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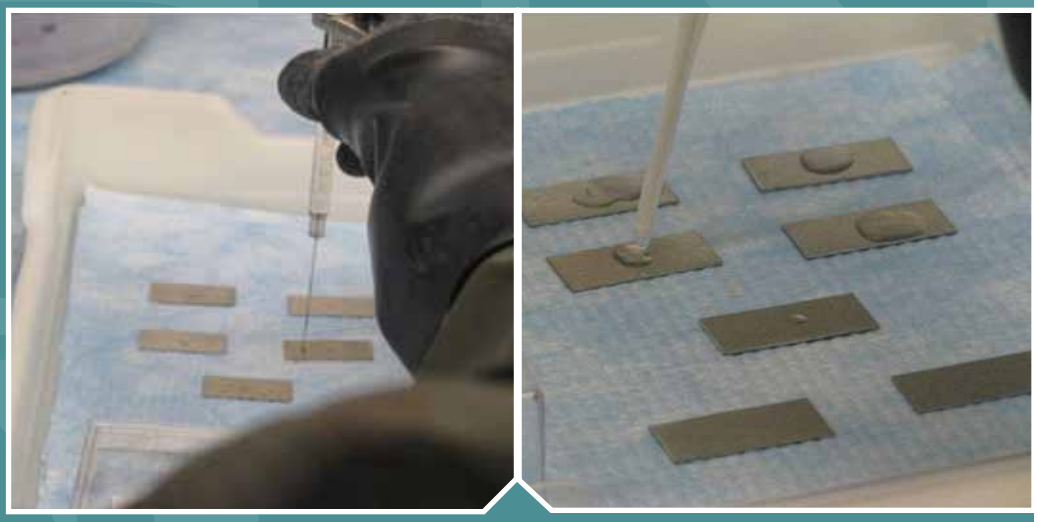
United States
Environmental Protection
Agency

Evaluation of Household or Industrial Cleaning Products for Remediation of Chemical Agents

EVALUATION REPORT



Office of Research and Development
National Homeland Security Research Center

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May 2011

Evaluation Report

Evaluation of Household or Industrial Cleaning Products for Remediation of Chemical Agents

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
RESEARCH TRIANGLE PARK, NORTH CAROLINA 27711

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Executive Summary

The U.S. Environmental Protection Agency (EPA) is the primary federal agency responsible for remediation of indoor and outdoor areas in the aftermath of a terrorist incident in which chemical agents are released. Therefore, as a part of EPA's homeland security research program, EPA conducts research to help first responders and decision-makers minimize environmental impact and human health effects following the release of a chemical agent. EPA has commissioned this evaluation into the efficacy of household or industrial cleaning products when applied to the cleanup of chemical agents. Bench-scale testing was utilized to evaluate the efficacy of household or industrial cleaning products on indoor surfaces contaminated with chemical agents agents (i.e., thickened sulfur mustard [THD], thickened soman [TGD], V-series nerve agent [VX], and sulfur mustard [HD]). The cleaning technologies evaluated were OxiClean® Versatile Stain Remover Powder, Zep® Cleaner and Degreaser Concentrate, K-O-K® Household Bleach (sodium hypochlorite, 5.25%), and Cascade® with Extra Bleach Action Gel dishwashing detergent. For cleaners that may reasonably be expected to react with chemical agents to produce toxic by-products, a qualitative assessment of decontamination by-products was performed. In addition, the corrosive and other potentially damaging effects of the cleaning technologies on the indoor materials were evaluated qualitatively by visual inspection.

The testing approach involved spiking excised samples (coupons) of four indoor building materials (galvanized metal ductwork, decorative laminate, wood flooring, and industrial grade carpet) with chemical agent followed by application of cleaning technology test solutions for one or two 30-minute contact periods. For Zep® industrial purple cleaner and K-O-K® bleach, tests were performed at two different strengths (i.e., degrees of dilution).

The results for the best performing cleaning technologies from the bench-scale testing are shown for the chemical agents (THD, TGD, VX, and HD) in Table ES-1. The results show a range of efficacies which are dependent on the chemical agent and the material onto which the chemical agent was applied.

Under the test conditions and with the materials tested, no cleaning technology was highly effective (>90% efficacy) in removing all chemical agents from all materials. Of the cleaning technologies tested, full-strength K-O-K® bleach generally had the highest efficacy against THD, TGD, VX, and HD. Toxic by-products, e.g., bis(beta-Chloroethyl) sulfone (Mustard sulfone; CAS 000471-03-4), were generated during the decontamination of HD with Zep® cleaner (full strength), K-O-K® bleach (10%), and K-O-K® bleach (full strength). EA 2192 (S-2-Diisopropylaminoethyl methylphosphonothioic acid) is another highly toxic by-product that, if present, would be a human-health hazard. This by-product was generated in the decontamination of VX with Zep® cleaner at full strength and K-O-K® bleach at 10%; no EA 2192 was measured in the decontamination of VX using K-O-K® bleach at full strength.

Little material damage was visually apparent from the use of the cleaning technologies. Zep® cleaner (full strength) caused visible discolorations on laminate coupons. No other surface damage was observed.

Table ES-1. Summary of Decontamination Efficacies against Chemical Agents

Chemical Agent	Cleaning Technologies (Concentration of Test Solutions) Cleaner Type	Galvanized Metal Ductwork	Decorative Laminate	Wood flooring	Industrial grade carpet
	30-Minute Contact Time				
THD	**K-O-K® Bleach (Full Strength)**	>99%	>99%	74%	82%
TGD	**OxiClean® Powder (0.06 g/mL)[a]**	21%	40%	44%	86%
VX	**K-O-K® Bleach (Full Strength)[b]**	97%	--	--	76%
HD	**K-O-K® Bleach (Full Strength)[c]**	>99%	>99%	76%	77%
	60-Minute Contact Time				
THD	**K-O-K® Bleach (Full Strength)**--	>99%	>99%	52%	60%
TGD	**K-O-K® Bleach (Full Strength)**	>98%	>98%	--	92%
VX	**K-O-K® Bleach (Full Strength)[b]**	>99%	>99%	58%	77%
HD	**K-O-K® Bleach (Full Strength)[c]**	>99%	>99%	66%	97%

-- Indicates that this combination of cleaner and material was not tested
a K-O-K® Bleach was not tested for this contact time
b Tested for EA 2192; none found (below the lower limit of quantitation).
c Tested for toxic or potentially toxic by-products;
Red highlight indicates that one or more toxic or potentially toxic by-products (toxicity data based upon non-representative exposure route and/or based upon animal studies) tentatively identified for this agent-material-cleaner combination.

Disclaimer

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Foreword

Following the events of September 11, 2001, EPA's mission was expanded to address critical needs related to homeland security. Presidential directives identify EPA as the primary federal agency responsible for the country's water supplies and for decontamination following a chemical, biological, and/or radiological (CBR) attack.

As part of this expanded mission, the National Homeland Security Research Center (NHSRC) was established to conduct research and deliver products that improve EPA's capability to carry out its homeland security responsibilities. One specific focus area of our research is on decontamination methods and technologies that can be used in the recovery efforts resulting from a CBR contamination event. In recovering from an event and decontaminating the area, it is critical to identify and implement appropriate decontamination technologies. The selection and optimal operation of an appropriate technology depends on many factors including the type of contaminant and associated building materials, temperature, relative humidity, decontaminant concentration, contact time, and others. This document provides information on how commercial cleaners performed in treatment of CWAs deposited on interior industrial building materials at various operational conditions.

These results, coupled with additional information in separate NHSRC publications (available at www.epa.gov/nhsrc) can be used to determine whether a particular decontamination technology can be effective in a given scenario. With these factors in consideration, the best technology or combination of technologies can be chosen that meets the clean up, cost and time goals for a particular decontamination scenario.

NHSRC has made this publication available to assist the response community to prepare for and recover from disasters involving chemical contamination. This research is intended to move EPA one-step closer to achieving its homeland security goals, and its overall mission of protecting human health and the environment, while providing sustainable solutions to environmental challenges.

Jonathan Herrmann, Director
National Homeland Security Research Center

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Abbreviations/Acronyms

BCa	bias-corrected and acceleration intervals
ºC	degrees Celsius
CAS	Chemical Abstract Service
CBR	chemical, biological, or radiological
cm	centimeters
CCV	Continuing calibration verification
DDTPP	decafluorotriphenylphosphine
DL	decorative laminate
EA 2192	S-2-diisopropylaminoethyl methylphosphonothioic acid
EPA	U.S. Environmental Protection Agency
ES	extraction solvent
g	grams
GC	gas chromatography
GD	G-Series nerve agent (soman), pinacolyl methyl phosphonofluoridate
GM	galvanized metal ductwork
HD	distilled mustard, bis (2-chloroethyl) sulfide
HPLC	High- performance liquid chromotographer
IC	industrial grade carpet
IS	internal standard
ISO	International Organization for Standardization
kHz	kilohertz
K-S	nonparametric Kolmogorov-Smirnov test
LC	liquid chromatography
LC_{Lo}	lowest lethal dose
LD_{50}	lethal dose, 50%
MDL	method detection limit
MS	mass spectrometry
µg	micrograms
µL	microliters
mg	milligrams
mL	milliliters
NHSRC	National Homeland Security Research Center
NIST	National Institute of Standards and Technology
PE	performance evaluation
Q	Quench
QA	quality assurance

QC	quality control
RH	relative humidity
RTECS®	Registry of Toxic Effects of Chemical Substances
SD	standard deviation
SOP	standard operating procedure(s)
SRC	surrogate recovery compound
TBP	tributyl phosphate
TGD	nerve agent GD, thickened with 5% poly(methylmethacrylate)
THD	distilled mustard (HD) thickened with 5% poly(methylmethacrylate)
VX	V-series nerve agent, O-ethyl-S-(2-diisopropylaminoethyl)methyl phosphonothiolate
WF	wood flooring

1.0 Introduction

In January of 2003, EPA established the National Homeland Security Research Center (NHSRC) to manage, coordinate, and support a wide variety of homeland security research and technical assistance efforts. One goal of EPA's homeland security research program has been to identify methods and equipment that can be used for decontamination following a terrorist attack using chemical, biological, or radiological (CBR) agents. Identification of such methods and equipment would be useful to emergency responders and decontamination decision-makers tasked with minimizing and mitigating environmental impacts after a CBR release. In a prior investigation, EPA (2009) evaluated the efficacy of chlorine dioxide fumigation, diluted household bleach (10% solution), and chlorine dioxide solution (~3000 ppm in aqueous solution) against sarin (GB), thickened soman (TGD), and V-series nerve agent (VX) on one or more materials. These studies showed that commercial cleaners, specifically bleach, are potential decontaminants for chemical agents.

NHSRC has found that the chemistries and/or available data suggest that common and readily available commercial cleaning products show promise for decontaminating chemical agents. This report describes an investigation of the efficacy of household and industrial cleaners for decontamination of indoor materials contaminated with chemical agents (specifically, thickened sulfur mustard [THD], TGD, VX, and sulfur mustard [HD]). The cleaning technologies evaluated were a household stain removal powder, an industrial cleaner and degreaser, household bleach, and an automatic dish detergent gel. The efficacy of the four decontamination technologies against each of the selected chemical agent agents was determined at two contact times on four common indoor surface materials. For cleaners that might reasonably be expected to react with chemical agents to produce toxic by-products, a qualitative assessment of decontamination by-products was performed. In addition, the effects of the cleaning technologies on the appearance of the building materials were evaluated qualitatively by visual inspection.

2.0 Investigation Approach

Bench-scale testing was utilized to evaluate the efficacy of cleaning technologies against surfaces contaminated with chemical agents specifically THD, TGD, VX, and HD listed. Approximately 4 mL of HD and GD was thickened by addition of poly(methyl methacrylate) (#182230, Sigma-Aldrich®, St. Louis, MO), 5% on a weight: volume basis to form THD and TGD. Viscosity of the resulting thickened agent was not measured. A team of NHSRC researchers and EPA program office and EPA region customers, who provide expertise on decontamination of sites contaminated with hazardous substances, provided input on the work described in this report. Variables such as contact time and household or industrial cleaner (cleaning technologies) were chosen by the team. The 30 and 60 minute contact times were recommended by the customers on the team because these times would require a minimum level of effort to keep the surfaces wetted. The project team selected cleaners that (1) had known chemistries (composition available on material safety data sheet), (2) would likely react with the chemical agents (based upon past Department of Defense stirred reactor or surface decontamination studies[(1)]), and (3) were broadly available and consistent in formulation across the United States.

Four types coupons (excised samples) of indoor building materials (galvanized metal ductwork, decorative laminate, wood flooring, and industrial grade carpet; described in detail in Section 2.1) were spiked with chemical agent followed by application of cleaning technologies for one or more contact times. The cleaning technologies (OxiClean® Versatile Stain Remover Powder, Zep® Industrial Purple Cleaner and Degreaser Concentrate, K-O-K® Household Bleach (sodium hypochlorite, 5.25%), and Cascade® with Extra Bleach Action Gel dishwashing detergent) were evaluated for the chemical agent and material combinations shown in Table 2-1. Note that not all combinations of chemical agent and cleaning technologies were tested due to initial testing results or expected reaction chemistries. A day of decontamination and subsequent extraction and analysis is referred to as a "trial." The trial number provides a reference between the test matrix and the results that follow. Missing trial numbers reflect trials that were anticipated, but were not performed based on adaptive management, i.e., based on results from previous trials, the subsequent trial conditions were changed (and assigned a different trial number).

Method demonstration was performed prior to beginning the decontamination evaluation. The method demonstration:

- Established that the extraction efficiencies for the recovery of chemical agents were sufficient for each chemical agent and material combination
- Determined how much of each cleaning technology would be delivered to a surface using a sprayer under a given set of representative operational conditions

- Established that a consistent application of the cleaning technologies onto test coupons mimic the amount delivered in field operations using a sprayer
- Evaluated methods for each cleaning technology to halt (quench) the decontamination process. (Note that loss of chemical agent due to processes not associated with the cleaning technology may continue after the cleaning technologies are quenched, e.g., water hydrolysis of GD.)

Table 2-1. Chemical Decontamination Test Matrix

Chemical Agent	Contact Time, Minutes	Cleaning Technology (Test Solution Concentration)[a]					
		OxiClean® Powder (0.06 g/mL)	Zep® Industrial Purple (25%)	Zep® Industrial Purple (Full Strength)	K-O-K® Bleach (10%)	K-O-K® Bleach (Full Strength)	Cascade® Gel (7.3%)
THD	30	Trial 13	Trial 15	--	--	Trial 17	--
THD	60	--	--	Trial 30	Trial 29	Trial 18	--
TGD	30	Trial 1	Trial 3	--	--	--	--
TGD	60	--	--	Trial 27	Trial 25	Trial 26	--
VX	30	Trial 21	Trial 23	Trial 34[b]	--	Trial 35[b]	--
VX	60	--	--	Trial 33	Trial 31	Trial 32	--
HD	30	Trial 5	Trial 7	--	Trial 9	Trial 19	Trial 11
HD	60	--	--	Trial 28	Trial 10	Trial 20	--

a The cleaners are mixed with distilled water. The concentrations represent the volume of product/total volume.

b The only materials included in this trial were galvanized metal ductwork and industrial grade carpet. In addition, a suspension test, mixing VX with the cleaning technology, was included.

The approach used for all decontamination tests is summarized in Figure 2-1. After application of the chemical agent to the test coupons, the spiked coupons were allowed to sit undisturbed for 30 minutes before decontamination was initiated. This waiting period was selected as a compromise between rapidly applying the cleaning technology test solution to minimize evaporative losses of agent and providing time for the chemical agent to soak into the coupon matrix to better gauge the efficacy for agent applied to the various coupon materials. Efficacy was determined to be the percent of mean mass of chemical agent recovered from the test coupons relative to the mean mass of chemical agent recovered from the positive control coupons.

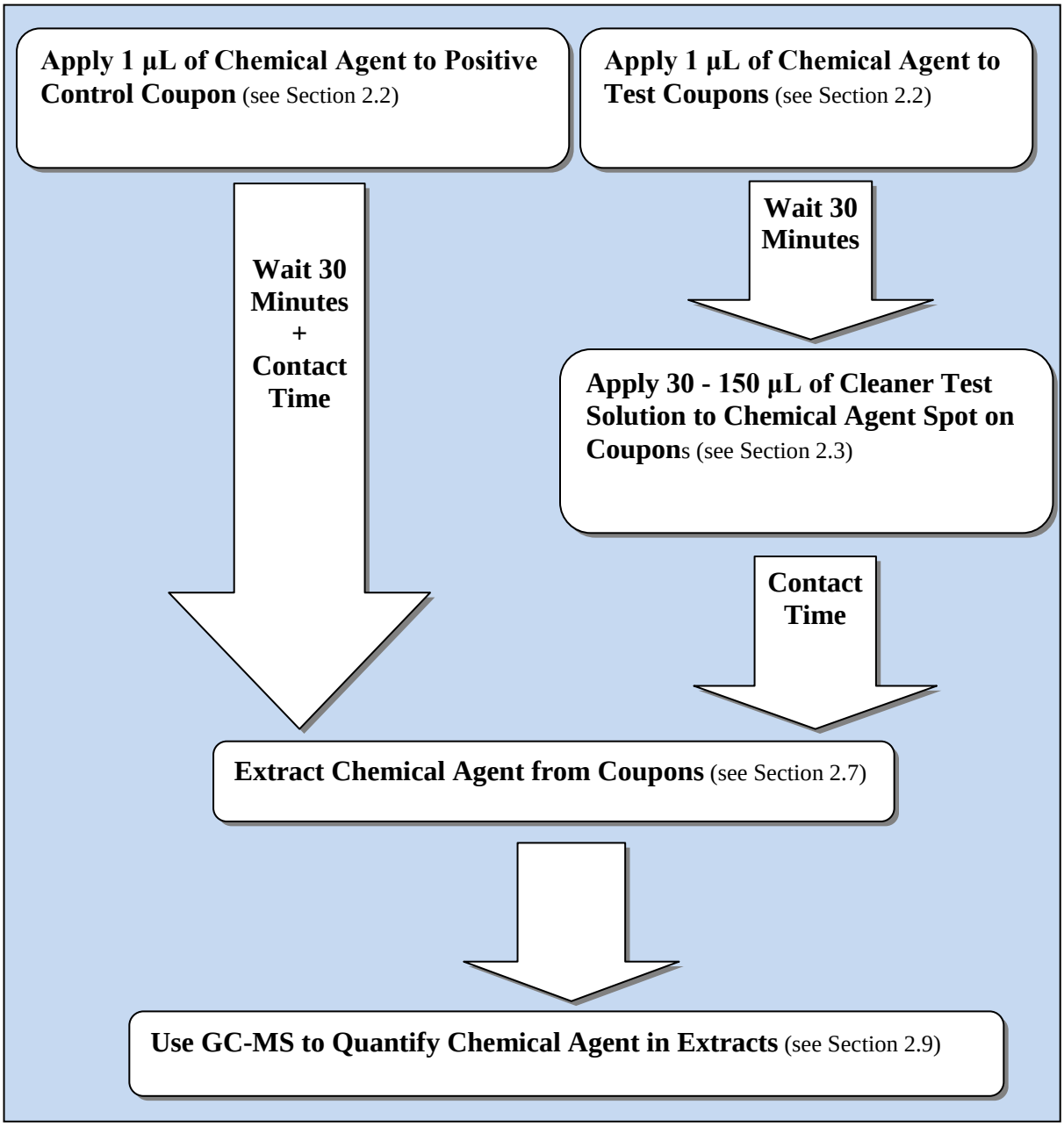

Figure 2-1. Summary of chemical agent decontamination testing.

All testing was conducted under ambient laboratory conditions, i.e., approximately 22 ^{o}C, 30% relative humidity (RH), and one atmosphere of pressure. The decontamination efficacy test matrix is presented in Table 2-1. For each chemical agent, cleaning technology, and material combination tested there were:

- Four laboratory blanks at each contact time that were not spiked with chemical agent and were not subjected to decontamination
- Four procedural blanks (two for each contact time) were not spiked with chemical agent but were subjected to decontamination

- Ten replicate test coupons (five for each contact time) were spiked with chemical agent and subsequently decontaminated
- Ten replicate positive controls (five for each contact time) were spiked with chemical agent but were not decontaminated

The materials were galvanized metal ductwork, decorative laminate, wood flooring, and industrial grade carpet.

The overall procedure for the evaluation of cleaning testing is outlined below.

1. Before any handling of the chemical agent, the laboratory blanks were spiked with a surrogate recovery compound (SRC), tributyl phosphate (TBP, CAS 127-73-8) (S2614-23-02, Fluka, St. Louis, MO), and then placed individually into a vial containing the specified extraction solution or quench solution (described in Section 2.7).
2. The positive control coupons were laid out in a fume hood. Each coupon was spiked with 1 μL of the designated chemical agent and allowed to sit undisturbed for 30 minutes plus the contact time for the corresponding decontamination test. When the appropriate time was reached (equivalent to the contact time for the associated test coupons), the positive control coupons were processed in the same manner as the test coupons, as described in Step 7.
3. Procedural blank coupons and test coupons were laid out horizontally in the fume hood. The coupons were placed into an appropriate open container. Each test coupon was spiked on the upper surface with the designated chemical agent. The test coupons were spiked in the same hood and at the same time as the positive control coupons.
4. Approximately 30 minutes after the chemical agent was spiked, a cleaning technology test solution was applied to the test coupons and to procedural blank coupons as described in Section 2.3.
5. The beginning of the contact time was documented when the cleaning technology test solution was applied to a coupon.
6. During the testing, all coupons remained at ambient room temperature (approximately 22 °C) and RH (approximately 30%) for the designated contact time. The contact time, temperature, and RH were monitored and documented. The temperature and RH were considered non-critical measurements and recorded at the beginning of each trial. Instruments used for these measurements were traceable to National Institute of Standards and Technology (NIST) or International Organization for Standardization (ISO) standards, but were not recalibrated. Coupons were covered loosely with a plastic lid (i.e., the lid did not touch the coupons) to prevent exposure to hood air flow.
7. After the appropriate contact time the test coupons and one of the corresponding procedural blank coupons of each material type were spiked with the SRC and immediately placed into an extraction bottle containing hexane and IS (internal standard). An additional quench was performed when needed (described in Section 2.7). The extraction bottles were subjected to sonication at 50 kHz for 10 minutes. An aliquot of the hexane layer was transferred to a vial and sealed.

8. This process was repeated in the same order that cleaning technology test solution was added to the coupons until the test coupons, positive control coupons, and procedural blank coupons were spiked with SRC, placed into individual vials containing extraction solution, and the vials capped, shaken, and sonicated.
9. All blank, test, and positive control coupons were individually extracted and the amount of chemical agent in the extraction solution was determined using GC/mass spectrometry (GC/MS). The GC/MS analysis generated a mass spectrum indicative of the chemicals present in the extract; a mass spectral library was used to tentatively identify compounds in the mass spectra.

The degradation of chemical warfare agents can, in some cases, produce toxic by-products. When water is present in excess for sufficient time, HD by-products are not a human health concern, i.e., the thioglycol product has low toxicity. However, at lower ratios of water to agent, there are known toxic intermediates, e.g., hemisulfur mustard (2-[2-chloroethylthio] ethanol). These known toxic partial-degradation products were analyzed in solution extracts using GC/MS. Peaks within quantifiable ranges were reported quantitatively. Peaks that were visible, but below the minimum quantifiable range, were reported as qualitatively present. GD hydrolyzes to form methyl phosphoric acid, a by-product with low toxicity. No known highly toxic by-products are formed in GD degradation. Certain highly toxic by-products from VX (e.g., EA 2192 shown in Figure 2-2) cannot be resolved using GC analytical methods. EA 2192 is a highly toxic by-product that, if present, would be a human-health hazard.

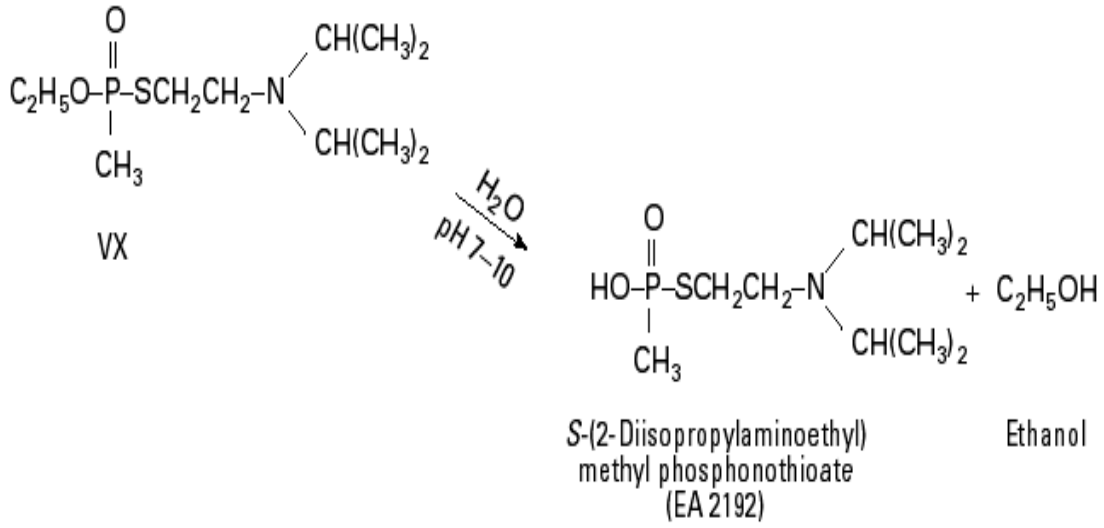

Figure 2-2. Breakdown of VX to form EA 2192. Modified from Munro et al., 1999.[2]

In cases where known toxic by-products could potentially be produced, GC (for HD) or liquid chromatography (LC) (for VX) coupled with qualitative MS analysis was used to evaluate whether such by-products arose from decontamination with various technologies. The qualitative by-product analysis was performed in parallel with the decontamination testing. The test matrix for by-product analysis is provided in Table 2-2.

GD decontamination is not known to produce toxic by-products that require qualitative analysis by either GC/MS or LC/MS. Therefore, by-product analysis was not performed for GD.

Prior testing had shown dilute bleach (10:1) to be "effective" against G agent and VX with 30 minutes contact times.[3] However, at 10% strength, production of EA 2192 may become a matter of concern because the expected pH of diluted bleach (11.5) is close to the pH range for optimum EA 2192 formation (pH 7-10). EPA had not evaluated whether diluted bleach converted VX to EA 2192 – hence evaluation of the decontamination of VX with diluted bleach at 30 minutes was initially included in this work. Decontamination with diluted bleach and analysis for EA 2192 in associated test samples showed that diluted bleach generated EA 2192. Using an adaptive management approach, the test matrix for VX was revised by EPA to subsequently use only full strength bleach. EA 2192 was not produced as a detectable byproduct with full strength bleach because full strength bleach has a high pH (~12.5).

Table 2-2. Test Matrix for Decontamination By-Product Analysis (number of coupon replicates per trial)

Agent	Description of Solutions Analyzed	OxiClean® Powder (full strength)	Zep® Industrial Purple (full strength)	K-O-K® Bleach (10%)	K-O-K® Bleach (full strength)	By-Product Analysis Technique
None	Quenched Cleaning Solution Blank	2	2	2	2	LC/MS
VX	Cleaner with Chemical Agent	2	2	2	2	LC/MS
None	Quenched Cleaning Solution Blank (Procedural Blank Coupon Extract)	2	2	2	2	GC/MS
HD	Cleaner with Chemical Agent (Test Coupon Extract[a])	2	2	2	2	GC/MS

Acronyms: GC, gas chromatography; LC, liquid chromatography; MS, mass sectrometry
a Coupon extracts were analyzed in full scan mode allowing by-product identification and agent quantitation to be done at the same time.

2.1 Test Materials

Information on the indoor materials and preparation procedures (if any) that were used for testing is presented in Table 2-3. The decontamination evaluation was conducted using coupons of the following types of materials, detailed in Figure 2-3: galvanized metal ductwork, decorative laminate, wood flooring, and industrial grade carpet. Coupons were cut to uniform length and width from a large piece of material. Edges and damaged areas were avoided in cutting test coupons. The test coupons were visually inspected prior to use; only coupons without surface anomalies were used.

Table 2-3. Test Materials

Material	Description	Supplier Name	Coupon Surface Area (thickness)	Material Preparation
Galvanized metal ductwork	Industry HVAC standard; 24 gauge galvanized steel	Adept Products, Inc., West Jefferson, OH	3.5 x 1.5 cm^2 (0.7 mm)	Clean with reagent-grade acetone
Decorative laminate	Pionite® laminate/white matte finish; grade 10	A' Jack Inc., Columbus, OH	3.5 x 1.5 cm^2 (1.2 mm)	None
Wood flooring	Fir plywood flooring (bare)	84 Lumber, Columbus, OH	3.5 x 1.5 cm^2 (0.9 cm)	Clean with dry air to remove loose dust
Industrial grade carpet	Shaw Industries Inc. style #M7832 color #00400	Carpet Corporation of America Rome GA	3.5 x 1.5 cm^2 (~0.7 cm)	None

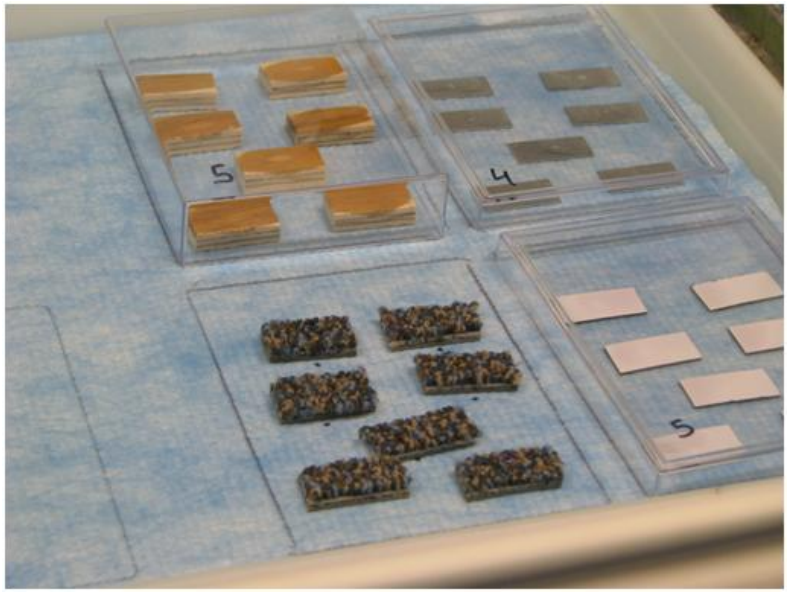

Figure 2-3. Galvanized metal ductwork (upper right), decorative laminate (lower right), wood flooring (upper left), and industrial grade carpet (lower right) with covers over coupons spiked with TGD.

2.2 Spiking Coupons

Shown in Figure 2-4, all test and positive control coupons were spiked using a syringe or pipette to deliver 1 µL of neat or thickened chemical agent (approximately 1.2 mg of THD, 0.9 mg of TGD or VX, and 1.3 mg of HD per microliter). This level of contamination is approximately 2 grams per square meter.

THD was dispensed using a Hamilton syringe (P/N 80565 [50 µL] equipped with 18-gauge needle [P/N 91018] and repeating dispenser [P/N 83700], Hamilton Co., Reno, NV). TGD was dispensed using a positive displacement pipette (P/N F148504 [5-10 µL] and C-10 [10 µL] tip, Rainin Instrument LLC, Oakland, CA). The pipette was set to dispense 1.4 µL to account for losses along the pipette wall and tip. VX, HD, and the SRC were dispensed using a Hamilton syringe (P/N 80565 [50 µL] equipped with 22-gauge needle [P/N 91022] and repeating dispenser [P/N 83700], Hamilton Co., Reno, NV).

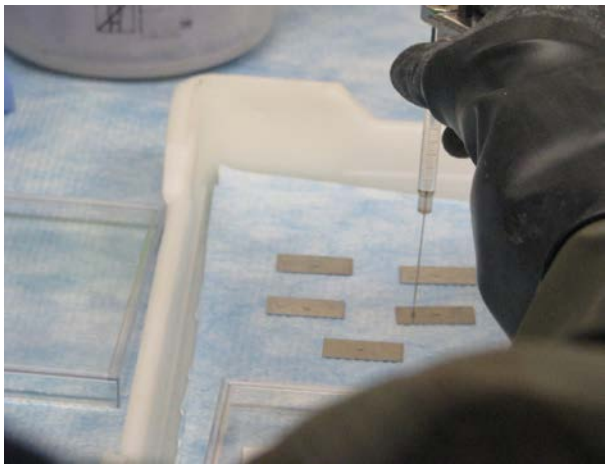

Figure 2-4. Spiking agent onto coupons.

Three polytetrafluoroethylene (Teflon®) spike control coupons (P/N 5Y43BYD, Thomas Scientific, Swedesboro, NJ) were evaluated in conjunction with each chemical agent trial. Polytetrafluoroethylene, a non-absorbent and non-reactive material, delivers nominally 100% recovery of chemical agents, and is used for spike control analysis. Each spike control coupon was contaminated with three 1 µL droplets of neat or thickened chemical agent, using the same pipette as the one used for contamination of the test and positive control coupons. The coupon was then immediately placed in 20 mL of chloroform (>99.9%, Fisher Scientific, Pittsburg, PA), shaken for 15 seconds, and extracted for one hour. The first spike control coupon was prepared at the beginning of the trial. The second spike control coupon was prepared midway though application of agent to test coupons and positive controls. The final spike control coupon was prepared after the last test coupon was contaminated.

2.3 Preparation and Application of Cleaning Technologies

The active ingredients for the cleaning technologies tested include:

- **OxiClean® Versatile Stain Remover Powder ("OxiClean powder"):** sodium percarbonate and sodium carbonate
- **Zep® Industrial Purple Cleaner and Degreaser Concentrate ("Zep purple industrial cleaner"):** sodium hydroxide and 2-butoxyethanol
- **K-O-K® Household Bleach 5.25% ("KOK bleach"):** sodium hypochlorite
- **Cascade® with Extra Bleach Action Gel ("Cascade gel"):** boric acid

The OxiClean powder forms hydrogen peroxide when dissolved in water. Household bleach (sodium hypochlorite 5.25%) is a commercially-available cleaner that is also a decontamination technology and is recommended by the U.S. Army to be used full-strength against blister agents, e.g., HD and VX.[4] Prior testing had shown dilute bleach (10:1) to be "effective" against G agent and VX with 30 minutes contact times.[3] However, EPA had not evaluated whether diluted bleach converted VX to EA 2192. The same 10:1dilution was selected by EPA to generate a complete set of data for the diluted bleach at 30 minutes. The inclusion of 10% bleach, and the analysis for EA 2192, showed that dilute bleach solutions generated EA 2192. Using an adaptive management approach, the test matrix for VX was revised by the task order project officer to subsequently use only full strength bleach. Due to its high pH (~12.5), full strength bleach does not generate EA 2192. With bleach at 10% strength, production of EA 2192 may occur because the expected pH is in the range at which EA 2192 is formed (pH 7 - 10).

Except when K-O-K bleach and Zep industrial purple cleaner were used full-strength, each cleaning technology was prepared as a mixture with distilled water. The maximum concentrations of each cleaning technology, recommended by the manufacturer and used in the testing, are provided in Table 2-4.

Table 2-4. Preparation and Concentrations of Cleaning Technology Test Solutions

Cleaning Technology	Manufacturer's Recommendation (product/water ratio)	Concentration, product mass/volume (g/mL) or volume / total volume ×100 % (target range)	Amount of Product to Use to Prepare 7571 mL Solution	
			Cleaner	Water
OxiClean Powder[a]	Add 1/4-scoop (28 g) to 473 mL warm or hot water	0.06 g/mL	488 g	7571 mL
Zep Industrial Purple Cleaner[b]	Dilute 946 mL to 121 L hot water	25% (24.5% - 25.5%)	1893 mL	5678 mL
Zep Industrial Purple Cleaner	Full strength	100%	7571 mL	0 mL
Cascade Gel[c]	See below for manufacturer's recommendation	7.3% (7.1% - 7.5%)	551 mL	7020 mL
K-O-K Bleach	Use 10% of full strength solution	10% (9.7%-10.3%)	757 mL	6814 mL
K-O-K Bleach	Full strength: sodium hypochlorite 5.25%	100%	7571 mL	0 mL
Cascade Gel[d]	Fill both dispenser cups completely	0.92%	70 mL	7501 mL

a Highest concentration is for pretreatment of stains on clothing and for spot removal from carpet.
b Highest concentration is for use on "tough soils". Use on alkaline-sensitive surfaces is not recommended.
c Selected concentration for evaluating use of this product for this evaluation.
d Manufacturer's instructions do not provide sufficient information to determine solution concentration.

The Cascade gel was used at higher concentration (7.3 v/v%) rather than the lower concentration (0.92 v/v%) that would be used in a dishwashing application. Full strength K-O-K household bleach (sodium hypochlorite 5.25% with a chlorine content of 5%-6%) and a 10% solution of the full strength K-O-K bleach in deionized water were used for decontamination testing. The sodium hypochlorite concentration was checked each day of use to ensure that the chlorine content was greater than 5% in the full strength solution. The material safety data sheet for the K-O-K household bleach (full strength) stated that the pH was 12.5 (not verified during testing). All of the cleaning technology manufacturers, except K-O-K bleach, recommend mixing the product with warm to hot water in order to dissolve the concentrate into solution. The cleaning technology test solutions, except K-O-K bleach, were prepared using deionized water heated to 40 °C-45 °C for dissolution and dilution to the designated concentration. Because the K-O-K bleach consists mostly of water, it was simply diluted with deionized water to the designated concentration.

The amounts of each cleaning test solution applied to each building material coupon, shown in Table 2-5, were based on the results of the spray application testing discussed in Section 4.1.2. The amount of cleaning test solution applied to each coupon corresponds to the mean mass that remained on the respective material as described in Section 2.5. By carefully applying this mass onto the coupons, there was no physical removal of the chemical agent by the cleaning test solutions. As shown in Figure 2-5, all cleaning

technology test solutions were dispensed using a positive displacement pipette (P/N M-100 [10-100 µL] and D-200 [2-200 µL] tip, Gilson Inc, Middleton, WS). The pipette was set to dispense 30, 60, or 90 µL depending on the cleaner that was used. The cleaning technology test solutions were dispensed twice on carpet samples, at settings of either 60 or 75 µL. The use of two applications, each of half of the required volume of the cleaning technology test solution was used on carpet in order to use a single pipette for all applications.

Table 2-5. Cleaning Technology Application Amounts

	Cleaning Test Solution Application (mL)	
Material	**Zep Industrial Purple Cleaner, Cascade Gel, or K-O-K Bleach**	**OxiClean Powder**
Galvanized metal ductwork	0.06	0.03
Decorative laminate	0.06	0.03
Wood flooring	0.09	0.09
Industrial grade carpet	0.12	0.15

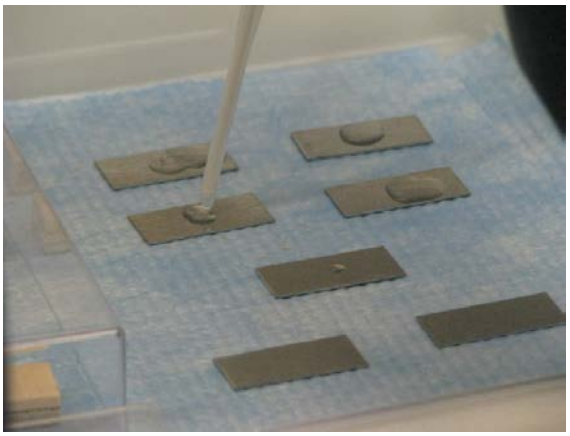

Figure 2-5. Applying cleaning test solution to test coupons spiked with chemical agent.

2.4 Method Demonstration – Recovery of Chemical Agent from Test Coupons

Method demonstration was conducted to establish sufficient extraction efficiencies (recoveries) for the chemical agents on wood flooring. For the other chemical agent – material combinations, previously determined MDLs[5] were used (see Table 2-6. MDL Values Previously Reported). Each of the wood flooring test materials was spiked with 10 µg of each unthickened agent (GD, VX, and HD) by placing a volume (e.g., 10 µL) of a dilute solution (e.g., 1,000 µg/mL) on the coupon surface. A decision was made, and documented in the test/QA plan, to use GD rather than TGD in the method demonstration. The thickener was expected to have a minimal impact on extraction efficiency, but would make handling and precise applications more difficult.

The SRC was also applied to the coupon surface. Sufficient hexane (with IS and neutralizer, when needed [Section 2.7]) to cover the coupons was used for extraction (e.g., 10 mL); the volume of hexane was constant for all extractions. The coupons were placed in the extraction solution within five minutes of spiking with dilute solution of agent.

Table 2-6. MDL Values Previously Reported

Material	MDL, µg (10 mL Extract)		
	GD	VX	HD
Galvanized metal ductwork	1.0	2.0	2.5
Decorative laminate	4.8	1.6	1.8
Industrial grade carpet	1.1	4.3	2.7

Source: U.S. EPA. 2010. Assessment of Fumigants for Decontamination of Surfaces Contaminated with Chemical Warfare Agents. U.S. Environmental Protection Agency. EPA/600/R-10/035.

Two extraction methods previously used by the EPA[5,6] were compared:

1. Overnight passive extraction
2. Active extraction

In passive extraction, coupons were extracted overnight (minimum 16 hours, maximum 24 hours). The coupons were placed into extraction bottles (P/N 89044-462, VWR, West Chester, PA) in contact with sufficient extraction solvent to cover the coupon. The vials were manually shaken at least three times throughout the workday, allowed to stand undisturbed overnight, and shaken again to assure complete mixing before removal of an aliquot for analysis.

In active extraction, the spiked samples were placed in 10 mL of extraction solution/quench solution specified in Section 2.7. Immediately after the coupon was placed into the vial with the extraction solution/quench solution, the vial was shaken by hand for 5-10 seconds, placed into the sonicator, and subjected to sonication at 50 kHz for 10 minutes. Within 30 minutes after the completion of sonication, an aliquot of extract was transferred with a pipette (P/N MR-1000 [500-1000 µL] and C 1000 [1000

µL] tip, Rainin, Oakland, CA) to a glass GC vial and closed with a cap (P/N HP-5181-880, VWR [Agilent Technologies], West Chester, PA).

GC/MS analysis was performed the same day or the following morning. The amount of spiked chemical agent was confirmed using control samples where dilute solution was spiked directly into hexane and analyzed. Eight replicates of each chemical agent on wood coupons were prepared, extracted and analyzed. The extraction efficiency was determined as a percent of the agent recovered from the spiked coupon relative to the amount spiked.

2.5 Method Demonstration – Spray Application

When cleaning technologies are used in field settings, they will be applied to contaminated surfaces using pressurized tank sprayers. In laboratory tests, these cleaning technologies were delivered to coupon surfaces as measured amounts from pipettes as described in Section 2.3. Prior to executing decontamination efficacy testing, target values for the appropriate amount of each cleaning technology were established and verified in controlled tests using a full-scale pressurized tank sprayer (Solo® Model 425 DLX, Solo, Newport News, VA). The sprayer was selected by EPA as representative of garden-type sprayers that would be commercially-available to decontamination response teams in local stores across the nation.

The amount of cleaning technology that carried over into extraction was determined so that the correct concentration of quench solution could be placed in the extraction solution. This amount was determined by:

- Weighing the coupon before application of the cleaning technology
- Applying the selected volume of cleaning technology to each type of coupon
- Waiting for the shortest contact time
- Weighing the coupon

The carryover was calculated as the difference in the mass of the coupon with residual cleaning technology after the shortest contact time less the mass of the coupon before application of the cleaning technology. These results are discussed in Section 4.1.2. Details of the approach follow.

Four 3.5 x 1.5 cm coupons of each test material were weighed on a calibrated balance (Mettler Toledo PG5002-SDR, Zürich, Switzerland). The coupons were placed on a horizontal surface and arranged side-by-side to form a row with the long sides next to each other and approximately 2-3 inches in between the coupons.

For each of the four diluted cleaning technologies, approximately 7.6 L of solution were prepared as described in Table 2-4. Except for K-O-K bleach, as noted above in Section 1.3, the cleaning technologies were diluted and mixed with deionized water (heated to 40 °C-45 °C). The solutions were allowed to cool in ambient laboratory conditions for at least one hour before using them in the sprayer.

The coupons were sprayed with a sweeping motion after establishing uniform flow of cleaner from the sprayer at 30 pounds per square inch (2.1 kilogram-force/square centimeter). The tip of the sprayer nozzle was held approximately 46-61 centimeters above the coupons and at an angle of 90 degrees to the substrate surface. Spraying by sweeping side-to-side continued until a continuous film covered the surface of the material. The rate of the sweeping motion was approximately 30 centimeters per second. The amount of time needed for spraying was recorded, along with the number of passes across the test surface. The test was repeated with three additional sets of coupons in order to characterize average results for sprayer performance with each cleaning technology.

After spraying was completed, the coupons were loosely covered for 1-5 minutes to hinder evaporation until the final weight of each coupon was determined. During the course of testing, each material type was tested in each matrix position, so relative position effects and time effects were averaged across the results. Each sprayed coupon was weighed on a calibrated balance to obtain its final weight. The mass of the cleaner applied to the coupon was determined by subtracting the initial coupon weight from the coupon weight post-spraying. For each material type, the average amount of each cleaner retained on a coupon was calculated.

The density of each cleaning test solution (based on the weight of known volumes of cleaning test solution) was used along with the average mass found for spraying the liquid on each of the materials to calculate the average volume of test solution applied to the coupons. The average volume for each cleaning technology was used to select the target amount to be applied by direct pipette onto coupon surfaces during the chemical agent testing. The designated amount was applied to coupons of each material type to verify coverage and ensure that the liquid cleaning technology remains on the coupon.

2.6 Method Demonstration – Termination of the Potential Decontamination Reaction

The decontamination reaction must be stopped at the end of a specified contact period in order to determine how much decontamination occurred during the contact period. The method demonstration described in this section determined the conditions necessary to stop (quench) the decontamination reaction. Rinsing of the coupons to remove the cleaning technologies or other post-decontamination steps directed by the manufacturer were not implemented in the method demonstration because such steps may not stop the decontamination reaction and do not allow for control of contact time.

In cases where extraction of the chemical agent with hexane from the cleaning solution proved sufficient to recover at least 50% of the chemical agent no additional quenching method was required. The sufficiency of hexane extraction alone was determined by mixing hexane containing 20 µg/mL of chemical agent with the cleaning solutions and sonicating for 10 minutes. The mixture was allowed to stand for 5 minutes for the polar and nonpolar layers to separate. A 1 mL aliquot was drawn from the hexane layer and

analyzed using GC/MS. If at least 50% of the chemical agent was recovered, no additive was necessary to quench the decontamination process.

In cases where hexane extraction alone was not sufficient to recover 50% of the chemical agent, the following approach was employed to evaluate potential additives to quench the reaction:

1. Prepare solutions containing 20 µg/mL of chemical agent in hexane.
2. Prepare neutralizer solutions, e.g., 0.2M sodium thiosulfate in deionized water.
3. Add 1mL neutralizer to 10 mL hexane containing chemical agent.
4. Sonicate solution for 10 minutes; let solution stand for 5 minutes to separate liquids.
5. Draw 1 mL aliquot from hexane layer; evaluate aliquots using GC/MS.

The recovery of chemical agent from quenched cleaning technologies was compared to the recovery of chemical agent from the hexane control (without mixing with cleaning technology) to determine the efficacy of the quenching procedure. The selected neutralizers are described in the following section.

2.7 Extraction of Chemical Agent and By-Products from Coupons for GC/MS Analysis

Immediately prior to extraction, every coupon was spiked with the SRC using a pipette (P/N MR-10, Rainin Instrument LLC, Oakland, CA) and transferred to an extraction bottle (P/N 89044-462, VWR International, West Chester, PA) containing 10 mL of hexane (GC Resolv grade, Fisher Scientific, Pittsburg, PA) and the IS (naphthalene-d_8), (with or without additive to quench the reaction, as appropriate). The extraction bottle was then sealed, shaken by hand for about 5-10 seconds, and placed into a sonicator. After all vials to be extracted at a given time point were placed in the sonicator (approximately 10 minutes later), they were sonicated at 50 kHz for 10 minutes. Within 30 minutes after the completion of sonication, an aliquot of the hexane layer was transferred to a GC vial (P/N HP-5181-880, VWR [Agilent Technologies], West Chester, PA) and sealed.

The extraction/quench solutions (Table 2-7) were prepared as follows:

- ES – Extraction Solvent (hexane/IS): hexane + 5µg/mL naphthalene-d_8
- Q1 – Quench Solution #1: deionized water + 0.2M sodium thiosulfate
- Q2 – Quench Solution #2: deionized water + potassium phosphate monobasic at super-saturation concentration

GC/MS evaluation of decontamination by-products was performed on samples from the HD-coupon extracts.

2.8 Test Solutions for VX Decontamination By-Product Analysis

Qualitative analysis for EA 2192 was performed using LC/MS. A reference sample of EA 2192 was run in parallel. The spectrum was used to identify EA 2192 but it was not quantified by comparison with the spectrum of a known mass of EA 2192. Test solutions for qualitative evaluation of VX decontamination by-products were prepared by spiking 1 µL of neat VX using a pipette (P/N MR-10 [5-10 µL] and C 10 [10 µL] tip, Rainin, Oakland, CA) into a 5 mL vial (P/N 60705-5, Fisher Scientific, Pittsburgh, PA), and then adding 50 µL of cleaner with a pipette (P/N MR-100 [50-100 µL] and C 50 [50 µL] tip, Rainin, Oakland, CA). The vial was capped and sonicated with a sonicator (5510R-DTH, Branson Ultrasonics Corp., Danbury, CT) for 1 minute, and allowed to react for 30 minutes. After 30 minutes, 1 mL of deionized water was added. The mixture was dispensed into two labeled sample vials (scintillation, 1 mL (P/N 60715-1, Fisher Scientific, Pittsburgh, PA) and analyzed for EA 2192 using LC/MS. The VX/decontamination solutions were pH-adjusted as necessary to prevent damage to the LC equipment. The procedure was repeated for the three cleaners.

Table 2-7. Matrix for Selected Extraction/Quench Solution

Agent	OxiClean Powder 0.06 g/mL	Zep Industrial Purple Cleaner 25%	Zep Industrial Purple Cleaner Full Strength	K-O-K Bleach 10%	K-O-K Bleach Full Strength	Cascade Gel
TGD	Sodium thiosulfate (Q1) in Hexane (ES)/ sonication	Potassium phosphate monobasic (Q2) in Hexane (ES)/ sonication	Potassium phosphate monobasic (Q2) in Hexane (ES)/ sonication	a	a	a
VX	No additional quenching agent: Hexane (ES)/ sonication only	No additional quenching agent: Hexane (ES)/ sonication only	No additional quenching agent: Hexane (ES)/ sonication only	a	a	a
THD, HD	No additional quenching agent: Hexane (ES)/ sonication only	No additional quenching agent: Hexane (ES)/ sonication only	No additional quenching agent: Hexane (ES)/ sonication only	Sodium thiosulfate (Q1) in Hexane (ES)/ sonication	Sodium thiosulfate (Q1) in Hexane (ES)/ sonication	No additional quenching agent: Hexane (ES)/ sonication only

Acronyms: Q, quench, ES, extraction solvent

a Not applicable; in some cases the extraction/quench method was already known from prior testing.

2.9 Analysis of Chemical Agent and By-Products in Extracts

The sample extracts were analyzed to quantify the amount of chemical agent remaining on each coupon. An Agilent (Santa Clara, CA) 6890 gas chromatograph and 5973 mass selective detector were used for the analysis of sample extracts. A single instrumental analysis method was used to analyze all sample extracts. The lowest standard used to establish the calibration curve was above, but near, the practical quantitation limit of the GC/MS. Samples with results below the lower calibration level were reported as less than the practical quantitation limit.

A practical quantitation limit is the analyte concentration range which produces quantifiable peaks with comparison to analytical standards. In these tests, a six-point calibration for GD, HD, VX and TBP was used with a lower calibration limit of 0.50 µg/mL and an upper range of approximately 50 µg/mL. Specifically, the six points included in the calibrations were 0.5, 1.0, 5.0, 10, 25, and 50 µg/mL. Due to saturation, only a five-point curve with an upper range of 25 µg/mL was used for some analytes (VX and TBP). Napthalene-d8 was used as an internal standard for quantitation GD, HD, VX and TBP concentrations. An average response (RSD <15%) or linear regression (r2 > 0.990) curve fit was applied to the calibration data. Any sample exceeding the upper calibration limit was diluted to an estimated concentration within the calibration range and reanalyzed. Samples with results at or below the lower calibration level were reported as "<0.5 µg/mL", the practical quantitation limit, and evaluated as "0.50 µg/mL" for statistical purposes.

The gas chromatograph/mass spectrometer was tuned initially and as needed following manufacturer's guidelines. A daily tune check was performed using decafluorotriphenylphosphine (DFTPP), a compound typically used for tuning during the analysis of semivolatile organics. Independently prepared continuing calibration verification (CCV) standards were analyzed prior to sample analysis, following every five samples and at the end of each batch of samples. Two or more CCV concentrations were used, one of which will be equal to the low calibration standard and the other(s) within the calibration range. CCV response was required to be within 25% of nominal concentration to be acceptable. Samples analyzed prior to, or following, CCVs that are outside of acceptance limits were re-analyzed. As stated in Section 2, a day of decontamination tests and subsequent extraction and analysis included construction of new calibration curves and extraction and analysis of three Teflon® coupon dosing standards for each agent.

A qualitative analysis for by-products of HD decontamination was performed. The gas chromatograph/mass spectrometer operated in the full scan mode was used to detect toxic by-products of HD in coupon extracts. The mass was not determined for by-products. A National Institute of Standards and Technology 2002 mass spectral library was used to tentatively identify compounds in the mass spectra. Reports were generated using ChemStation software (Agilent, Santa Clara, CA). Semi-quantitative results for each tentatively-identified compound were reported based on the IS response. A detailed interpretation of mass spectra data was not part of this testing.

LC/MS was used for semi-quantitative analysis of highly toxic EA 2192 in VX test solutions prepared as described in Section 2.8. The test solutions were analyzed for EA 2192 via LC/MS along with blanks of the cleaning solution.

The LC/MS-MS system consisted of an Agilent 1100/1200 liquid chromatography system (Santa Clara, CA) and an Applied Biosystems API 4000 mass spectrometer (Applied Biosystems, Foster City, CA) operated using positive electrospray. An Inertsil ODS-3 2.1 x 150 mm, 5 µm analytical column (Phenomenex, Torrance, CA) was employed for chromatographic separation. Detailed LC/MS/-MS parameters are shown in Table 2-8. A calibration curve of EA 2192 in deionized water was prepared at 1, 2, 5, 10, and 25 ng/mL. Data were acquired analyzing for ion transition 240>139 and 240>128. The response to EA 2192 was found to be quadratic over this range of concentration. Because the assay was not validated and was run without an internal standard, the results, while reported as masses of EA 2192 based on comparison to the calibration response curve, should be considered semi-qualitative and the masses understood to be relative to the calibration curve.

Table 2-8. Detailed Parameters for the LC/MS Analysis for EA 2192

HPLC[a]	Agilent 1100/1200		
Mass Spectrometer	Applied Biosystem API 4000		
Mass Spec Source	Electrospray, positive ion mode		
Mass Spec Software	Analyst 1.4		
HPLC Column	Inertsil ODS-3, 2.1 x 150 mm, 5 μm or equivalent		
HPLC Column Temperature	Ambient		
Mobile Phase Components	A = water: acetonitrile, 98:2 (v:v)		
	B = 0.2% formic acid in acetonitrile: isopropanol 80:20 (v:v)		
Gradient Profile	Time, min	%B	Flow rate, mL/min
	0	0	0.2
	1	0	0.2
	15	50	0.2
	18	50	0.2
	18.5	100	0.3
	30	100	0.3
	All changes are linear with respect to time.		
Injection Volume	50 μL		
Run Time	30 min		

Acronyms: HPLC, high-performance liquid chromotographer

2.10 Surface Damage

The effect of the cleaning technology on the appearance of test coupons was evaluated during the decontamination testing. Before and after decontamination of the test coupons, the appearance of the decontaminated coupons was visually inspected, and any obvious changes in the color, reflectivity, and apparent roughness of the coupon surfaces were recorded in the evaluation. In addition, photographs were taken before and after testing to document any changes that have occurred. Coupons subjected to the cleaning technology, but not contaminated with a chemical agent (i.e., procedural blanks), were inspected for surface damage after application of the cleaning technology.

2.11 Decontamination Calculations

Decontamination efficacy was determined by measuring the extracted amount of residual chemical agent on test coupons and comparing with the extracted amount remaining on the corresponding positive controls (spiked with chemical agent, not decontaminated and analyzed after the same "contact time" as the test coupons). Aliquots of extracts from laboratory blank, procedural blank, test, and positive control coupons were analyzed for chemical agents according to methods described in Section 2.10. Decontamination efficacy is calculated using a series of equations:

Chemical agent (or SRC) concentration in a coupon extract sample was determined by Equation 1:

$$\frac{A_s}{A_{is}} = M\frac{C_s}{C_{is}} + W \tag{1}$$

where:

A_s = Area of target analyte peak in sample
A_{is} = Area of internal standard peak
C_s = Concentration of target analyte in sample (μg/mL)
C_{is} = Concentration of internal standard (μg/mL)
M = slope of the gas chromatograph calibration line
W = Y intercept of the gas chromatograph calibration line.

GC concentration results (μg/mL) were converted to total mass by multiplying by extract volume:

$$M_m = C_s \times E_v \tag{2}$$

where:

M_m = Measured mass of chemical agent (μg)
C_s = Gas chromatograph determined concentration (μg/mL), see Equation (1)
E_v = Volume of extract (mL).

Decontamination efficacy was then defined as:

$$E = \left(1 - \frac{M_m \ of \ Chemical \ Agent \ on \ Test \ Coupon}{M_m \ of \ Chemical \ Agent \ on \ Control \ Coupon}\right) \times 100\% \tag{3}$$

where:

M_m = Measured mass of chemical agent (μg)
E = Decontamination efficacy or percent removal achieved during Decontamination (%).

Decontamination efficacy (mean ± standard deviation) was calculated for each type of test material spiked with each chemical agent. The mean and range of the efficacy values were reported for each material and chemical agent combination.

2.12 Statistical Comparisons

The objective of the statistical analysis was to estimate the median efficacy of each combination of chemical agent, cleaning technology, contact time, and material. Additionally, the median efficacy was to be evaluated to determine if the treatment could be concluded to be efficacious (i.e., median efficacy greater than zero). Finally, a series of comparisons was made to determine the difference in median efficacy between decontamination concentrations, contact times, and materials.

The measure of decontamination effectiveness was efficacy. Efficacy is expressed as a percentage of one and is derived here as: one minus the ratio of amount of recovered chemical agent after contamination with decontamination *to* the amount without decontamination. It requires the measurement of recoverable chemical agent both with and without decontamination. Given the destructive nature of the extraction measurement process for the material coupons, this quantity is not directly measurable for a single coupon. As a proxy for true efficacy, efficacy was defined and calculated as described in Section 2.11.

This definition provides twenty-five* possible estimated efficacy values, with the five possible test coupons each paired with the five possible control coupons. A reasonable estimate of central tendency for efficacy in this case is the median of the 25 possible efficacy values that could be generated from the five control and five test coupons.

In addition to the measure of central tendency, it was also desirable to generate a 95% confidence interval for the true median efficacy. This interval was one which would be expected to contain the true median efficacy of chemical removal from coupons after decontamination with 95% probability. For this evaluation, formation of the confidence intervals was a challenge for two reasons: (1) the small sample sizes, and (2) the form of the efficacy estimator (i.e., one minus a ratio of two random variables). In this case, a simple t-test or approach based on assumed normality can produce statistically inappropriate intervals whose true coverage probabilities are very different (possibly higher or lower) than the desired 95%. To address these issues, a bootstrap resampling approach was utilized with 95% bias-corrected and acceleration intervals (BCa). The BCa method, a result of the work of Efron and Tibshirani [6],was programmed in SAS® 9.1 based on guidance from Barker.[7]

The bootstrap method consisted of resampling, with replacement, 25 efficacy values from the original 25 possible values, and then determining the median of these 25 resampled efficacy values. This process was replicated until 20,000 separate median estimates were established. These 20,000 bootstrap median estimates were then ranked from smallest to largest and the BCa method determined the lower and upper percentiles of this distribution required to achieve a 95% confidence interval.

An important application of the confidence interval is that it provides a statistical basis to determine if the median efficacy is positive. If the lower endpoint of the 95% confidence

*20 in the case of Trial 7, agent HD, decontamination with ZEP for 30 minutes on carpet.

interval for the median efficacy is greater than zero, we can conclude, with at least 95% confidence, that the median efficacy exceeds zero. Note that an upper endpoint of the 95% confidence interval that is negative would also lead to a conclusion of statistical significance. However, this is a case of negative median efficacy.

In some instances, the recoverable agent on the test or control coupons at the conclusion of the testing was less than the 5 µg (0.5 µg/mL) lower practical quantitation limit of the extraction procedure. If only test coupons exhibited this result, the coupon recovery values were set to 5 µg and the median efficacy calculations were performed as defined above. In this case, the median efficacy is the most conservative possible value, and therefore the estimated median efficacy is reported as 'greater than' this value. Confidence intervals cannot be properly determined in this case. However, the identification of whether the recovery from the test coupons is systematically lower than that of the control coupons (i.e., the treatment was effective in decontamination) can be achieved through a nonparametric Kolmogorov-Smirnov (K-S) test. For this test, the five control coupon recoveries are one sample and the five test coupon with a substituted 5 µg recovery for each coupon below the practical quantitation limit are the comparison sample. A two-sided K-S test is performed to assess whether the recoveries are different for the test coupons than for the control coupons. If the K-S test p-value is less than or equal to 0.05, we conclude with at least 95% confidence then that the treatment is associated with different decontamination than the controls. Since the treatment group is the one with the lower recovery in all cases, this is equivalent to concluding that the treatment is efficacious.

In addition to estimating median efficacy for individual combinations of agent, cleaning technology, contact time, and material, it was also desired to compare median efficacies between these combinations. Specifically, the following comparisons were performed:

- Median efficacy between the 30 and 60 minute contact times for a particular agent, cleaning technology, and material
- Median efficacy of cleaning technology at full strength versus regular strength for a particular agent, contact time, and material
- Median efficacy between all pairs of materials for a particular agent, cleaning technology, and contact time

These comparisons were produced in all cases possible, noting that the test matrix did not include all possible conditions.

For the comparison of median efficacies, a similar approach was used to that detailed above. Where all recovery values were quantifiable, the estimated difference in median efficacy was found from the median of the 625 (25 possible efficacy values for the comparison condition times the 25 possible efficacy values for the reference condition) possible efficacy difference values. The same bootstrap resampling method with BCa was used to develop 95% confidence intervals, though the bootstrap samples were of size 625, rather than 25, and consisted of differences in efficacy rather than efficacy values

directly. When the resulting interval is entirely on one side of zero, it provides at least 95% confidence in the conclusion that the two median efficacy values are not identical.

When either of the two treatment groups (but not both) featured treatment coupons with recovery levels below the practical quantitation limit of quantitation, a nonparametric analysis was performed. The 625 possible efficacy differences are calculated with 5 µg substituted for values below the quantitation limit. The median of the 625 values is then reported as a conservative (i.e., ">x" or "<-x") value. The corresponding statistical comparison of the difference in efficacy requires an additional step. First, a K-S test was performed to assess if the recoveries for the five control coupons were different for the two groups of the comparison. If this two-sided test result was not significant, the recoveries of the two treatment groups were compared by a K-S test. If this test resulted in a p-value less than 0.05, it was concluded that the difference in median efficacy was statistically significant (in the same direction as the estimated difference in median efficacy).

For two conditions, neither the bootstrap resampling method nor the nonparametric K-S comparisons could be used. These conditions include:

- Both treatment groups in the comparison have at least one coupon with no measureable recovery.
- The preliminary K-S test on the control coupons for two separate treatment groups, as a prelude to the nonparametric comparison of the treated coupons in the two groups, showed a statistically significant result. In this case, the comparison of treated recovery values is not meaningful since the control recoveries to which they are referenced did not appear comparable for the two treatment groups.

In these conditions, the reported results are so noted and the magnitude of the difference in median efficacy as well as whether it is statistically significant are both indeterminate.

Note that the 95% confidence intervals and statistical tests identified as significant for p-values less than 0.05 are equivalent in terms of the conclusion in that they support the confidence level associated with that conclusion. In each case, the desirable property is that a conclusion of difference (e.g., median efficacy different from zero or difference in median efficacy between groups not zero) has no more than a 1 in 20 chance of being reached erroneously (i.e., where no such difference exists). However, this property only applies when a single interval or test is considered. When a very large number of comparisons is performed, as is the case in this evaluation, each with a 1 in 20 chance of erroneously concluding significance where none truly exists, the cumulative probability that no erroneous conclusion of significance has been reached actually becomes very small. It actually becomes likely for some cases that random chance has generated data that make treatments appear statistically significantly different when they are not. Statistical methods are available to adjust the observed results to take into account the potential for errors with multiple evaluations, but they come at the cost of reducing the sensitivity to observing significant results. Since the broad objective of this evaluation

was to examine many different cleaning technologies to decontaminate multiple chemical agents on a variety of indoor materials for various periods of time, the results can be considered more as a screening of efficacy rather than absolute estimates with given levels of statistical confidence. From this perspective, treatments appearing effective in this study could be further evaluated with targeted studies to better quantify efficacy or to compare between treatments. These studies should be designed to include multiple comparison adjustments to control the possible error rate for the entire evaluation. However, the corresponding loss of sensitivity for individual comparisons could be offset by designs that featured larger sample sizes and/or fewer test conditions.

3.0 Quality Assurance/Quality Control

Quality assurance (QA)/quality control (QC) procedures were performed in accordance with the existing quality management plan developed for NHSRC[8]. QA/QC procedures are summarized below.

3.1 Performance Evaluation Audits

Performance evaluation (PE) audits conducted in this investigation are summarized in Table 3-1. The working SRC solution was utilized in the PE audit of the gas chromatograph/mass spectrometer used to quantitate the chemical agent. The concentration of the working SRC solution was verified by analyzing a SRC standard obtained from a secondary source.

No PE audit was performed for the chemical agents as secondary standards were not available. The performance of the chronometer was also audited.

Table 3-1. Performance Evaluation Audits

Measurement	Audit Procedure	Allowable Tolerance	Actual Tolerance
Time	Compared to independent clock or watch value	2 seconds/hour	Done one time over 20 hours; 0.05 seconds/hour variance
Chemical Mass	Use GC/MS to analyze SRC from a secondary source and compare to primary source	± 10%	Done one time, < 5%

3.2 Technical Systems Audit

Battelle QA staff conducted a technical systems audit during February 17, 2010, to ensure that the investigation was being conducted in accordance with the test/QA plan[2] and associated amendments and the quality management plan.[8] As part of the audit test procedures were compared to those specified in the test/QA plan and data acquisition and handling procedures were reviewed. Observations and findings from the audit were documented and submitted to the Battelle task order leader for response. Minor QC issues were noted but did not have any impact on the quality of results. Audit records were permanently stored with the Battelle QA manager.

3.3 Data Quality Audit

At least 10% of the data acquired during the investigation were audited. A Battelle QA auditor traced the data from the initial acquisition, through reduction and statistical analysis, to final reporting to ensure the integrity of the reported results. All calculations performed on the data undergoing the audit were checked. Minor QC issues were noted and corrected by technical staff. These issues had no impact on the quality of results.

3.4 QA/QC Reporting

Each assessment and audit was documented in accordance with the test/QA plan[(2)] and QMP.[(8)] Results of the audit/data quality audit were submitted to EPA.

3.5 Other Data Quality Objectives

The data met the data quality objectives specified in the test/QA plan.

- Chemical agent, measured in the hexane blanks run with each batch of samples, was below the lower limit of the calibration curve (approximately the practical quantitation limit) of 0.5 µg/mL.
- Chemical agent, measured in the extracts of laboratory blanks run with each batch of samples, was below the lower limit of the calibration curve (approximately the practical quantitation limit) of 0.5 µg/mL.
- Chemical agent, measured in the extracts of procedural blanks run with each batch of samples, was <10% of the amount recovered from the corresponding positive control coupons.
- At least 50% of the surrogate recovery compound was found in the analysis of extracts of positive controls.
- At least 50% of each chemical agent was demonstrated to be recovered from each coupon type.

3.6 Deviations

The test/QA plan specified that the mean efficacy would be reported. The median efficacy was included in this report instead of the mean. Reporting the central tendency as the median rather than the mean reduces the bias that can arise from a single particularly high or low value when the sample size is small.

4.0 Test Results

4.1 Method Demonstration Results

4.1.1 Recovery of Chemical Agent from Test Coupons

Method demonstration was conducted to establish sufficient extraction efficiencies (recoveries > 50%) for the chemical agents and wood flooring. Recovery results are shown in Table 4-1. VX recovery from wood flooring was 63% with sonication but only 12% with passive extraction for 24 hours. Sonication provided recoveries >50% for all the chemical agents tested, meeting the requirement from the test/QA plan for the extraction method to be acceptable. Consequently, sonication was selected for use in subsequent decontamination testing.

Table 4-1. Mean Measured Recovery of Chemical Agents from Wood Flooring

Agent	Wood Flooring	
	Sonication, % Recovery	**Passive, % Recovery**
GD	61%	68%
VX	63%	12%
HD	61%	62%

Note: Each result is mean recovery from eight coupons.

4.1.2 Spray Application

Shown in Table 4-2, the spray demonstration was used to determine the average mass of each cleaning technology that remained on each type of material under typical spray application of the technologies. Because the measured densities of the four cleaning technologies were all 1.0 g/mL, the residual volume was numerically the same as the mass. The residual volume of each cleaning technology that remained on the various materials was selected as the target amount to be applied onto coupon surfaces during the chemical agent testing. Based on the results of the spray application testing, volumes of cleaning technologies to be applied to building material coupons were selected, as shown in Table 4-3. For a given material, the same volume of Zep industrial purple cleaner, K-O-K bleach, and Cascade gel was used. The volumes of OxiClean powder were different from those of the other cleaning technologies for industrial grade carpet (where slightly higher volumes were used) and for galvanized metal ductwork and decorative laminate (where lower volumes were used).

Table 4-2. Residual Decontamination Technology (Mean Measured Mass and SD) Remaining on Coupons after Spray Application

Material	OxiClean Powder 0.06 g/mL, g (SD)	Zep Industrial Purple Cleaner 25%, g (SD)	K-O-K Bleach (10%), g (SD)	Cascade Gel (7.3%), g (SD)
Galvanized metal ductwork	0.031 (0.011)	0.054 (0.013)	0.072 (0.008)	0.066 (0.015)
Decorative laminate	0.034 (0.010)	0.063 (0.011)	0.072 (0.012)	0.064 (0.017)
Wood flooring	0.102 (0.019)	0.097 (0.021)	0.087 (0.017)	0.086 (0.021)
Industrial-grade carpet	0.161 (0.032)	0.124 (0.023)	0.130 (0.025)	0.109 (0.021)

Note: Each result is mean (and standard deviation) of mass remaining on 12 coupons for each material type and cleaning technology. The 12 coupons comprised four coupons from each of three independent sprayings with a given cleaning technology.

Table 4-3. Volume of Cleaning Technology Selected for Application onto Test Coupons

Material	Cleaner Application (mL)	
	Zep Cleaner[a] 25% and Full Strength/ K-O-K Bleach 10% and Full Strength / Cascade Gel 7.3%	OxiClean Powder 0.06 g/mL%
Galvanized metal ductwork	0.06	0.03
Decorative laminate	0.06	0.03
Wood flooring	0.09	0.09
Industrial grade carpet	0.12	0.15

[a] Zep industrial purple cleaner

4.1.3 Termination of the Potential Decontamination Reaction

Method demonstration was used to determine whether hexane extraction alone was sufficient to terminate the chemical decontamination process and, if not, to identify additives to the extraction solution that would terminate (quench) the chemical decontamination process. Results from the method demonstration are provided in Table 4-4. Hexane extraction alone was determined to be sufficient quench for OxiClean and Zep industrial purple cleaner used to decontaminate VX, THD, and HD; hexane alone was also sufficient for Cascade gel used to decontaminate THD and HD. The addition of sodium thiosulfate to hexane was sufficient to quench K-O-K bleach (10%) when used to decontaminate THD and HD. The addition of sodium thiosulfate to hexane was sufficient to quench OxiClean powder when used to decontaminate TGD. The addition of potassium phosphate monobasic to hexane was sufficient to quench the Zep cleaner

when used to decontaminate TGD. These results were the basis for the selection of the quench methods described in Section 2.7.

The extraction solution used for each combination of chemical agent and cleaning technology is shown in Table 4-4. The extraction solutions were prepared as described in Section 2.7.

Table 4-4. Method Demonstration Results for Termination of the Decontamination Reaction

Agent	OxiClean Powder, 0.06 g/mL	Zep Industrial Purple Cleaner, 25%	Zep Industrial Purple Cleaner, full strength	K-O-K Bleach, 10%	K-O-K Bleach, full strength	Cascade Gel, 7.3%
TGD	Sodium thiosulfate (Q1) in hexane (ES) (73% recovery with thiosulfate quench)	Potassium phosphate monobasic (Q2) in hexane (ES) (100%[a] recovery with potassium phosphate quench)	Potassium phosphate monobasic (Q2) in hexane (ES) (100%[a] recovery with potassium phosphate quench)	--	--	--
VX	No additional quenching agent: Hexane (ES)/ sonication only (88% - 99% recovery with hexane extraction)	No additional quenching agent: Hexane (ES)/ sonication only (100%[a] recovery with hexane extraction)	No additional quenching agent: Hexane (ES)/ sonication only (100%[a] recovery with hexane extraction)	--	--	--
THD, HD	No additional quenching agent: Hexane (ES)/ sonication only (93% - [a] recovery with hexane extraction)	No additional quenching agent: Hexane (ES)/ sonication only (100% [a] recovery with hexane extraction)	No additional quenching agent: Hexane (ES)/ sonication only (100% [a] recovery with hexane extraction)	Sodium thiosulfate (Q1) in hexane (98% recovery with thiosulfate quench)	Sodium thiosulfate (Q1) in hexane (98% recovery with thiosulfate quench)	No additional quenching agent: Hexane (ES)/ sonication only (84% recovery with hexane extraction)

Symbols: -- = not applicable

a The recoveries from one or more of the replicate tests were greater than the mean recoveries from the positive control solution.

4.2 Decontamination Results

The results for the decontamination testing are reported as tables of efficacy results supplemented with figures showing the mean and SD of the measured mass of chemical agent recovered from each type of material. Note that the chemical agent, e.g., HD, not THD, is extracted and measured from coupons onto which the thickened agent, e.g., THD, was applied.

The results for the spike control coupons that were evaluated in conjunction with each chemical agent trial are shown in the Appendix (Table A- 21). The spike control results showed that application and recoveries were relatively consistent except that a TGD exhibited greater variability, partially shown in the coefficients of variance for within the trials in Table A-1, both within and between trials, than other chemical agents. This implies that greater variance in the TGD mass recoveries during the decontamination and positive control tests is likely due to the variability in application of the agent. The differences in variance affects interpretation of the data in that larger standard deviations will be observed in the calculated efficacies which may in turn inhibit comparisons across cleaners, materials and contact time.

4.2.1 THD Decontamination

Five decontamination technology preparations (three cleaning technologies) were used against THD on four materials (galvanized metal ductwork, decorative laminate, wood flooring, and industrial grade carpet):

- OxiClean powder (0.06 g/mL) with 30-minute contact time
- Zep industrial purple cleaner (25%) with 30-minute contact time
- Zep industrial purple cleaner (full strength) with 60-minute contact time
- K-O-K bleach (10%) with 60-minute contact time
- K-O-K bleach (full strength) with 30- and 60-minute contact times.

Figure 4-1 shows the mass recoveries for the test and positive control coupons for the OxiClean powder (0.06 g/mL) against THD on the materials. The low recoveries of HD from wood flooring positive control coupons were observed both with thickened agent (THD) and with neat agent (HD) (see Section 4.2.4).

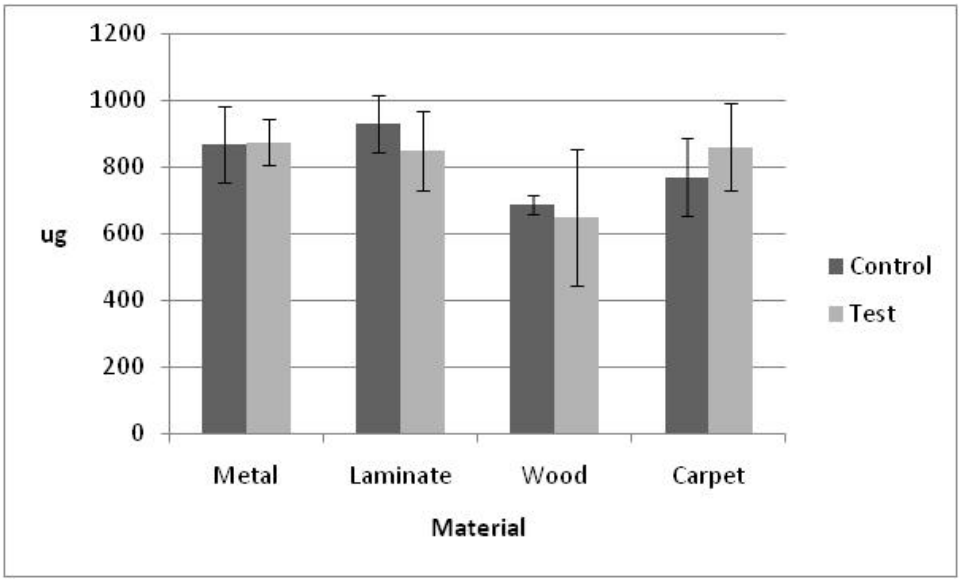

Figure 4-1. Mean measured mass and SD of HD (applied as THD) recovered from building materials after exposure to OxiClean (0.06 g/mL) for 30 minutes (Trial 13).

Figures 4-2 and 4-3 show results of Zep industrial purple cleaner (25%) at a 30-minute contact time and the Zep cleaner (full strength) at a 60-minute contact time, respectively, against THD. The substantial decrease in recoverable HD from laminate after the 60-minute contact time is an unexplained anomaly and is not consistent with the results observed when Zep cleaner (full strength) was applied to HD (without thickener) for 60 minutes. (See Section 4.2.4.)

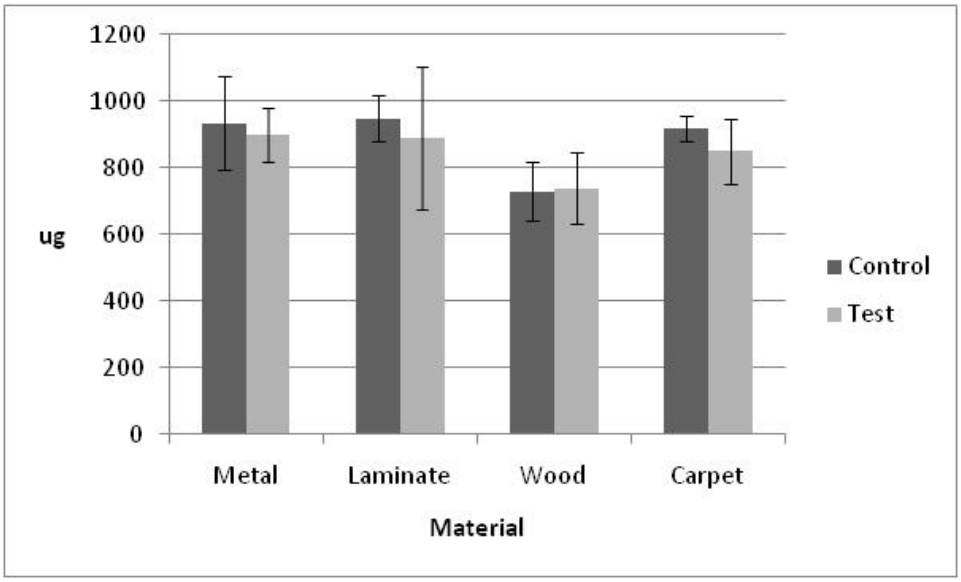

Figure 4-2. Mean measured mass and SD of HD (applied as THD) recovered from building materials after exposure to Zep industrial purple cleaner (25%) for 30 minutes (Trial 15).

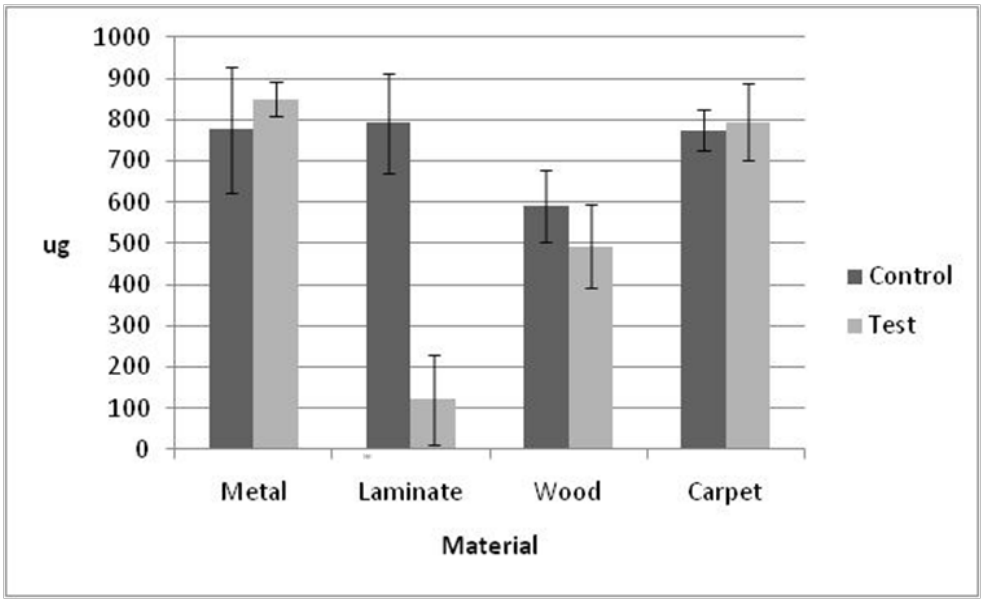

Figure 4-3. Mean measured mass and SD of HD (applied as THD) recovered from building materials after exposure to Zep industrial purple cleaner (full strength) for 60 minutes (Trial 30).

Figure 4-4, shows the mass of HD recovered from the test and positive control coupons for the 60 minute contact time when K-O-K bleach (10%) was used against THD on the materials. The metal and laminate coupons had the largest discernable differences in recoveries between the test and positive control coupons.

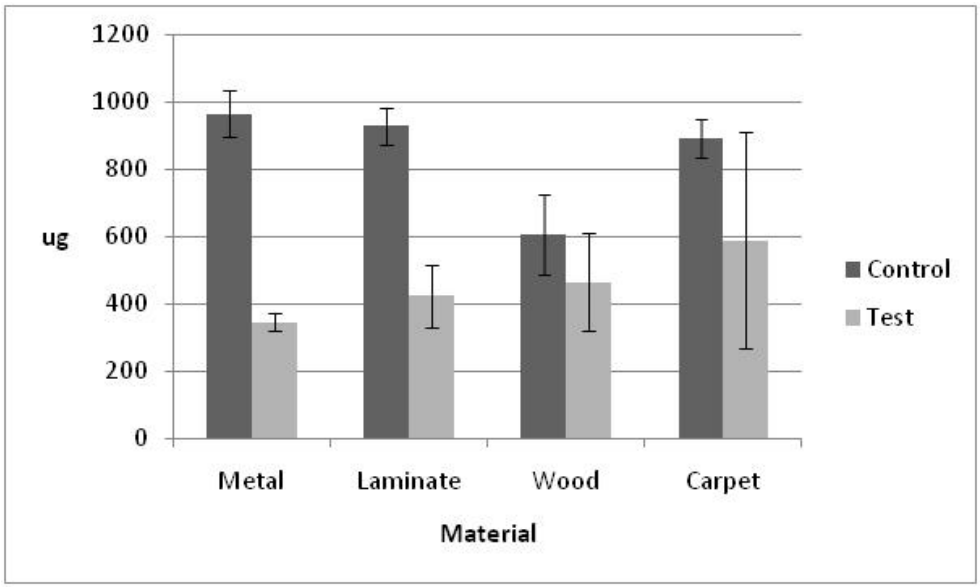

Figure 4-4. Mean measured mass and SD of HD (applied as THD) recovered from building materials after exposure to K-O-K bleach (10%) for 60 minutes (Trial 29).

Figures 4-5 and 4-6 show results of K-O-K bleach tests at the 30- and 60-minute contact times, respectively. As shown in Figures 4-5 and 4-6, increasing the contact time did not reduce the amount of HD recovered from the test coupons after the treatment.

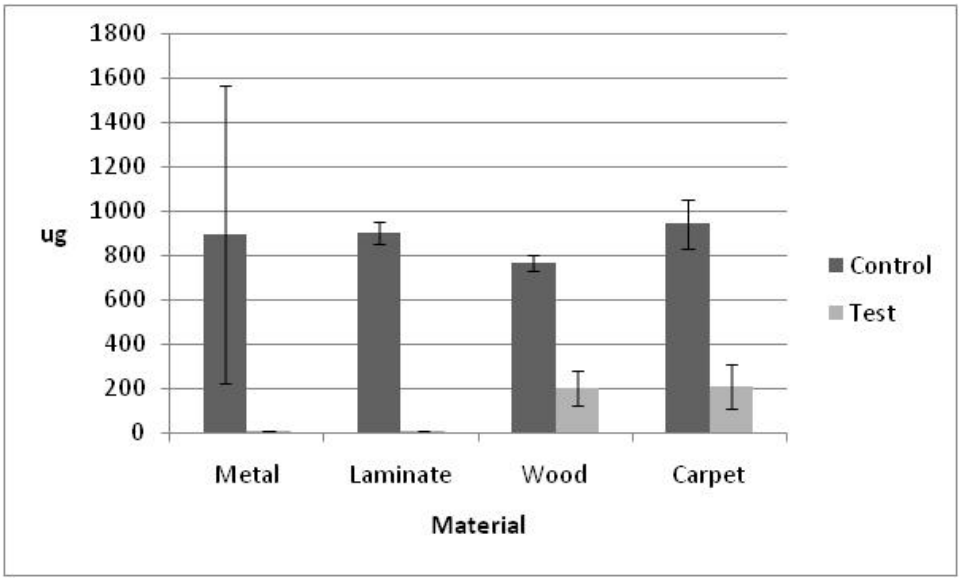

Figure 4-5. Mean measured mass and SD of HD (applied as THD) recovered from building materials after exposure to K-O-K bleach (full strength) for 30 minutes (Trial 17).

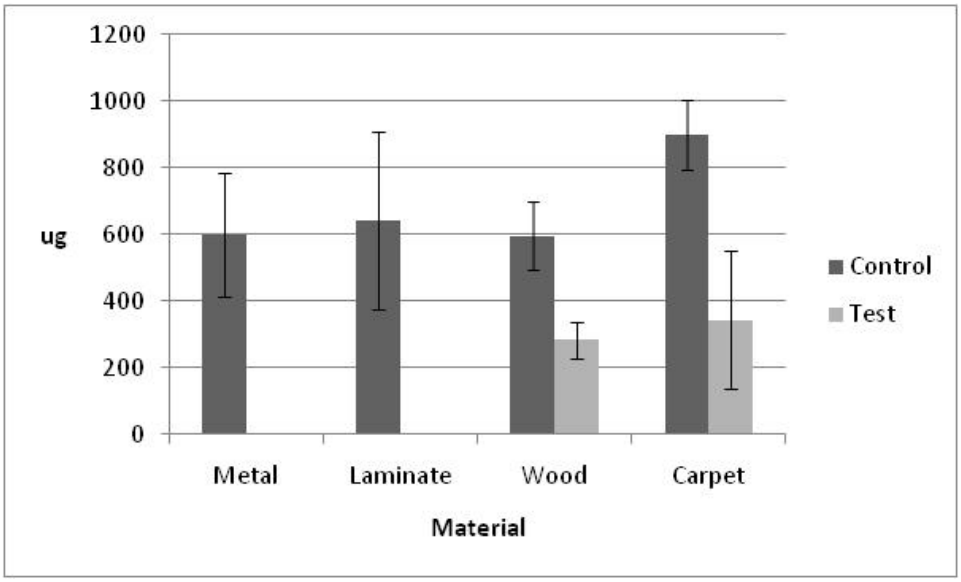

Figure 4-6. Mean measured mass and SD of HD (applied as THD) recovered from building materials after exposure to K-O-K bleach (full strength) for 60 minutes (Trial 18).

Table 4-5 summarizes the median efficacies and corresponding 95% confidence intervals. Results in which the differences were not significant are so noted in Table 4-5.

OxiClean powder (0.06 g/mL) and Zep industrial purple cleaner (25%) showed no efficacy with a 30-minute contact time. Efficacies (85%) were exhibited by the Zep cleaner (full strength) on decorative laminate with a 60-minute contact time. K-O-K bleach (full strength) exhibited the highest efficacy (74% - >99% at 30-minute contact time). Increasing the K-O-K bleach contact time to 60 minutes did not appear to increase efficacy.

Note that in some cases, both with THD and with other chemical agents, the recoveries of chemical agent from individual test coupons or the mean recovery from test coupons is greater that the mean recovery from positive control coupons. Such cases are noted in the summary tables and are attributable to the imprecision of the measurements.

Table 4-5. Median THD Decontamination Efficacy Results (95% confidence interval)[a] or Efficacy (Number of Test Coupons below the Practical Quantitation Limit)[b]

Contact Time, Minutes	Material	Cleaning Technologies (Concentration)				
		OxiClean Powder (0.06 g/mL)	Zep Industrial Purple Cleaner (25%)	Zep Industrial Purple Cleaner (Full Strength)	K-O-K Bleach (10%)	K-O-K Bleach (Full Strength)
		Trial 13	Trial 15	--	--	Trial 17
30	Galvanized metal ductwork	NS	NS	--	--	>99% (5/5)
30	Decorative laminate	NS	NS	--	--	>99% (5/5)
30	Wood flooring	NS	NS	--	--	74% (71% - 81%)
30	Industrial grade carpet	0%[c] (I)	NS	--	--	82% (79% - 83%)
				Trial 30	Trial 29	Trial 18
60	Galvanized metal ductwork	--	--	0% [c] (I)	65% (62% - 66%)	>99% (5/5)
60	Decorative laminate	--	--	82% [d] (1/5)	52% (47% - 62%)	>99% (5/5)
60	Wood flooring	--	--	NS	21% (11% - 35%)	52% (47% - 58%)
60	Industrial grade carpet	--	--	0%[c] (I)	49% (10% - 56%)	60% (43% - 75%)

Symbols and acronyms: -- is not tested, NS indicates that efficacy is not significantly different from 0 at the 95% confidence level. I indicates that recoveries from one or more of decontaminated test coupons were greater than mean recoveries from positive control coupons at test condition.

a Estimated efficacy is median of efficacy (defined as one minus recovery of treated coupon divided by recovery of control coupon) from all possible combinations, expressed as percentage. Confidence intervals are 95% bias-corrected and acceleration intervals of simulation using bootstrap sampling approach.

b One or more of test coupons had no recovered agent (the exact number is shown in parentheses). The reported "$>x$" value is estimated efficacy with five CFU substituted for all zero recovery coupon values. For these trials, test of statistical significance is a non-parametric Kolmogorov-Smirnov test where a p-value less than 0.05 indicates a statistically significantly difference between test and positive control coupons.
c Mean recovery from decontaminated test coupons was greater than mean recovery from positive control coupons at test condition.
d Substantial decrease in recoverable HD from laminate after 60-minute contact time is not consistent with the Zep cleaner (full strength) results for other test materials.

4.2.2 TGD Decontamination

Five decontamination technology preparations (three cleaning technologies) were used against TGD on four materials (galvanized metal ductwork, decorative laminate, wood flooring, and industrial grade carpet):

- OxiClean powder (0.06 g/mL) with a 30-minute contact time
- Zep industrial purple cleaner (25%) with a 30-minute contact time
- Zep industrial purple cleaner (full strength) with a 60-minute contact time
- K-O-K bleach (10%) with a 60-minute contact time
- K-O-K bleach (full strength) with a 60-minute contact time.

The mass of GD (applied as TGD) recovered from test and control coupons after 30 minute contact with OxiClean powder (0.06 g/mL) is shown in Figure 4-7. The mean mass of GD recovered from the carpet on the positive controls was very high. Thickened agents are difficult to dispense accurately and the thickened agents are not uniform. Carpet is particularly difficult to apply a specific volume of this viscous material.

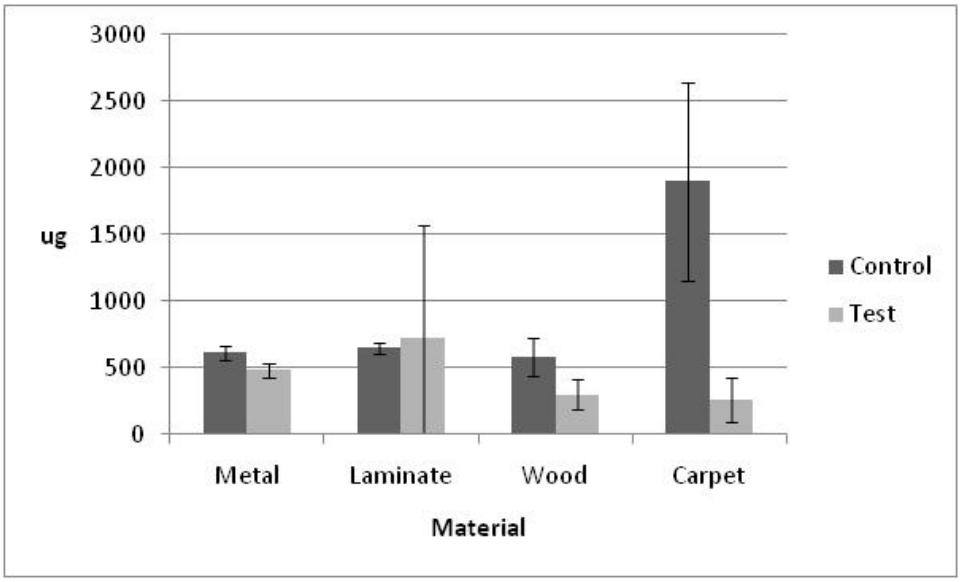

Figure 4-7. Mean measured mass and SD of GD (applied as TGD) recovered from building materials after exposure to OxiClean powder (0.06 g/mL) for 30 minutes (Trial 1).

Figures 4-8 and 4-9 show results of Zep industrial purple cleaner (25%) at a 30-minute contact time and the Zep cleaner (full strength) at a 60-minute contact time, respectively. Large differences in the recovered mass of GD on all materials tested were observed for Zep cleaner (full strength) after the 60-minute contact time.

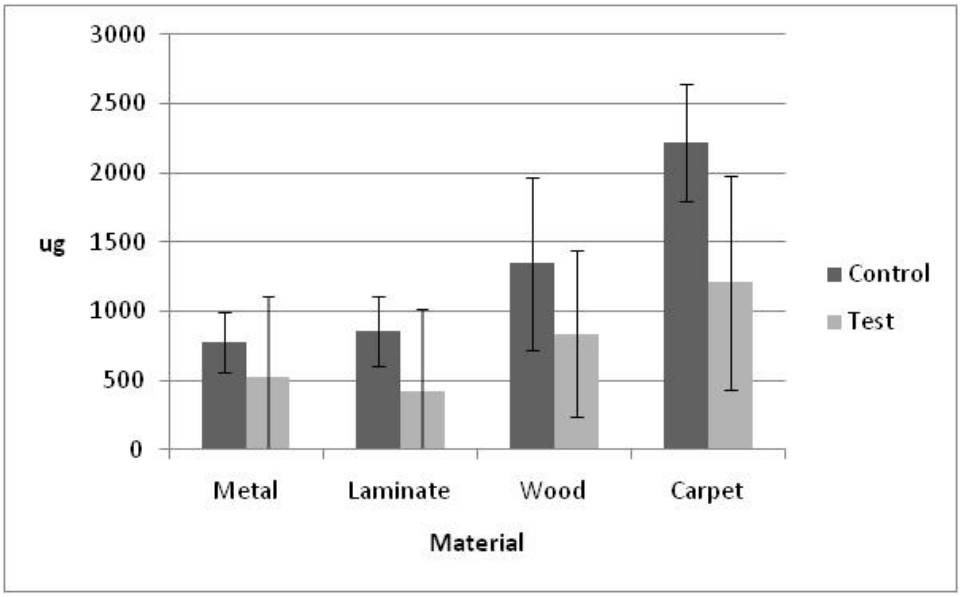

Figure 4-8. Mean measured mass SD of GD (applied as TGD) recovered from building materials after exposure to Zep industrial purple cleaner (25%) for 30 minutes (Trial 3).

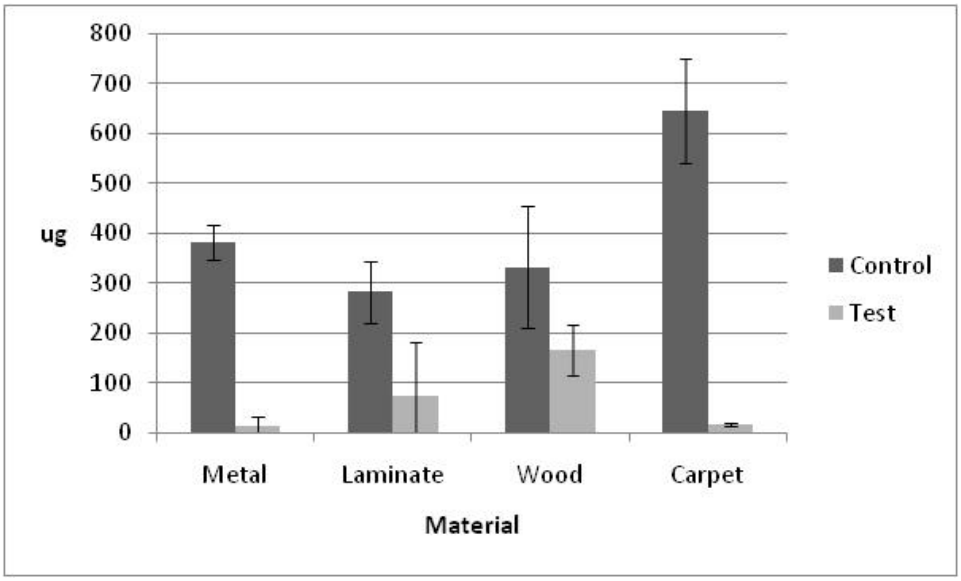

Figure 4-9. Mean measured mass SD of GD (applied as TGD) recovered from building materials after exposure to Zep industrial purple cleaner (full strength) for 60 minutes (Trial 27).

As shown in Figure 4-10 and 4-11, less mass of GD was recovered from the coupons at the 60-minute contact time for the K-O-K bleach (full strength) versus K-O-K bleach (10%) against TGD for all the materials. In the tests for both bleach strengths there were also discernible differences in recovered mass of GD from the test coupons compared to the control coupons for the metal, laminate and carpet materials.

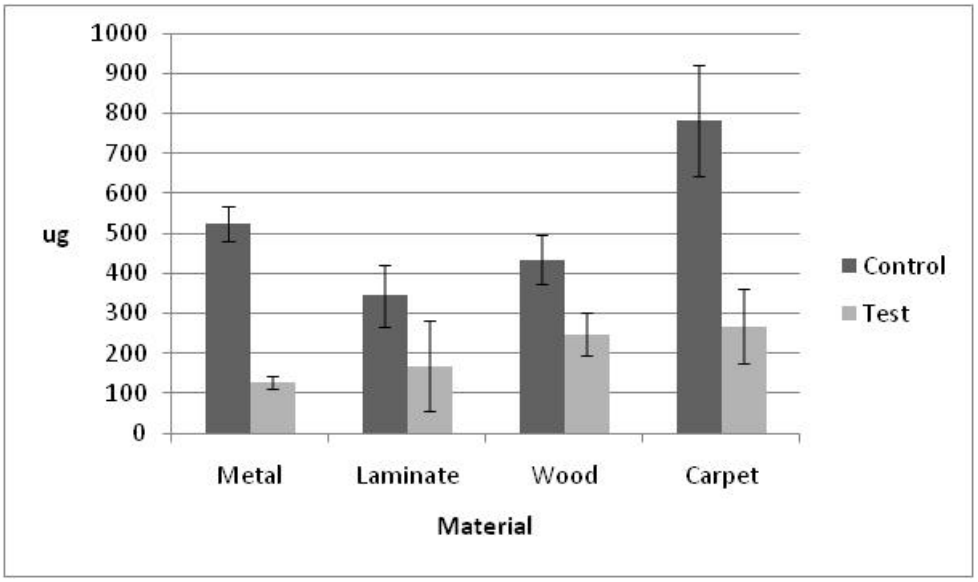

Figure 4-10. Mean measured mass and SD of GD (applied as TGD) recovered from building materials after exposure to K-O-K bleach (10%) for 60-minutes (Trial 25).

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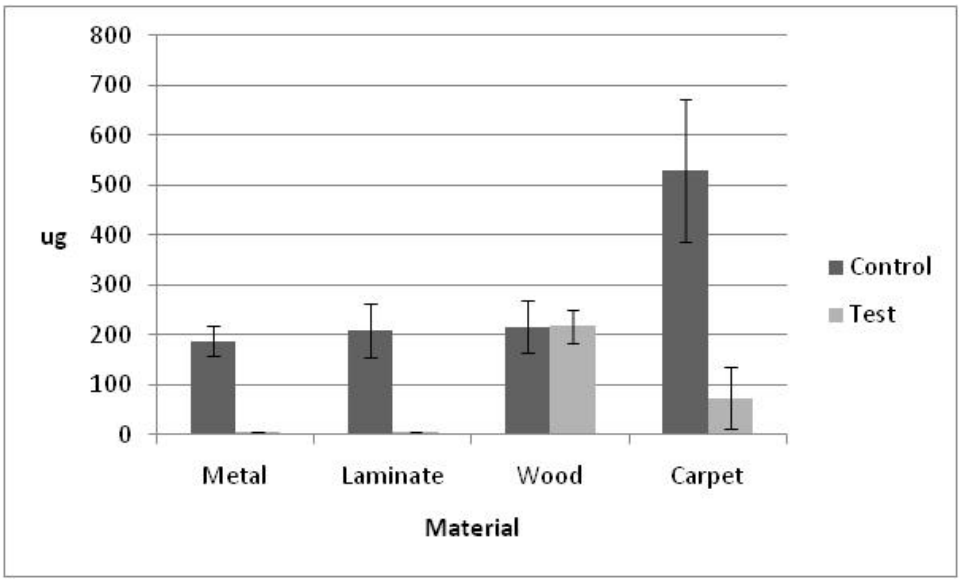

Figure 4-11. Mean measured mass and SD of GD (applied as TGD) recovered from building materials after exposure to K-O-K bleach (full strength) with a 60-minute contact time (Trial 26).

Table 4-6 summarizes the efficacies measured after applying OxiClean powder (0.06 g/mL), Zep industrial purple cleaner (25%), the Zep cleaner (full strength), K-O-K bleach (10%), and K-O-K bleach (full strength) to decontaminate TGD from galvanized metal ductwork, decorative laminate, wood flooring, and industrial grade carpet. There was significant difference in recovered mass from most materials after decontamination by all

cleaning technologies tested. Zep cleaner (full strength) exhibited moderate to high efficacies (49% - 99%) at a 60-minute contact time. K-O-K bleach (full strength) exhibited high efficacies (92% - 98%) at a 60-minute contact time, except no efficacy was observed against TGD on wood flooring.

Table 4-6. Median TGD Decontamination Efficacy Results (95% confidence interval)[a] or Efficacy (Number of Test Coupons below the Practical Quantitation Limit)[b]

Contact Time, Minutes	Material	Cleaning Technologies (Concentration)				
		OxiClean Powder (0.06 g/mL)	Zep Industrial Purple Cleaner (25%)	Zep Industrial Purple Cleaner (Full Strength)	K-O-K Bleach (10%)	K-O-K Bleach (Full Strength)
		Trial 1	Trial 3	--	--	--
30	Galvanized metal ductwork	21% (16% - 26%)	NS	--	--	--
30	Decorative laminate	40% (I - 63%)	78% (49% - 99%)	--	--	--
30	Wood flooring	44% (32% - 56%)	43% (10% - 61%)	--	--	--
30	Industrial grade carpet	86% (79%-90%)	35% (22% - 58%)	--	--	--
		--	--	Trial 27	Trial 25	Trial 26
60	Galvanized metal ductwork	--	--	>99% (3/5)	76% (74% - 76%)	>98% (5/5)
60	Decorative laminate	--	--	NS	36% (30% - 79%)	>98% (5/5)
60	Wood flooring	--	--	49% (33% - 56%)	42% (33% - 49%)	NS
60	Industrial grade carpet	--	--	97% (97% - 98%)	66% (57% - 69%)	92% (81% - 92%)

Symbols and acronyms: -- is not tested, NS indicates that efficacy is not significantly different from 0 at the 95% confidence level. I indicates that recoveries from one or more of decontaminated test coupons were greater than mean recoveries from positive control coupons at test condition.

a Estimated efficacy is median efficacy (defined as one minus recovery of treated coupon divided by recovery of control coupon) from all possible combinations, expressed as percentage. Confidence intervals are 95% bias-corrected and acceleration intervals of a simulation using bootstrap sampling approach.

b One or more of test coupons had no recovered agent (exact number is shown in parentheses). The reported ">x" value is estimated efficacy with five CFU substituted for all zero recovery coupon values. For these trials, test of statistical significance is a non-parametric Kolmogorov-Smirnov test where a p-value less than 0.05 indicates a statistically significantly difference between test and positive control coupons.

4.2.3 VX Decontamination

Five decontamination technology preparations (three cleaning technologies) were used against VX on four materials (galvanized metal ductwork, decorative laminate, wood flooring, and industrial grade carpet):

- OxiClean powder (0.06 g/mL) with a 30-minute contact time
- Zep industrial purple cleaner (25%) with a 30-minute contact time
- Zep industrial purple cleaner (full strength) with a 30- and 60-minute contact time
- K-O-K bleach (10%) with a 60-minute contact time
- K-O-K bleach (full strength) with a 30- and 60-minute contact time

As shown in Figure 4-12, after exposure to OxiClean powder (0.06 g/mL) for 30 minutes, there were small differences in the average recovered mass of VX recovered from the test coupons compared to the control coupons for all of the materials tested.

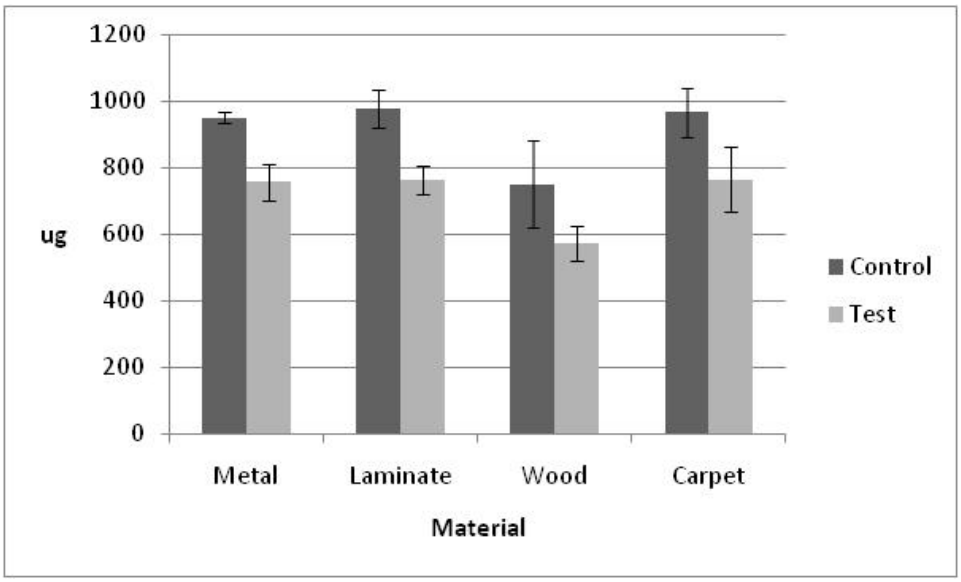

Figure 4-12. Mean measured mass and SD of VX recovered from building materials after exposure to OxiClean powder (0.06 g/mL) for 30 minutes (Trial 21).

As shown in Figure 4-13, after exposure to Zep industrial purple cleaner (25%) for 30 minutes, there were observable differences in recovered mass of VX from test coupons compared to control coupons for all of the materials tested.

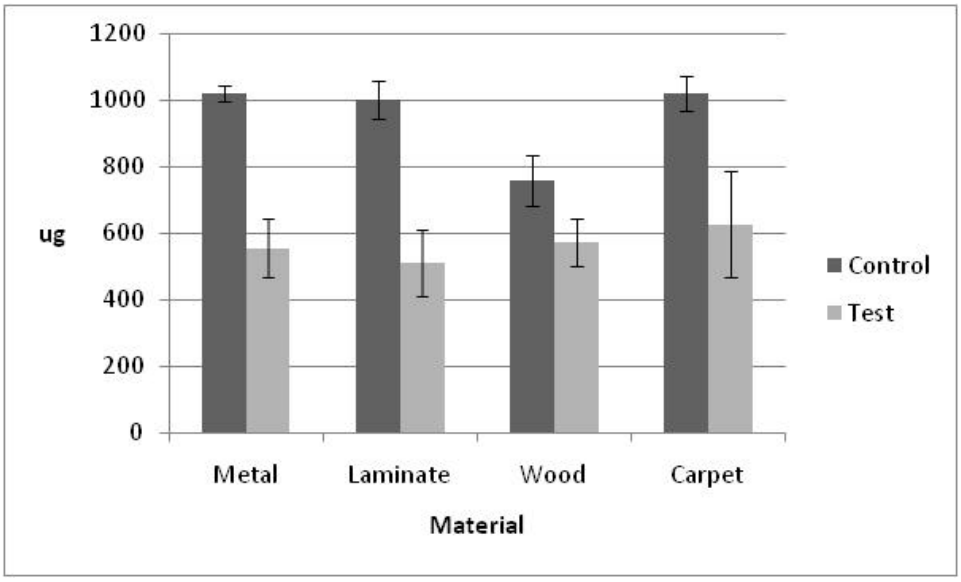

Figure 4-13. Mean measured mass and SD of VX recovered from building materials after exposure to Zep industrial purple cleaner (25%) for 30 minutes (Trial 23).

As shown in Figure 4-14, after exposure to Zep industrial purple cleaner (full strength) for 30 minutes, there were observable differences in recovered mass of VX from test coupons compared to control coupons for both of the materials tested especially for the galvanized metal material.

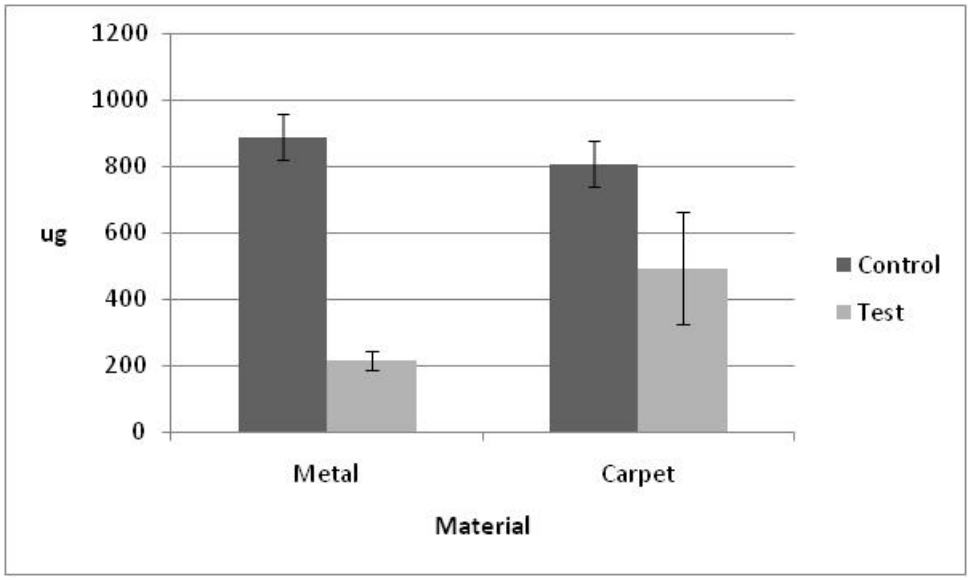

Figure 4-14. Mean measured mass and SD of VX recovered from building materials after exposure to Zep industrial purple cleaner (full strength) for 30 minutes (Trial 34).

As shown in Figure 4-15, after exposure to Zep industrial purple cleaner (full strength) for 60 minutes, there were apparent differences in recovered mass of VX from test

coupons compared to control coupons for all of the materials tested and this difference was most pronounced for the galvanized metal and decorative laminate materials.

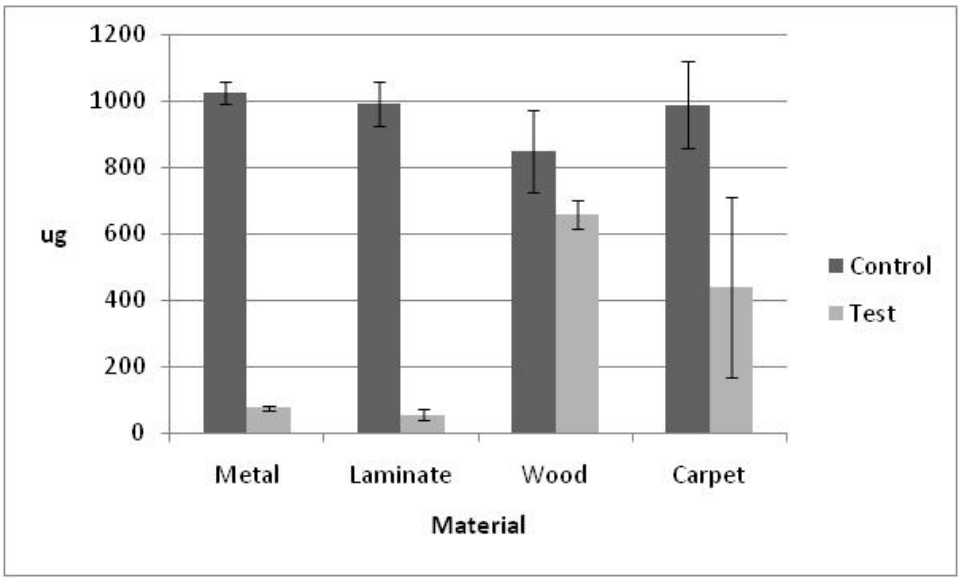

Figure 4-15. Mean measured mass and SD of VX recovered from building materials after exposure to Zep industrial purple cleaner (full strength) for 60 minutes (Trial 33).

As shown in Figure 4-16, after exposure to K-O-K bleach (10%) for 60 minutes, there were differences in recovered mass of VX from test coupons compared to control coupons for all of the materials tested. The most observable differences were seen for the wood and carpet materials.

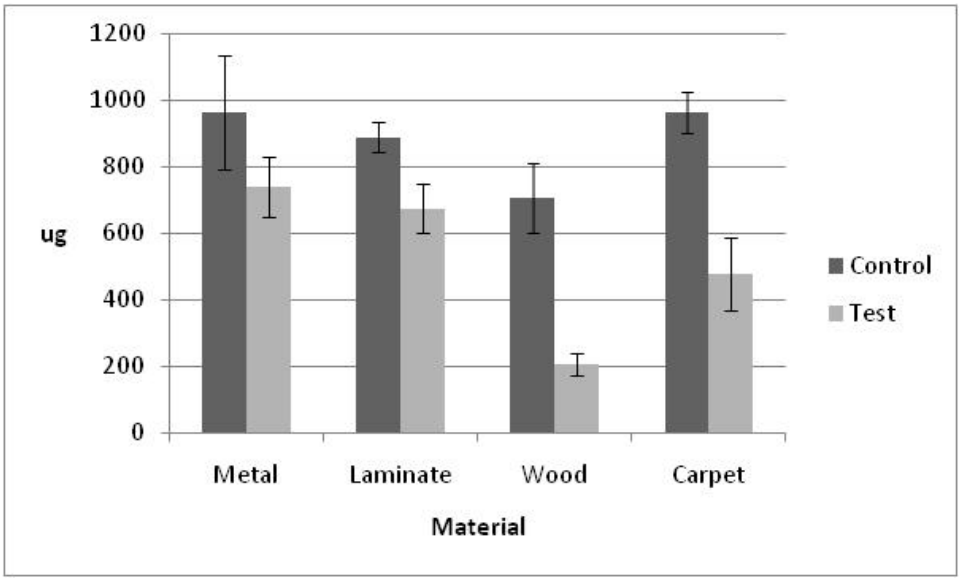

Figure 4-16. Mean measured mass and SD of VX recovered from building materials after exposure to K-O-K bleach (10%) for 60 minutes (Trial 31).

As shown in Figure 4-17, after exposure to K-O-K bleach (full strength) for 30 minutes, there were apparent differences in recovered mass of VX from test coupons compared to control coupons for galvanized metal ductwork and industrial grade carpet.

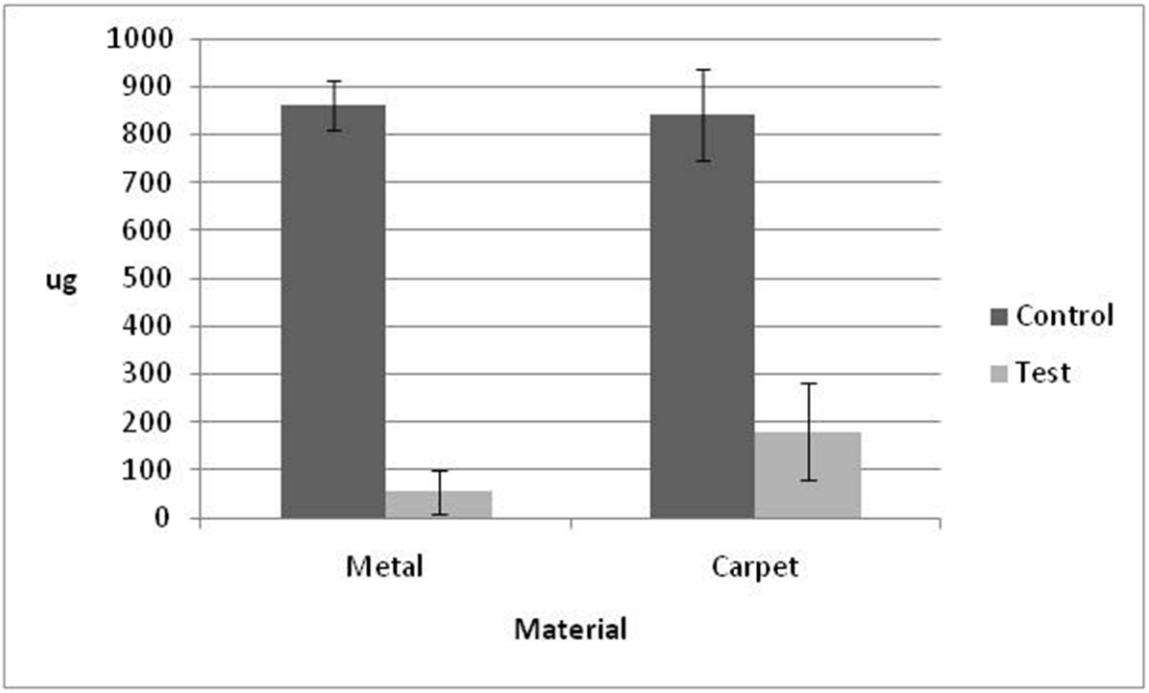

Figure 4-17. Mean measured mass and SD of VX recovered from building materials after exposure to K-O-K bleach (full strength) for 30 minutes (Trial 35).

As shown in Figure 4-18, after exposure to K-O-K bleach (full strength) for 60 minutes, there were noticeable differences in recovered mass of VX from test coupons compared to control coupons for all of the materials tested and this was most pronounced for the galvanized metal and decorative laminate materials.

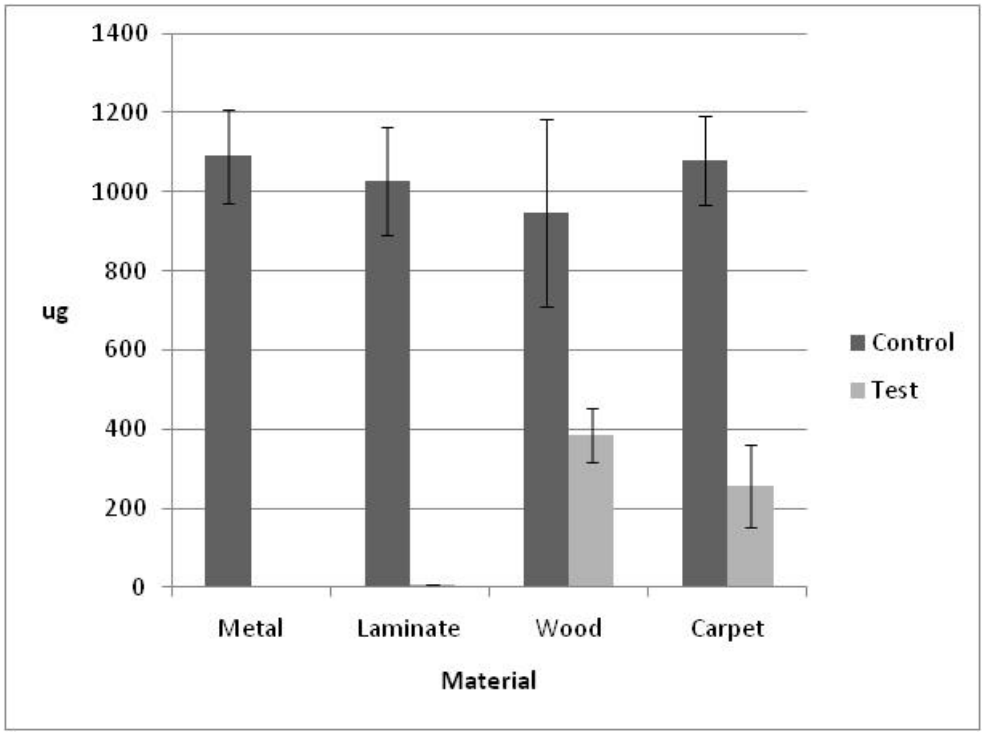

Figure 4-18. Mean measured mass of chemical agent and SD of VX recovered from building materials after exposure to K-O-K bleach (full strength) for 60 minutes (Trial 32).

Table 4-7 summarizes the median efficacy estimates and corresponding 95% confidence intervals. Efficacy was significantly different from 0 for all cleaners and materials tested. OxiClean powder (0.06 g/mL) exhibited low efficacies (18% - 22%) with a 30-minute contact time. Zep industrial purple cleaner (25%) exhibited low to moderate efficacies with a 30-minute contact time (24% - 52%). K-O-K bleach (10%) with a 60-minute contact time exhibited low to moderate efficacy (24% - 71%, respectively). The Zep cleaner (full strength) exhibited low to high efficacies (23% - 94%) at 60-minute contact time. K-O-K bleach (full strength) exhibited moderate to high efficacies (58% - >99%) at a 60-minute contact time.

Table 4-7. Median VX Decontamination Efficacy Results (95% confidence interval)[a] or Efficacy (Number of Test Coupons below the Practical Quantitation Limit)[b]

Contact Time, Minutes	Material	Cleaning Technologies (Concentration)				
		OxiClean powder (0.06 g/mL)	Zep Industrial Purple Cleaner (25%)	Zep Industrial Purple Cleaner (Full Strength)	K-O-K Bleach (10%)	K-O-K Bleach (Full Strength)
		Trial 21	Trial 23	Trial 34	--	Trial 35
30	Galvanized metal ductwork	18% (16% - 23%)	43% (39% - 51%)	76% (73% - 77%)	--	97% (91% - 98%)
30	Decorative laminate	21% (17% - 23%)	52% (41% - 55%)	--	--	--
30	Wood flooring	20% (13% - 29%)	24% (18% - 28%)	--	--	--
30	Industrial grade carpet	22% (14% - 25%)	43% (32% - 48%)	34% (25% - 40%)	--	76% (71% - 79%)
		--	--	Trial 33	Trial 31	Trial 32
60	Galvanized metal ductwork	--	--	93% (93% - 93%)	24% (10% - 29%)	>99% (5/5)
60	Decorative laminate	--	--	94% (93% - 95%)	23% (18% - 26%)	>99% (4/5)
60	Wood flooring	--	--	23% (12% - 28%)	71% (66% - 73%)	58% (49% - 65%)
60	Industrial grade carpet	--	--	66% (41% - 71%)	53% (43% - 57%)	77% (69% - 80%)

Symbols and acronyms: -- is not tested, I indicates that recoveries from one or more of decontaminated test coupons were greater than mean recoveries from positive control coupons at test condition.

a Estimated efficacy is median efficacy (defined as one minus recovery of treated coupon divided by recovery of control coupon) from all possible combinations, expressed as percentage. Confidence intervals are 95% bias-corrected and acceleration intervals of a simulation using bootstrap sampling approach.

b One or more of test coupons had no recovered agent (exact number is shown in parentheses). The reported ">x" value is estimated efficacy with five CFU substituted for all zero recovery coupon values. For these trials, test of statistical significance is a non-parametric Kolmogorov-Smirnov test where a p-value less than 0.05 indicates a statistically significantly difference between test and positive control coupons.

4.2.4 HD Decontamination

Six decontamination technology preparations (four cleaning technologies) were used against HD on four materials (galvanized metal ductwork, decorative laminate, wood flooring, and industrial grade carpet):

- OxiClean powder (0.06 g/mL) with a 30-minute contact time
- Zep industrial purple cleaner (25%) with a 30-minute contact time
- Zep industrial purple cleaner (full strength) with a 60-minute contact time
- K-O-K bleach (10%) with a 30- and 60-minute contact time
- K-O-K bleach (full strength) with a 30- and 60-minute contact time
- Cascade gel (7.3%) with a 30-minute contact time

As shown in Figure 4-19, after exposure to OxiClean powder (0.06 g/mL) for 30 minutes, there were no clearly discernible reductions in recovered mass of HD from test coupons compared to control coupons for all of the materials tested.

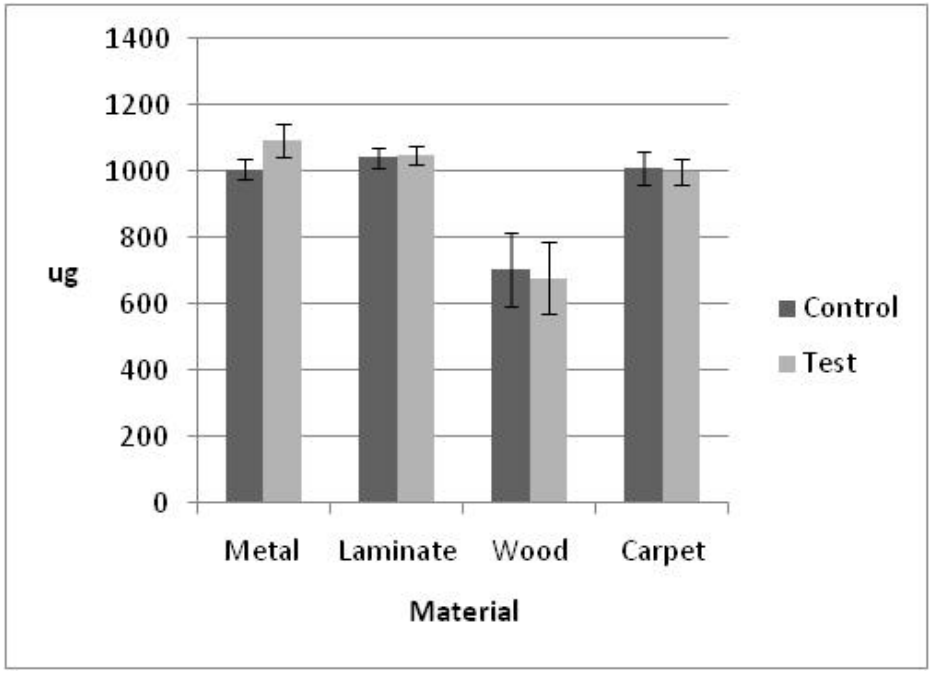

Figure 4-19. Mean measured mass and SD of HD recovered from building materials after exposure to OxiClean powder (0.06 g/mL) for 30 minutes (Trial 5).

Figure 4-20 and 4-21 show Zep industrial purple cleaner (25%) with a 30-minute contact time and the Zep cleaner (full strength) with a 60-minute contact time. After exposure to Zep cleaner (25%) for 30 minutes, there was no observable decrease in recovered mass of HD from test coupons compared to control coupons for all of the materials tested. After exposure to Zep industrial purple cleaner (full strength) for 60 minutes, there were small discernible differences in average recovered mass of HD from galvanized metal ductwork and industrial grade carpet test coupons compared to control coupons.

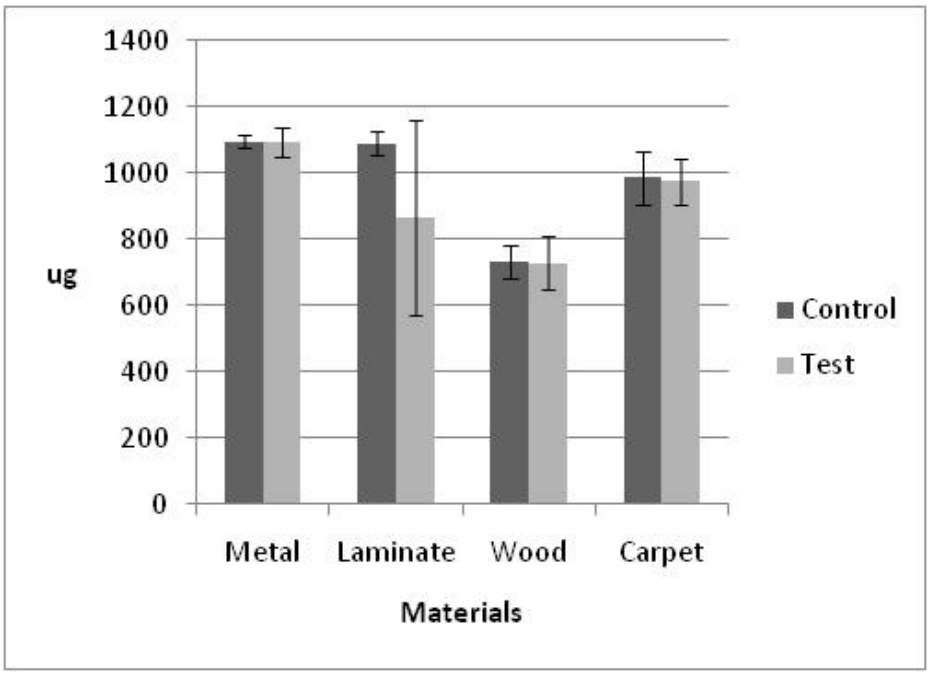

Figure 4-20. Mean measured mass and SD of HD recovered from building materials after exposure to Zep industrial purple cleaner (25%) for 30 minutes (Trial 7).

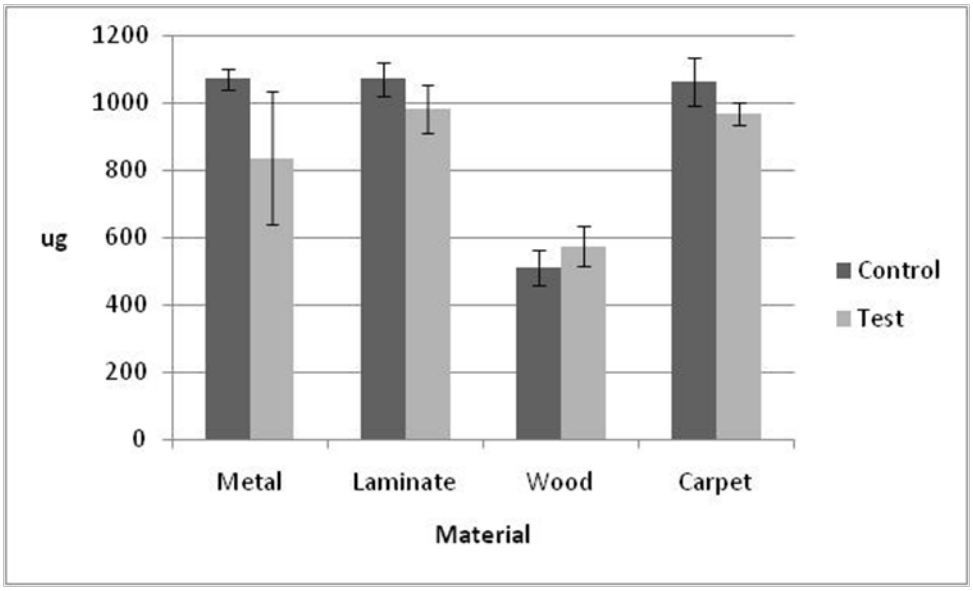

Figure 4-21. Mean measured mass and SD of HD recovered from building materials after exposure to Zep industrial purple cleaner (full strength) for 60 minutes (Trial 28).

Figure 4-22 shows that after exposure to K-O-K bleach (10%) for 30 minutes, there were apparent differences in recovered mass of HD from galvanized metal ductwork and

decorative laminate test coupons compared to control coupons while there appears to be no observable difference in the test and control recoveries for the wood flooring material.

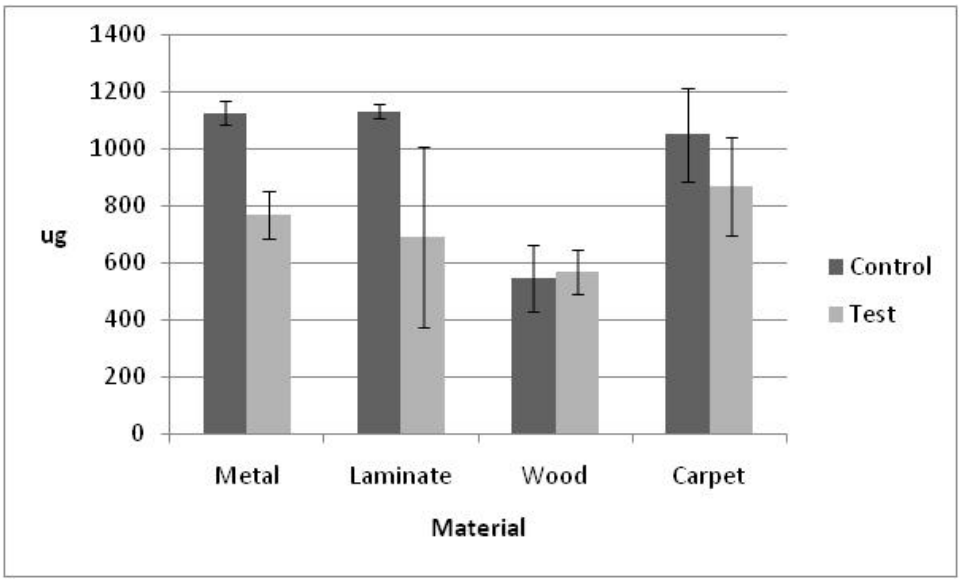

Figure 4-22. Mean measured mass and SD of HD recovered from building materials after exposure to K-O-K bleach (10%) for 30 minutes (Trial 9).

Figure 4-23 shows that after exposure to K-O-K bleach (10%) for 60 minutes, there were discernable differences in recovered mass of HD from galvanized metal ductwork, and decorative laminate, and a small difference in the average recoveries for industrial grade carpet test coupons compared to control coupons.

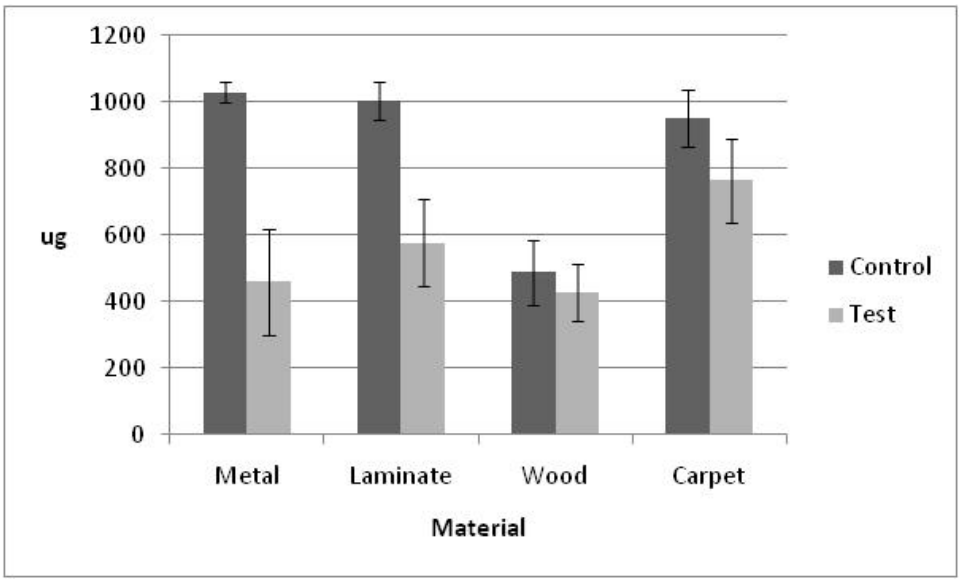

Figure 4-23. Mean measured mass and SD of HD recovered from building materials after exposure to K-O-K bleach (10%) for 60 minutes (Trial 10).

Figures 4-24 and 4-25 show that after exposure to K-O-K bleach (full strength) for 30 or 60 minutes, there were apparent differences in recovered mass of HD from test coupons compared to control coupons for all materials tested. Increasing the K-O-K bleach (full strength) contact time from 30 minutes to 60 minutes did not decrease the mass of HD recovered from the test coupons.

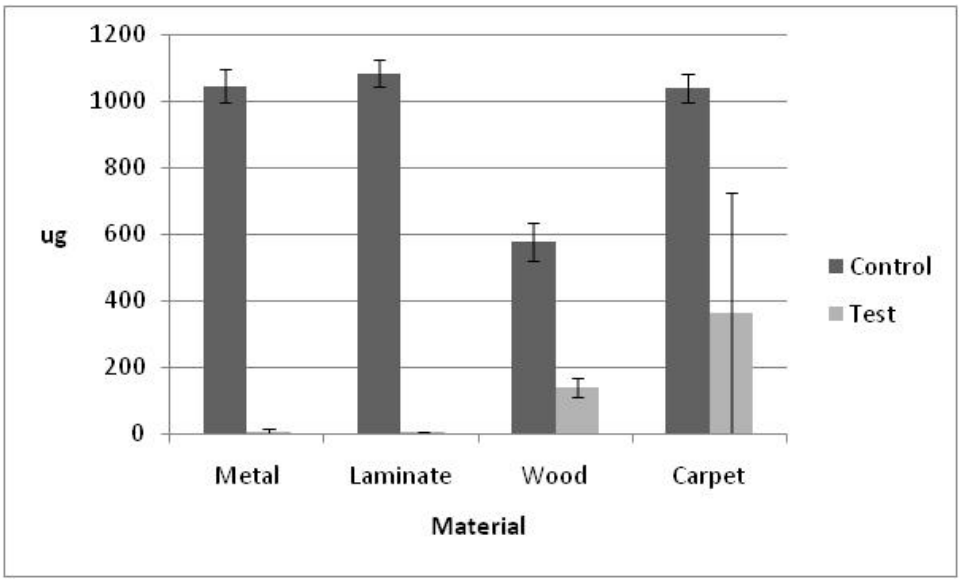

Figure 4-24. Mean measured mass and SD of HD recovered from building materials after exposure to K-O-K bleach (full strength) for 30-minutes (Trial 19).

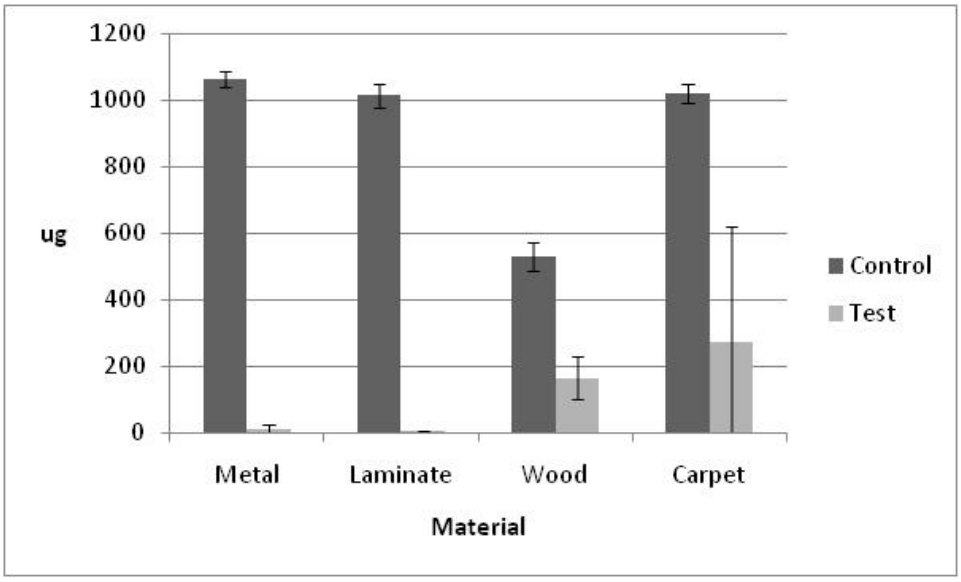

Figure 4-25. Mean measured mass and SD of HD recovered from building materials after exposure to K-O-K bleach (full strength) for 60 minutes (Trial 20).

As shown in Figure 4-26, after exposure to Cascade gel (7.3%) for 30 minutes, there was no discernible decrease in recovered mass of HD from test coupons compared to control coupons for all of the materials tested.

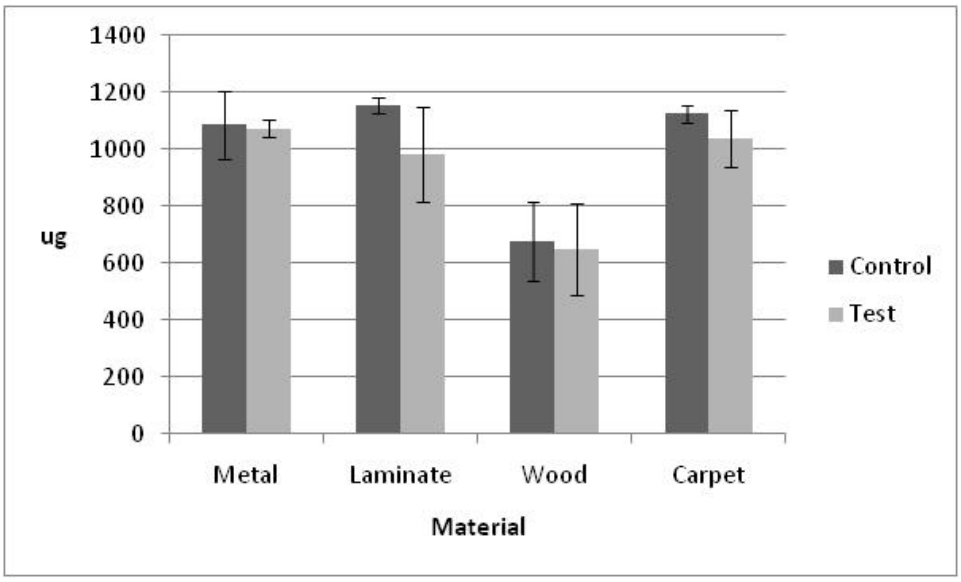

Figure 4-26. Mean measured mass and SD of HD recovered from building materials after exposure to Cascade gel for 30 minutes (Trial 11).

Table 4-8 summarizes the median efficacy estimates and corresponding 95% confidence intervals. Tests in which efficacy was not significantly different from 0 are noted. In the majority of cases, the decontamination exhibited some efficacy. The use of OxiClean powder (0.06 g/mL) and Zep industrial purple cleaner (25%) for 30 minutes to decontaminate HD was largely ineffective.

The Zep cleaner (full strength) after a 60-minute contact time exhibited low efficacy (0% - 13%) against HD. K-O-K bleach (10%) after a 30- or 60-minute contact time exhibited low efficacy to moderate efficacy, except that no significant efficacy was exhibited against HD on wood. K-O-K bleach (full strength) exhibited significant efficacy (66% - >99%) after a 30- or 60-minute contact time. The longer contact time had little or no impact on the efficacy of the K-O-K bleach (full strength) against HD.

Table 4-8. Median HD Decontamination Efficacy Results (95% confidence interval)[a] or Efficacy (Number of Test Coupons below the Practical Quantitation Limits)[b]

Contact Time, Minutes	Material	Cleaning Technologies (Concentration)					
		OxiClean powder (0.06 g/mL)	Zep Industrial Purple Cleaner (25%)	Zep Industrial Purple Cleaner (Full Strength)	K-O-K Bleach (10%)	K-O-K Bleach (Full Strength)	Cascade gel (7.3%)
		Trial 5	Trial 7	--	Trial 9	Trial 19	Trial 11
30	Galvanized metal ductwork	0%[c] (I)	NS	--	33% (27% - 35%)	>99% (4/5)	NS
30	Decorative laminate	NS	5% (1% - 48%)	--	31% (23% - 32%)	>99% (5/5)	9% (7% - 10%)
30	Wood flooring	NS	NS	--	NS	76% (72% - 79%)	NS
30	Industrial grade carpet	NS	NS	--	18% (1% - 24%)	77% (67% - 83%)	8% (0% - 11%)
		--	--	Trial 28	Trial 10	Trial 20	--
60	Galvanized metal ductwork	--	--	13% (8% - 40%)	59% (54% - 63%)	>99% (4/5)	--
60	Decorative laminate	--	--	8% (5% - 11%)	41% (34% - 43%)	>99% (5/5)	--
60	Wood flooring	--	--	0%[c] (I)	NS	66% (63% - 69%)	--
60	Industrial grade carpet	--	--	7% (4% - 11%)	21% (10% - 27%)	97% (49% - 97%)	--

Symbols and acronyms: -- is not tested, NS indicates that efficacy is not significantly different from 0 at the 95% confidence level. I indicates recoveries from one or more of decontaminated test coupons were greater than mean recoveries from positive control coupons at test condition.

a Estimated efficacy is median efficacy (defined as one minus recovery of treated coupon divided by recovery of control coupon) from all possible combinations, expressed as percentage. Confidence intervals are 95% bias-corrected and acceleration intervals of a simulation using bootstrap sampling approach.

b One or more of test coupons had no recovered agent (exact number is shown in parentheses). The reported ">x" value is estimated efficacy with five CFU substituted for all zero recovery coupon values. For these trials, test of statistical significance is a non-parametric Kolmogorov-Smirnov test where a p-value less than 0.05 indicates a statistically significantly difference between test and positive control coupons.

[C] Mean recovery from decontaminated test coupons was greater than mean recovery from the positive control coupons at test condition.

4.2.5 Results of Statistical Comparisons

Table 4-9 compares the median efficacy for agent, cleaning liquid, and material combinations where testing was conducted at both 30 and 60 minute contact times. The results show with the longer contact time results are mixed. Some combinations (e.g., HD decontaminated with K-O-K bleach [10%] on galvanized metal ductwork, VX decontaminated with Zep industrial purple cleaner [Full Strength] on galvanized metal ductwork) show a significant improvement in efficacy for the longer contact time. However, other cases (K-O-K bleach [Full Strength] to decontaminate HD or THD from wood flooring) do not show increased decontamination at 60 minutes than at 30 minutes.

Table 4-9. Estimated Difference in Efficacy between 30 Minutes and 60 Minutes within Each Combination of Chemical Agent, Cleaning Technology, and Material

Chemical Agent	Cleaning Technology	Material			
		Galvanized Metal Ductwork	Decorative laminate	Wood flooring	Industrial Grade Carpet
THD	K-O-K Bleach (Full Strength)	NA[a]	NA [a]	NS[b]	NS[b]
VX	Zep Industrial Purple Cleaner (Full Strength)	**17% (16% – 17%)**	--	--	**20% (14% - 24%)**
VX	K-O-K Bleach (Full Strength)	NA[a]	--	--	NS[b]
HD	K-O-K Bleach (10%)	**27% (26% - 28%)**	**10% (9% - 11%)**	**17% (14% - 19%)**	**3% (1% - 6%)**
HD	K-O-K Bleach (Full Strength)	NA[a]	NA[a]	NS[b]	**14% (13% - 15%)**

Symbols and acronyms: --is not tested,
Note: Results show in **bold** indicate that efficacy (reduction in recovered HD) is significant at the 95% confidence level.
a Differences in means of positive control coupons or patterns of complete decontamination in test coupons rendered any statistical comparisons indeterminate.
b No significant efficacy (reduction in recovered chemical agent) was observed.

Table 4-10 compares the median efficacy for agent and material combinations where testing was conducted with both diluted and full-strength liquid decontamination technologies. K-O-K bleach (10%) showed a statistically significantly better decontamination efficacy than the K-O-K bleach (Full Strength) against TGD and VX on wood; Zep industrial purple cleaner against VX on carpet showed little difference in efficacy.

Table 4-10. Estimated Difference in Efficacy between Reduced Strength and Full Strength Cleaning Technology within Each Combination of Chemical Agent Contact Time and Material

Chemical Agent	Cleaning Technology	Contact Time, minutes	Estimated Difference in Efficacy (95% confidence interval)[a] or Estimated Difference in Efficacy (number of treatment coupons that had no recovered agent)[b]			
			Galvanized Metal Ductwork	Decorative laminate	Wood flooring	Industrial Grade Carpet
THD	K-O-K Bleach (Full Strength) vs. K-O-K Bleach (10%)	60	**>35% (5/5)**	**>47% (5/5)**	**28% (25% - 30%)**	**24% (21% - 27%)**
TGD	K-O-K Bleach (Full Strength) vs. K-O-K Bleach (10%)	60	NA[c]	NA[c]	d	**22% (19% - 24%)**
VX	Zep® Industrial Purple Cleaner (Full Strength) vs. Zep Industrial Purple Cleaner (25%)	30	**32% (31% - 33%)**	--	--	d
VX	K-O-K Bleach (Full Strength) vs. K-O-K Bleach (10%)	60	**>76% (5/5)**	NA[c]	d	**25% (24% - 27%)**
HD	K-O-K Bleach (Full Strength) vs. K-O-K Bleach (10%)	60	**>39% (4/5)**	**59% (5/5)**	**57% (54% - 59%)**	**64% (62% - 65%)**
HD	K-O-K Bleach (Full Strength) vs. K-O-K Bleach (10%)	30	**>66% (4/5)**	**>69% (5/5)**	**79% (76% - 81%)**	**57% (54% - 59%)**

Symbols and acronyms: -- Indicates that the test was not performed.
Note: Results in **bold** indicate that the difference in efficacy is significant at the 95% confidence level.
a Estimated efficacy difference is efficacy for full strength minus efficacy for reduced strength cleaning technology. Confidence intervals are the 95% bias-corrected and acceleration intervals of a simulation using a bootstrap sampling approach.
b One or more of treatment coupons for full strength cleaning technology had no recovered agent (the exact number is shown in parentheses). Reported ">x" value is estimated efficacy difference with five CFU substituted for all zero recovery coupon values. For these trials, test of statistical significance is non-parametric Kolmogorov-Smirnov test where p-value less than 0.05 indicates statistically significantly difference between efficacy for full strength cleaning technology and the efficacy for reduced strength cleaning technology. Approach assumes that there is no significant difference between positive controls for full strength cleaning technology and positive controls for reduced strength cleaning technology. This condition was verified through a separate Kolmogorov-Smirnov test comparing positive control coupons.
c Differences in means of positive control coupons or patterns of complete decontamination in test coupons rendered any statistical comparisons indeterminate.
d No significant increase in efficacy was observed at the higher concentration of the cleaning technology; efficacy was significantly higher at the lower concentration.

The relative efficacy for different coupon materials for a given agent, cleaning technology, and contact time was also analyzed. With occasional exceptions, significant differences in efficacy were observed depending on the material onto which the chemical agent was applied. There was not a clear pattern in which one material was consistently easier or more difficult to decontaminate than another.

4.2.6 Toxic By-products from Decontamination

Table 4-11 summarizes the results of the qualitative analyses to tentatively identify toxic HD by-products. For HD by-product evaluation, procedural blank and HD test coupon extracts decontaminated with Zep industrial purple cleaner (full strength), K-O-K bleach (10%), and K-O-K bleach (full strength) were analyzed by GC/MS in the full-scan mode. A mass spectral library was used to tentatively identify compounds in the mass spectra. The analysis indicated that toxic by-products were generated by Zep cleaner (full strength), K-O-K bleach (10%), and K-O-K bleach (full strength).

Table 4-12 summarizes the results of the semi-quantitative analyses to determine if EA 2192, the toxic VX decontamination by-product that can be potentially formed, is produced as a by-product of decontamination. EA 2192 measurements are based on comparison to the calibration response curve, which was not validated, and should be considered relative to the calibration curve. (Testing for by-products for this task order was only at a qualitative level.) With this caveat, the semi-quantitative results are included in this report.

With Zep cleaner (full strength) in contact for 30 minutes with VX, 14 μg/mL of EA 2192 was measured by LC/MS. The molecular weight of VX is 267.38 and EA 2192 is 239.32. For each microgram of VX converted to EA 2192, there are 0.895 (ratio of molecular weights of EA 2192 to VX) micrograms of EA 2192 produced. Therefore, 14 μg/mL EA 2192 / 0.895 = 15.6 μg/mL VX converted to EA 2192 by treatment with Zep cleaner (full strength). Because 856 μg/mL of VX were in the original sample, about 1.8% of the VX was in the EA 2192 by-product after decontamination with Zep cleaner (full strength).

With K-O-K bleach (10%) in contact for 30 minutes with VX, 4 μg/mL of EA 2192 was measured by LC/MS. Therefore, 4 μg/mL / 0.895 = 4.5 μg/mL of VX converted to EA 2192 by treatment with K-O-K bleach (10%). In the 1,051 mL of total solution there were 900 μg of VX, yielding an initial concentration of 856 μg/mL of VX. Given 860 μg/mL of VX were in the original sample, about 0.5% of the VX was in the EA 2192 by-product after decontamination with K-O-K bleach (10%). This is expected because the pH of the KOK bleach (10%) is around 7 which is in the optimal pH range (7-10) for EA-2192 formation.

With K-O-K bleach (full strength) decontamination of VX, no EA 2192 was detected in a 1:100 dilution of the samples by LC/MS. The lower limit of quantitation was 1 ng/mL. Therefore with K-O-K bleach (full strength), decontamination of VX, accounting for the dilution, less than 0.1 μg /mL EA 2192 was present in the sample. This result sets an upper limit for VX conversion to EA 2192 of about 0.01%.

Table 4-11. Tentatively Identified Potentially Toxic HD Decontamination By-Products

Material	Cleaning Solution	Toxic HD Decontamination By-Products and Toxicity Data
Galvanized metal ductwork	Zep Industrial Purple Cleaner (full strength)	[a]O-Mustard [CAS 063918-89-8] Human: inhalation; lowest lethal concentration (LC_{Lo}): 400 mg/m3; [a]2-Thiophene acetonitrile [CAS 020893-30-5] Rat: oral lethal dose, 50% (LD_{50}): 87 mg/kg
Decorative laminate	Zep Industrial Purple Cleaner (full strength)	[a]*3-Chloro-2-methylthiopropene* [CAS 015893-05-7]; [a]Thiocyanic acid, 2-(2-butoxyethoxy)ethyl ester [CAS 000112-56-1] Dog: oral LD_{50}: 30 mg/kg
Wood flooring	Zep Industrial Purple Cleaner (full strength)	None
Industrial grade carpet	Zep Industrial Purple Cleaner (full strength)	None
Galvanized metal ductwork	K-O-K bleach (10%)	[b]Divinyl sulfone [CAS 000077-77-0], Rat: oral LD_{50}: 170 mg/kg
Decorative laminate	K-O-K bleach (10%)	[a]Divinyl sulfone [CAS 000077-77-0]
Wood flooring	K-O-K bleach (10%)	None
Industrial grade carpet	K-O-K bleach (10%)	[a]*1,3-bis(Ethylthio)-propane* [CAS 33672-52-5]
Galvanized metal ductwork	K-O-K bleach (full strength)	[b]Divinyl sulfone [CAS 000077-77-0] Rat: oral LD_{50}: 32 mg/kg; [a]bis (beta-Chloroethyl) sulfone (Mustard sulfone) [CAS 000471-03-4] Cat: inhalation LC_{Lo} - 1430 mg/m^3/10 months
Decorative laminate	K-O-K bleach (full strength)	[b]Divinyl sulfone [CAS 000077-77-0]; [a]*bis(beta-Chloroethyl) sulfoxide (Mustard sulfoxide)* [CAS 005819-08-9]; [b]*bis(beta-Chloroethyl) sulfone* [CAS 000471-03-4]
Wood flooring	K-O-K bleach (full strength)	None
Industrial grade carpet	K-O-K bleach (full strength)	None

Note: Toxicity information is from the RTECS® (Registry of Toxic Effects of Chemical Substances) [9] and TOMES system.[10]
Toxicity data were not available in RETECS or TOMES for by-products shown in italics. LC_{Lo} is the lowest concentration of a material in air reported to have caused the death of an animal or human. LD_{50} is the median lethal dose, the dose at which half the members of a tested population are killed.
a Found in only one of the replicate extracts
b Found in both replicate extracts

Table 4-12. EA 2192 VX Decontamination By-Product

Decontamination Solution	EA 2192 after VX Decontamination (approximate)	% of Initial VX Remaining as EA 2192 (approximate)
Zep Industrial Purple Cleaner (full strength)	14 µg/mL	1.6
K-O-K bleach (10%)	4 µg/mL	0.5
K-O-K bleach (full strength)	<0.1 µg /mL	<0.01

4.2.7 Observations of Damage to Coupons from Decontamination

One cleaning technology, Zep industrial purple cleaner, caused visible discolorations on laminate coupons when used at full strength. (No photograph is shown here because the observed discoloration was not clearly visible in the printed photograph.) This discoloration occurred on coupons exposed only to the cleaner, as well as on coupons exposed to chemical agent (VX, THD, or HD) and cleaner for both contact times.

A second cleaner technology, K-O-K bleach (full strength), exhibited a visible reaction with chemical agent VX when used at full strength on galvanized metal coupons. Shown in Figure 4-27, the reaction produced gas bubbles at the contact zone between the agent droplet and the cleaner. The metal coupons did not appear to be affected by this reaction. Metal coupons exposed only to the K-O-K bleach (full strength) did not exhibit any signs of reaction or discoloration. No other surface damage was observed.

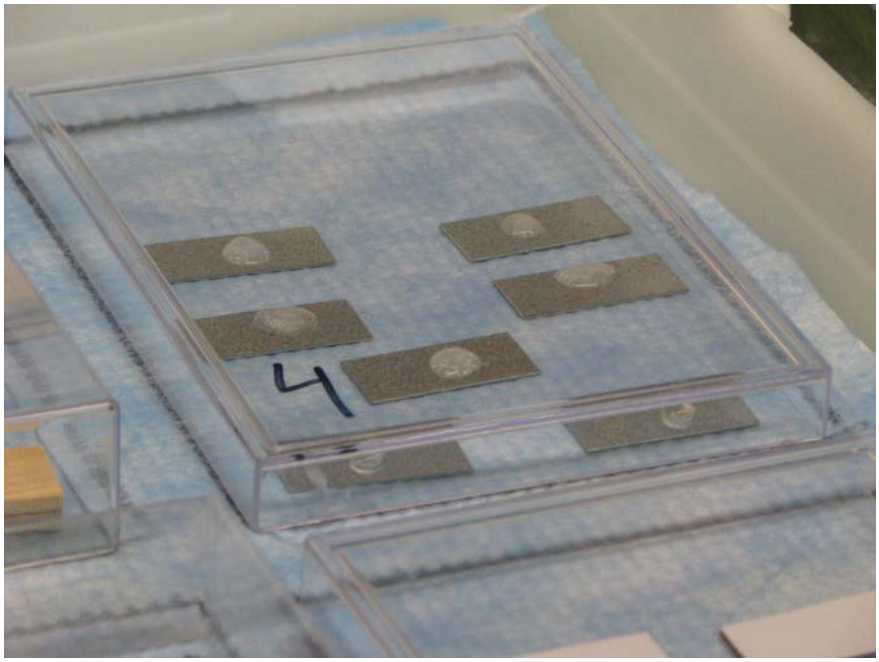

Figure 4-27. Galvanized metal coupons with K-O-K bleach (full strength) showing bubbles from reaction with VX.

5.0 Summary

The results of the bench-scale testing to evaluate the efficacy of cleaning technologies (OxiClean Versatile, Zep industrial purple cleaner, K-O-K bleach, and Cascade with Extra Bleach Action Gel) on chemical agents (THD, TGD, VX, and HD) are summarized in Table 5-1 and 5.2. Note that not all combinations of chemical agents and cleaning technologies were evaluated. The results showed a range of efficacies that were dependent on the type and strength of the decontamination technology, the chemical agent, and the material onto which the chemical agent was applied. Under the conditions and with the materials tested, only K-O-K (full strength) was highly effective (≥50% efficacy) in removing all chemical agents from all materials, that is, with the exception TGD. The Zep cleaner (full strength) exhibited moderate to high efficacies against GD (49% - 99%) at a 60-minute contact time on all surfaces except decorative laminate where the efficacy was not significant (NS). Of the cleaning technologies tested, full-strength K-O-K bleach generally had the highest efficacy against THD, VX, and HD. In addition, the statistical comparisons showed that increasing contact time for this decontaminant did not always result in improved efficacy, specifically, for some of the porous material-agent combinations. This was observed for THD on wood flooring and carpet where increasing the contact time from 30 to 60 minutes did not increase the full strength K-O-K's efficacy. This effect was also seen full strength K-O-K decontamination of VX deposited on carpet and HD deposited on wood flooring.

On some materials, toxic by-products of HD were generated by Zep cleaner (full strength), K-O-K bleach (10%), and K-O-K bleach (full strength). Both Zep cleaner (full strength) and K-O-K bleach (10%) decontamination of VX for a contact time of 30 minutes yielded measurable quantities of the toxic by-product EA 2192. The amounts of EA 2192 represent conversion of about 1.8% and 0.5%, respectively, of the VX. After K-O-K bleach (full strength) decontamination of VX with a contact time of 30 minutes, EA 2192 was not detected, indicating an upper limit for VX conversion to EA 2192 of about 0.01%. VX is reported to convert to EA 2192 under mildly alkaline conditions (pH 7–10). Because K-O-K bleach (10%) is in this range, EA 2192 is formed. The use of full strength bleach (pH > 12), as recommended by the military, appears to be the best proven solution at this time.

Little material damage was visually apparent from the use of the cleaning technologies. Zep cleaner (full strength) caused visible discolorations on laminate coupons. No other surface damage was observed.

Table 5-1. Summary of Decontamination Efficacies for the Cleaners against Chemical Agents Deposited on Non-Porous Surfaces.

Chemical Agent	Cleaning Technologies (Concentration)					
	OxiClean® Versatile (0.06 g/mL)	Zep® Industrial Purple (25%)	Zep® Industrial Purple (Full Strength)	K-O-K® Bleach (10%)	K-O-K® Bleach (Full Strength)	Cascade® with Extra Bleach Action Gel (7.3%)
30-Minute Contact Time						
THD	GM-NS, DL-NS	GM-NS, DL-NS	--	--	GM->99%, DL->99%	--
TGD	GM-21%, DL-40%	GM-NS, DL-78%	--	--	--	--
VX	GM-18%, DL- 21%	GM-43%, DL-52%	GM-76%, DL-ND[a]	--[a]	GM-97%, DL-ND[a]	--
HD	GM-0%, DL-NS	GM-NS, DL-5%	--	GM-33%[b], DL-31%[b]	GM->99%[b], DL->99%[b]	GM-NS, DL-9%
60-Minute Contact Time						
THD	--	--	GM-0%, DL-82%[c]	GM- 65%, DL-52%	GM->99%, DL->99%	--
TGD	--	--	GM->99%, DL-NS	GM-76%, DL-36%	GM>98%, DL>98%	--
VX	--	--	GM-93%, DL-94%	GM-24%, DL-23%	GM- >99%, DL->99%	--
HD	--	--	GM-13%[b], DL-8%[b]	GM-59%, DL-41%	GM- >99%, DL->99%	--

GM=galvanized metal ductwork, DL=decorative laminate

-- or ND Indicates that this combination of chemical agent-cleaning technology-surface material was not studied.

NS indicates that efficacy is not significantly different from 0 at the 95% confidence level.

[a] Tested for EA 2192 in solution based testing (cleaner and agent combined in vial – no building materials present). Red color indicates that EA 2192 was found during this solution based testing.

[b] Tested for toxic or potentially toxic by-products for HD decontamination. Orange color indicates that one or more toxic or potentially toxic HD decontamination by-products were tentatively identified for this material-cleaner combination.

[c] Significant efficacy was only observed for decorative laminate. Result is not consistent with results from other materials when Zep® cleaner (full strength) was applied for 60 minutes; efficacy should be considered questionable unless supported by additional testing.

Table 5-2. Summary of Decontamination Efficacies for the Cleaners against Chemical Agents Deposited on Porous Surfaces.

Chemical Agent	Cleaning Technologies (Concentration)					
	OxiClean® Versatile (0.06 g/mL)	Zep® Industrial Purple (25%)	Zep® Industrial Purple (Full Strength)	K-O-K® Bleach (10%)	K-O-K® Bleach (Full Strength)	Cascade® with Extra Bleach Action Gel (7.3%)
30-Minute Contact Time						
THD	WF-NS, IC-0%	WF-NS, IC-NS	--	--	WF-74%, IC-82%	--
TGD	WF-44%, IC-86%	WF-43%, IC-35%	--	--	--	--
VX	WF-20%, IC-22%	WF-24%, IC-43%	WF-ND, IC-34%[a]	--[a]	WF-ND, IC-76%[a]	--
HD	WF-NS, IC-NS	WF-NS, IC-NS	--	WF-NS[b], IC-18%[b]	WF-76%[b], IC-77%[b]	WF-NS, IC-8%
60-Minute Contact Time						
THD	--	--	WF-NS, IC-0%	WF-21%, IC-49%	WF-52%, IC-60%	--
TGD	--	--	WF-49%, IC-97%	WF-42%, IC-66%	WF-NS IC-92%	--
VX	--	--	WF-23%, IC-66%	WF-71%, IC-53%	WF-58%, IC-77%	--
HD	--	--	WF-0%[b], IC-7%[b]	WF-NS, IC-21%	WF-66%, IC-97%	--

WF=wood flooring, IC=industrial grade carpet
-- or ND Indicates that this combination of chemical agent-cleaning technology-surface material was not studied.
NS indicates that efficacy is not significantly different from 0 at the 95% confidence level.
[a] Tested for EA 2192 in solution based testing (cleaner and agent combined in vial – no building materials present). Red color indicates that EA 2192 was found during this solution based testing.
[b] Tested for toxic or potentially toxic by-products for HD decontamination.
Orange color indicates that one or more toxic or potentially toxic HD decontamination by-products were tentatively identified for this material-cleaner combination.

6.0 References

1. Yang, Yu-Chu, James A. Baker, and J. Richard Ward. 1992. Decontamination of Chemical Warfare Agents. *Chemical Reviews,* 92(8): 1729-1743.

2. Munro, Nancy B., Sylvia S. Talmage, Guy D. Griffin, Larry C. Waters, Annetta P. Watson, Joseph F. King, and Veronique Hauschild. 1999. The Sources, Fate, and Toxicity of Chemical Warfare Agent Degradation Products. *Environmental Health Perspectives*, 107(12): 933 – 974.

3. U.S. EPA. 2009. *Decontamination of Toxic Industrial Chemicals and Chemical Warfare Agents on Building Materials Using Chlorine Dioxide Fumigant and Liquid Oxidant Technologies: Technology Investigation Report.* U.S. Environmental Protection Agency. EPA/600/R-09-0121.

4. *NBC Decontamination*. 2000. Headquarters, Department of the Army and Commandant, U.S. Marine Corps: Washington, D.C. FM 3-5. MCWP 3-37.3.

5. U.S. EPA. 2010. *Assessment of Fumigants for Decontamination of Surfaces Contaminated with Chemical Warfare Agents*. U.S. Environmental Protection Agency. EPA/600/R-10/035.

6. Bradley Efron and Robert J. Tibshirani. 1993. *An Introduction to the Bootstrap.* Boca Raton, FL: Chapman & Hall.

7. Nancy Barker, Oxford Pharmaceutical Sciences. 2005. *A Practical Introduction to the Bootstrap Using the SAS System*. Paper PK02, Pharmaceutical Users Software Exchange, October 2005, Heidelberg, Germany, http://www.lexjansen.com/phuse/2005/pk/pk02.pdf.

8. Battelle, *Quality Management Plan (QMP) for the Technology Testing and Evaluation Program (TTEP); Version 3*. January 2008.

9. RTECS® (Registry of Toxic Effects of Chemical Substances). Canadian Centre for Occupational Safety and Health.

10. *Micromedex® Healthcare Series*. n.d. Thomson Reuters (Healthcare) Inc., Greenwood Village, CO. 24 Jan. 2006 <http://www.thomsonhc.com>.

Appendix A

Table A- 2. Spike Control Results for All Trials

Recovery of Chemical Agent from Teflon® coupons					Chemical Agent in 20 mL Chloroform
Table	**Trial**	**Chemical Agent**	**Recovery (µg/mL)**	**Mean Recovery (µg/mL), Coefficient of Variance (%)**	**Spike (SD), µL**
4-22	Trial 09	HD	189.52 191.73 189.94	190.40 (0.61)	3.00 (0.02)
4-26	Trial 11	HD	192.3 188.4 187.91	189.54 (1.27)	2.99 (0.04)
4-12	Trial 21	VX	159.58 160.92 160.93	160.48 (0.49)	2.53 (0.01)
4-13	Trial 23	VX	156.62 168.39 158.16	161.06 (3.97)	2.54 (0.10)
4-20	Trial 07	HD	99.96 96.74 100.45	99.05[a] (2.04)	1.56 (0.03)
4-19	Trial 05	HD	188.62 186.28 187.77	187.56 (0.63)	2.96 (0.02)
4-7	Trial 01	TGD	78.31 177.3 200.56	152.06[b] (42.69)	2.98 (1.27)
4-8	Trial 03	TGD	282.83 310.27 200.55	264.55[b] (21.58)	5.18 (1.12)
4-1	Trial 13	THD	162.21 166.45 169.94	166.20 (2.33)	2.62 (0.06)
4-2	Trial 15	THD	169.96 178.21 171.89	173.35 (2.49)	2.73 (0.07)

a Possible that the jars were filled with 40 ml $CHCl_3$ instead of 20 mL
b Agent content in thickened Soman is not consistent

Table A-1. Spike Control Results for All Trials (continued)

Recovery of Chemical Agent from Teflon®					Chemical Agent in 20 mL Chloroform
	Trial	**Chemical Agent**	**Recovery (µg/mL)**	**Mean Recovery (µg/mL), Coefficient of Variance (%)**	**Spike (SD), µL**
4-24	Trial 19	HD	186.82 189 182.12	185.98 (1.89)	2.93 (0.06)
4-5	Trial 17	THD	182.67 173.12 170.82	175.54 (3.58)	2.77 (0.10)
4-25	Trial 20	HD	189.73 188.63 188.92	189.09 (0.30)	2.98 (0.01)
4-23	Trial 10	HD	186.07 188.53 187.26	187.29 (0.66)	2.95 (0.02)
4-6	Trial 18	THD	171.22 162.39 163.85	165.82 (2.85)	2.62 (0.07)
4-3	Trial 30	THD	162.71 161.81 127.22	150.58 (13.44)	2.38 (0.32)
4-21	Trial 28	HD	185.74 186.21 187.69	186.55 (0.55)	2.94 (0.02)
4-16	Trial 31	VX	161.48 159.65 161.04	160.72 (0.60)	2.54 (0.02)
4-11	Trial 26	TGD	99.97 117.93 96.01	104.64[b] (11.16)	1.65 (0.18)
4-18	Trial 32	VX	168.76 146.58 154.46	156.60 (7.18)	2.47 (0.18)

b Agent content in thickened Soman is not consistent

Table A-1. Spike Control Results for All Trials (continued)

Recovery of Chemical Agent from Teflon®					Chemical Agent in 20 mL Chloroform
Table	**Trial**	**Chemical Agent**	**Recovery (µg/mL)**	**Mean Recovery (µg/mL), Coefficient of Variance (%)**	**Spike (SD), µL**
4-15	Trial 33	VX	172.08 166.05 151.23	163.12 (6.58)	2.57 (0.17)
4-4	Trial 29	THD	184.57 179.89 172.94	179.13 (3.27)	2.83 (0.09)
4-10	Trial 25	TGD	146.72 143.21 131.91	140.61 (5.50)	2.22 (0.12)
4-9	Trial 27	TGD	145.11 162.04 144.05	150.40 (6.72)	2.37 (0.16)
4-5	Trial 17r	THD	153.11 168.07 171.19	164.12 (5.89)	2.59 (0.15)

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