of a composite sample, each wipe within a composite sample was analyzed separately to determine the recovery from a surface that is wiped multiple times. TW wipes were able to recover nearly half of the malathion deposited on the surface with the first wipe; however, considerable quantities (~25% of the original amount) were recovered with a second wipe as well. The third wipe did not recover (~4–8%) as much as the first two wipes. The data suggest that most of the material can be collected within two wipes. CG wipes did not efficiently recover malathion from the tested surface, with total recoveries of <35%. While acceptable malathion recovery was possible with the first two CB wipes, CB wipes may require more than two wipes to achieve benchmark recovery levels (70% recovery and RSD < 20%). CB wipe materials are smaller in size, resulting in potential solvent and wipe saturation. CB wipe materials may not retain as much of the target analyte within the first wipe as other wipe materials. The observed recovery distribution was more evenly spread across the first two CB wipes, unlike TW wipes where the first wipe collected a majority of the malathion. Similar trends were reported with a single TW wipe by Deziel et al. (Deziel et al., 2011). IPA-PW wipes were able to recover considerable quantities of the malathion within the first wipe. As previously mentioned, wipe composition (e.g., pre-wetted with water and IPA and/or densely woven) may likely play a role, especially when related to wetting solvent effects. While denser wipe materials may aid in analyte recoveries, loosely woven materials could reduce the ability of the wipe to recover target analytes from the surface. This effect may be observed (negatively impacting recovery) for CG wipes and (positively impacting recovery) for IPA-PW wipes.

Similar trends were observed at lower (0.3 mg/coupon) malathion concentrations (Supporting Information). All the tested wipes were able to recover malathion at benchmark levels (70%) and exhibit low variability (RSD < 7%), except CG wipes. Greater than 70% malathion recoveries were achieved by using only two CB and IPA-PW wipes. Two or three wipes may be needed to achieve 70% recoveries for TW wipes, based on the recoveries presented in (Supporting information). CG wipes performed poorly and recoveries at low concentrations were similar to high concentrations.

An ANOVA statistical analysis compared concentration effects (high versus low) to determine if there were statistically significant differences ($p < 0.05$) at the 95% confidence level. The analysis indicated that there was no statistical significance between tested malathion application concentrations for TW, CG, and CB wipes on the metal surface. Varying the concentration did not appear to have a significant effect on malathion wipe recoveries on a nonporous surface. A statistical significance was observed for IPA-PW wipes relative to concentration; however, IPA-PW wipe recoveries were $>70\%$ for malathion at both concentration levels. Overall, malathion concentrations (at high and low levels) appear to be less important than wipe material, number of wipes used on the surface (composite sampling), and wetting solvent, especially if benchmark recoveries are possible. Three potential wipe materials (TW, CB, and IPA-PW wipes) could be suitable for surface wiping of malathion on a nonporous surface.

3.3.2. Concentration and composite sampling effects for carbaryl recovery—

High concentrations (24 mg/coupon) of carbaryl were spiked on a metal surface. Carbaryl recovery results were similar for TW and CG wipe materials (Table 3). Most of the carbaryl spiked on the metal surface was recovered with the first TW and CG wipe (87–106%), with $<19\%$ recovered with the second wipe for both wipe materials. The third wipe collected only small quantities (2–8%) of carbaryl on the surface. IPA-PW wipes exhibited recoveries similar to TW and CG wipes, but at lower recovery concentrations for the first wipe. Nonetheless, two IPA-PW wipes were sufficient to recover carbaryl from the surface ($>92\%$). Two or three CB wipes were needed to achieve a recovery efficiency of $>70\%$, with most of the target analyte recovered with the first two wipes. All four of the tested wipes were able to achieve carbaryl recoveries greater than the benchmark level ($>70\%$) with low variability ($RSD < 9\%$).

The observed trend for carbaryl recoveries at lower concentrations (0.5 mg/coupon) was similar to the trend observed at high concentrations for TW and IPA-PW wipes ($>70\%$ and $RSD < 6\%$) (Supporting Information). CB wipe recoveries were lower (65%) and CG wipe recoveries were more variable ($RSD 27\%$). TW and IPA-PW wipes were able to recover most of the analyte within the first two wipes. CG and CB wipe recovery results were lower and required a third wipe to achieve benchmark levels. It is uncertain whether lower recoveries were due to analytical complications, the degradation of the analyte at low levels, or related to wipe material composition. Nonetheless, wipe composition, number of wipes, and wetting solvent appear to be more of a factor than concentration for carbaryl recoveries. It is important to note that concentration effects may have a greater impact if degradation is occurring.

An ANOVA statistical analysis (high versus low concentration) for carbaryl suggests that statistically significant recoveries occurred with IPA-PW and CB wipes. Again, carbaryl wipe recoveries for IPA-PW were $>70\%$ at both concentrations so a statistical significance associated with concentration effects may be less important than wipe material and wetting solvent. Concentration effects were not statistically significant for TW and CG wipes. The data suggest that a third wipe may provide only minimal analyte recoveries from the surface for malathion and carbaryl regardless of surface concentration. Wetting solvent and wipe type (composition) may play a larger role than concentration levels on a nonporous surface.

3.4. Selecting appropriate materials for additional testing

TW and IPA-PW wipe materials were able to yield acceptable malathion and carbaryl recoveries (>70%) and low variability (RSD < 20%) at both concentration levels. TW and IPA-PW wipes were selected for further testing based on their performance. CB and CG wipe materials resulted in higher variability, lower recoveries, or both at the tested malathion or carbaryl concentrations. Despite the poor performance of the CG wipe material, CG wipes are commonly used in the field and readily available as a nationwide commercial product. Further evaluation of the CG wipe may provide important information for field samplers therefore, TW, IPA-PW, and CG wipe materials were selected for further evaluation on different surfaces.

3.5. Surface effects on malathion and carbaryl recovery

Porous surfaces are problematic because they may allow target analytes to penetrate into the material, which may be further exacerbated when solvent from a wetted wipe is applied to the surface. Information is still needed to represent surface sampling performance in real-world scenarios for surfaces that vary in surface composition and porosity. Valuable information can still be obtained from sampling porous surfaces, even though it is unlikely that recoveries will achieve benchmark levels.

TW, CG, and IPA-PW wipes were tested on a variety of surfaces (Table 4). High malathion and carbaryl (4 mg and 24 mg, respectively) concentrations were selected to represent potential pesticide misuse concentrations. Two wipes were used to wipe each surface, and the two wipes were analyzed as a composite sample for total malathion or carbaryl recovery. Malathion recoveries were >70% on glass (nonporous surface) for IPA-PW and CG wipes. Carbaryl recoveries were 82 and 96% for IPA-PW and CG wipes, respectively. Malathion and carbaryl recoveries from porous surfaces were <8% for vinyl, plywood, and painted drywall. TW wipes were not tested on the glass surface, but it is expected that TW wipe materials would have achieved acceptable recoveries.

3.6. Technical formulation effects on malathion and carbaryl recovery

The impacts of commercial product formulations in wipe method performance were tested (Table 4). The wipe media were tested using diluted Ortho® Max Malathion (technical malathion formulation) and Sevin® (technical carbaryl formulation) (Supporting information). The technical formulations and the neat malathion and carbaryl solutions were compared on metal and glass surfaces and across multiple wipe types.

Recovery results for TW, IPA-PW, and CG wipes on glass and metal surfaces were (73–78%) for malathion, (61–124%) for technical malathion, (82–96%) for carbaryl, and (77–132%) for technical carbaryl. Higher variation was observed from the technical formulation recoveries. Technical formulations can be complex proprietary mixtures of chemicals that create analysis complications versus a single chemical, so the higher variation is reasonable, expected, and likely the cause for >100% recovery results. Overall, TW and IPA-PW wipe materials performed well for both the neat and technical formulations of malathion and carbaryl on nonporous, nonpermeable surfaces.

An ANOVA statistical analysis compared neat analyte solutions and technical formulations to determine if there were statistically significant differences ($p < 0.05$) at the 95% confidence level on nonporous/nonpermeable surfaces. The data suggest that there is a statistical significance in the recovery results between neat malathion and technical malathion on glass for IPA-PW wipes. However, as previously observed with IPA-PW wipes, the recoveries were $>70\%$ and variance for each data set was low. A matrix interference associated with technical malathion data may have resulted in the cause for the statistical significance between neat malathion and technical malathion recovery results. Benchmark recovery levels ($>70\%$) were still achieved for both neat malathion and technical formulation. No statistical significance was observed between malathion and technical malathion on metal for IPA-PW wipes. There was no statistical significance for CG wipes and malathion and technical malathion on glass, but a significance was observed on metal. CG recoveries for technical malathion were $<70\%$. A significant difference for CG wipes, between neat malathion recoveries and technical malathion, on the metal surface were observed (Table 4).

An ANOVA statistical analysis compared neat carbaryl and the technical carbaryl formulation. The data suggest that IPA-PW and CG wipes both resulted in a statistically significant difference in recoveries on the glass surface. IPA-PW results continue to demonstrate recoveries above the benchmark level with low variance between each data point despite the statistical significance. An ANOVA analysis comparing CG wipes and neat carbaryl and carbaryl formulation on metal did not result in a statistically significant difference.

3.7. Technical formulation effects on porous/permeable surfaces

As expected, wipe sampling results from technical formulations on porous surfaces exhibited lower recoveries than from nonporous surfaces (Table 4). The only exception was the TW wipe and the technical carbaryl solution on vinyl tile. Unfortunately, only the TW wipe was tested on the vinyl surface, so it is unknown whether the wipe type, surface, or the technical formulation played a significant role in carbaryl recovery. Neat carbaryl solution recoveries on the vinyl surface were considerably lower (8%) than the solution recoveries of the technical carbaryl (82%), and the technical carbaryl samples exhibited low variability ($RSD < 11\%$). The carbaryl technical formulation selected for this study was water-based while the neat carbaryl solutions were in organic solvent. The lack of organic solvent may have allowed the technical formulation to remain on the surface. It is also possible that the constituents of the technical formulation, which are designed to stabilize and aid in the efficacy of carbaryl for its intended use, may be responsible for allowing the carbaryl to remain on the surface rather than penetrate into it. A myriad of factors can affect reproducibility and analyte recovery as noted by Cettier et al. (Cettier et al., 2015). Any one of these scenarios would result in enhanced recoveries for the technical formulation over the technical grade on the vinyl surface.

Malathion, technical malathion, carbaryl and technical carbaryl recoveries on the painted drywall were low ($<18\%$). Painted drywall surfaces can create several complications. Drywall can be considered a porous surface. Matrix interferences associated with the paint

dissolving in the wetting solvent during laboratory sample extraction and preparation procedures will occur and were observed. High variability was associated with painted drywall despite the wipe material. Painted drywall continues to be a complex surface and problem for surface wipe sampling and analysis.

4. Conclusions

Current sampling plans that are described in the literature and used during operational activities do not adequately address the effects that sampling variables have on recovery efficiency results. Understanding the underlying cause for how well (or poorly) a specific wipe medium performs is important if it is to be used during remediation activities. In this study, we examined the impacts of wipe media, wipe solvent (solvent used to wet a wipe material), surface materials, pesticide concentrations, technical formulations, and the number of sampling wipes used per surface (to inform composite sampling) on wipe sampling method performance. A wipe sampling procedure was established and used so that each variable could be controlled and considered reproducible, without creating a significant variance during sampling. Because common wipe types and solvents, as well as other representative conditions, were studied, these findings can assist decision makers in understanding and interpreting sampling data.

This study adds insight into the use of multiple wipes on a single surface, which in some instances may be used to obtain acceptable wipe recoveries (>70%) with low variability (RSD < 20%). For example, multiple wipes may improve recovery results when using a wetting solvent that is less compatible with the target analyte or a wipe that is not efficient at extracting the analyte from the surface due to size or composition. The data also suggest that most of the target analytes can be recovered with two wipes, after which recovery is reduced when additional wipes are used to wipe the same surface. Wipe media, such as the CG wipe, resulted in higher variability and wider recovery ranges than other tested wipes, which may be problematic during field applications. A pre-packaged, pre-moistened wipe, like the IPA-PW material, may perform better than other tested wipes, simply because it may reduce the number of variables associated with wipe sampling (wetting solvent, wipe composition/density, etc.). Further testing is needed to confirm a best wipe material and, more importantly, chemical compatibility with each wipe type.

This study provides critical insights into wipe sampling method performance that may be applicable to operational use in the field when high pesticide surface concentrations are present. Generally speaking, neither high concentrations of the tested chemicals nor technical formulations of the target analytes appear to affect wipe recovery results on a nonporous surface. It is important to note that there are different types of technical formulations (wetable powders, emulsifiable concentrates, microencapsulations, etc.) that may impact recovery results. Further testing is needed to evaluate additional technical formulations and active ingredients with differing physicochemical properties. The data suggest that wetting solvents did affect recovery results, and additional wetting solvents should be investigated to assess efficacy. Regardless of data availability, individuals employing these methods should be cognizant of such inherent variability and potential effects on method performance. Furthermore, when an observed statistically significant

difference related to concentration and formulation effects occurs, as with IPA-PW wipes, the result may not be as meaningful if benchmark recovery levels (and low variability) were easily achieved.

Finally, certain trends were observed that may be applicable to chemicals that are structurally similar or contain comparable physicochemical properties as malathion and carbaryl (e.g., carbamates or organophosphorus-based chemicals). Although the information should not be generalized for all chemicals, some of the information obtained from this investigation can help drive future method development, improve method performance, and assist the assessment of surface wipe sampling data when evaluating pesticides with similar properties. Sampling variables will inevitably affect recovery results, and these variables should be carefully considered when developing sampling plans.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

Acknowledgments and disclaimer

The U.S. Environmental Protection Agency (EPA) through its Office of Research and Development funded and managed the research described herein under EPA Contract EP-C-15-008, Work Assignment # 2-084 with Jacobs Technologies Inc. It has been reviewed by the Agency but does not necessarily reflect the Agency's views. No official endorsement should be inferred. EPA does not endorse the purchase or sale of any commercial products or services.

References

- ASTM International, 2011 ASTM International Standard Practice for Field Collection of Organic Compounds from Surfaces Using Wipe Sampling D6661-10 (2011)
- Bernard et al., 2008 Bernard BE, Berry MR, Wymer LJ, Melnyk LJ Sampling household surfaces for pesticide residues: comparison between a press sampler and solvent-moistened wipes *Sci. Total Environ.*, 389 (2008), pp. 514–521
- Carr and Hill, 1989 Carr BL, Hill DF Sampling of Common Pesticides and PCBs from Inert Surfaces EPA/330/1-90-001, National Enforcement Investigations Center, Denver, CO (10 1989)
- Cettier et al., 2015 Cettier J, Bayle ML, Béranger R, Billoir E, Nuckols JR, Combourieu B, Fervers B Efficiency of wipe sampling on hard surfaces for pesticides and PCB residues in dust *Sci. Total Environ.*, 505 (2015), pp. 11–21
- Clark et al., 2002 Clark J, Bing-Canar J, Renninger S, Dollhopf R, El-Zein J, Star D, Zimmerman D, Anisuzzaman A, Boylan K, Tomaszewski T, Pearce K, Yacovac R, Erlwein B, Ward J Methyl parathion in residential properties: relocation and decontamination methodology *Environ. Health Perspect.*, 110 (6) (2002), pp. 1061–1070
- Deziel et al., 2011 Deziel NC, Viet SM, Rogers JW, Camann DE, Marker DA, Hukkinen MS, Yau AY, Stout DM II, Dellarco M Comparison of wipe materials and wetting agents for pesticide residue collection from hard surfaces *Sci. Total Environ.*, 409 (2011), pp. 4442–4448 [PubMed: 21816452]
- EPA/600/R-07/004, 2007 EPA/600/R-07/004 A Literature Review of Wipe Sampling Methods for Chemical Warfare Agents and Toxic Industrial Chemicals, Prepared by Battelle, Columbus, OH 43201, for Stephen Billets U.S. Environmental Protection Agency, Office of Research and Development National Exposure Research Laboratory, Environmental Sciences Division, Las Vegas, NV 89119 (1 2007)
- Geno et al., 1996 Geno PW, Camann DE, Harding HJ, Villalobos K, Lewis RG Handwipe sampling and analysis procedure for the measurement of dermal contact with pesticides *Arch. Environ. Contam. Toxicol.*, 30 (1996), pp. 132–138

- Kamel and Hoppin, 2004 Kamel F, Hoppin JA Association of pesticide exposure with neurologic dysfunction and disease *Environ. Health Perspect.*, 112 (9) (2004), pp. 950–958
- Mercier et al., 2011 Mercier F, Glorennec P, Thomas O, Le Bot B Organic contamination of settled house dust, a review for exposure assessment purposes *Environ. Sci. Technol.*, 45 (2011), pp. 6716–6727 [PubMed: 21667945]
- MMWR, 2011 MMWR Acute Illnesses Associated With Insecticides Used to Control Bed Bugs — Seven States, 2003–2010 60, Centers for Disease Control and Prevention (2011), pp. 1270–1274
- >NIST/EPA/NIH Mass Spectral Library (NIST 05), n.d NIST/EPA/NIH Mass Spectral Library (NIST 05) <https://chemdata.nist.gov/>, Accessed 23rd Apr 2018
- Roberts and Reigart, 2013 Roberts JR, Reigart JR Recognition & Management of Pesticide Poisonings (6th ed.), EPA 735K13001, U.S. Environmental Protection Agency, Office of Pesticide Programs (2013)
- Rubin et al., 2002 Rubin C, Esteban E, Kieszak S, Hill RH Jr., Dunlop B, Yacovac R, Trotter J, Boylan K, Tomaszewski T, Pearce K Assessment of human exposure and human health effects after indoor application of methyl parathion in Lorain County, Ohio, 1995–1996 *Environ. Health Perspect.*, 110 (6) (2002), pp. 1047–1051
- Ruckart et al., 2004 Ruckart PZ, Kakolewski K, Bove F, Kaye WE Long-term neurobehavioral health effects of methyl parathion exposure in children in Mississippi and Ohio *Environ. Health Perspect.*, 112 (1) (2004), pp. 46–51
- Stout II et al., 2009 Stout DM, Bradham KD, Egeghy PP, Jones PA, Croghan CW, Ashley PA, Pinzer E, Friedman W, Brinkman MC, Nishioka MG, Cox DC American healthy homes survey: a national study of residential pesticides measured from floor wipes *Environ. Sci. Technol.*, 43 (2009), pp. 4294–4300
- SW-846 Compendium, n.d SW-846 Compendium Test Methods for Evaluating Solid Waste: Physical/ Chemical Methods Compendium <https://www.epa.gov/hw-sw846/sw-846-compendium>, Accessed 23rd Apr 2018
- Tinoco-Ojanguren and Halperin, 1998 Tinoco-Ojanguren R, Halperin DC Poverty, production and health: inhibition of erythrocyte cholinesterase via occupational exposure to organophosphate insecticides in Chiapas, Mexico *Arch. Environ. Health*, 53 (1998), pp. 29–35 [PubMed: 9570306]
- Tulve et al., 2006 Tulve NS, Jones PA, Nishioka MG, Fortmann RC, Croghan CW, Zhou JY, Fraser A, Cave C, Lewis RG Pesticide measurements from the first National Environmental Health Survey of child care centers using a multi-residue GC/MS analysis method *Environ. Sci. Technol.*, 40(2006), pp.6269–6274

Highlights

- Pesticide contamination poses health exposure risks to responders and public.
- Results are hard to compare and interpret if multiple sampling procedures are used.
- Wipe media and wipe wetting solvents affect recovery results.
- High concentrations and technical formulations did not affect wipe recovery results.
- Composite sampling improves recoveries for less compatible solvents and wipe media.

Table 1.

Average wipe recovery results from a single wipe on steel surface varying wipe type and wetting solvent.

Average (<i>n</i> = 5) wipe recovery results for malathion and carbaryl on stainless steel										
	Cotton twill (TW)		Cotton gauze (CG)		IPA-pre-wetted (IPA-PW)		Cotton ball (CB)		Filter paper (FP)	
Wetting solvent	Acetone				N/A		Acetone			
	% Recovery	% RSD	% Recovery	% RSD	% Recovery	% RSD	% Recovery	% RSD	% Recovery	% RSD
Malathion ^a	87	4	98	5	N/A	–	74	11	45	19
Carbaryl ^a	85	7	56	9	N/A	–	86	4	62	18
Wetting solvent	IPA									
	% Recovery	% RSD	% Recovery	% RSD	% Recovery	% RSD	% Recovery	% RSD	% Recovery	% RSD
Malathion ^a	48	19	64	18	118	18	42	8	26	23
Carbaryl ^a	68	5	73	6	81	7	63	16	57	6

^aSpiked nominal concentrations for malathion and carbaryl were 4 mg/ml and 24 mg/ml, respectively. % RSD-percent relative standard deviation, N/A-not applicable since acetone was not added to wipe material.

Table 2.

Malathion wipe recoveries from IPA-wetted TW, CG, CB and IPA-PW wipes at high concentration (4 mg/coupon (sample analysis concentration of 4000 ng/ml)) on metal surface using IPA wetting solvent. Wipes were analyzed separately.

Malathion wipe recoveries from metal surface at high concentration (4 mg/coupon)				
Cotton twill (TW)			Cotton gauze (CG)	
Wipe #	Recovered concentration (ng/ml)	% Recovery	Recovered concentration (ng/ml)	% Recovery
1A	2131	53	629	16
2A	703	18	386	10
3A	171	4	286	7
Composite	3005	75	1301	33
1B	1898	47	708	18
2B	748	19	280	7
3B	329	8	279	7
Composite	2974	74	1267	32
1C	1892	47	399	10
2C	740	19	387	10
3C	337	8	264	7
Composite	2969	74	1050	26
Composite % RSD		1	Composite % RSD	11
Cotton ball (CB)			IPA pre-wetted (IPA-PW)	
Wipe #	Recovered concentration (ng/ml)	% Recovery	Recovered concentration (ng/ml)	% Recovery
1A	1549	39	2829	71
2A	1137	28	669	17
3A	352	9	228	5
Composite	3038	76	3726	93
1B	1193	30	2461	62
2B	1111	28	776	19
3B	639	16	200	5
Composite	2943	74	3438	86
1C	2237	56	2368	59
2C	783	20	698	18
3C	258	6	255	6
Composite	3278	82	3321	83
Composite % RSD		6	Composite % RSD	6

Table 3.

Carbaryl recoveries from IPA-wetted TW, CG, CB and IPA-PW wipes at high concentration (24 mg/coupon (sample analysis concentration of 4800 ng/ml) on metal surface. Wipes were analyzed separately.

Carbaryl recoveries from metal surface at high concentration (24 mg/coupon)					
Cotton twill (TW)			Cotton gauze (CG)		
Wipe #	Recovered concentration (ng/ml)	% Recovery	Recovered concentration (ng/ml)	% Recovery	
1A	4169	87	5002	104	
2A	608	13	214	4	
3A	261	5	119	3	
Composite	5038	105	5335	111	
1B	4650	97	5106	106	
2B	895	19	266	6	
3B	401	8	131	3	
Composite	5946	124	5503	115	
1C	4324	90	4691	98	
2C	898	19	230	5	
3C	276	6	111	2	
Composite	5498	115	5032	105	
Composite % RSD		8	Composite % RSD		5
Cotton ball (CB)			IPA pre-wetted (IPA-PW)		
Wipe #	Recovered concentration (ng/ml)	% Recovery	Recovered concentration (ng/ml)	% Recovery	
1A	2373	49	3641	76	
2A	794	17	781	16	
3A	531	11	407	9	
Composite	3698	77	4829	101	
1B	3203	67	3142	66	
2B	734	15	1244	26	
3B	242	5	402	8	
Composite	4180	87	4788	100	
1C	2569	54	3649	76	
2C	954	20	978	20	
3C	502	10	386	8	
Composite	4025	84	5013	104	
Composite % RSD		6	Composite % RSD		3

Table 4.

Average recoveries (n = 5) of malathion, carbaryl, and technical formulations from surfaces with an IPA-wetted twill wipe (TW), cotton gauze (CG), and pre-packaged, pre-wetted IPA wipe (IPA-PW).

Malathion (4 mg/coupon)			Carbaryl (24 mg/coupon)	
Twill wipe (TW)			Twill wipe (TW)	
Surface type	Average recovered concentration * (ng/ml) (±SD)	Average % recovery	Average recovered concentration * (ng/ml) (±SD)	Average % recovery
Vinyl	286 (20)	7	384 (110)	8
Plywood	40 (10)	1	32 (7)	1
IPA pre-wetted (IPA-PW)			IPA pre-wetted (IPA-PW)	
Painted drywall	52 (10)	1	50 (10)	1
Glass	2905 (60)	73	3926 (120)	82
Cotton gauze (CG)			Cotton gauze (CG)	
Painted drywall	150 (60)	4	69 (20)	1
Glass	2858 (200)	71	4615 (400)	96
Average (n = 5) recovery of technical formulations on surfaces				
Technical formulation malathion (ortho™ max)			Technical formulation carbaryl (sevin™)	
Surface type	Average recovered concentration * (ng/ml) (±SD)	Average % recovery	Average recovered concentration * (ng/ml) (±SD)	Average % recovery
Twill wipe (TW)			Twill wipe (TW)	
Vinyl	92 (10)	2	3957 (440)	82
Plywood	30 (5)	1	35 (4)	1
Metal	2449 (60)	61	5010 (230)	104
IPA pre-wetted (IPA-PW)			IPA pre-wetted (IPA-PW)	
Painted drywall	60 (15)	2	312 (90)	7
Glass	4933 (150)	124	4830 (100)	77
Metal	3117 (160)	78	N/A	N/A
Cotton gauze (CG)			Cotton gauze (CG)	
Painted drywall	112 (30)	3	865 (300)	18
Glass	3940 (670)	99	5257 (430)	110
Metal	3290 (450)	110	5261 (100)	110

N/A-wipe was not tested with formulation on surface.

* Spiked nominal concentration values were 4 mg and 24 mg per surface for malathion and carbaryl, respectively. Solution concentrations after dilution were 4000 ng/ml and 4800 ng/ml for malathion and carbaryl, respectively.