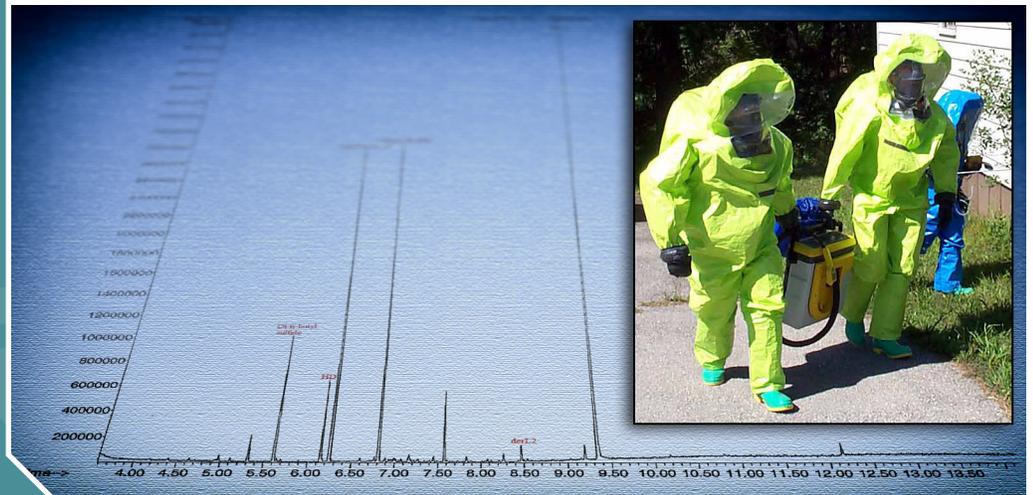


# Decontamination of Agent Yellow, a Lewisite and Sulfur Mustard Mixture



**Decontamination of Agent Yellow,  
a Lewisite and Sulfur Mustard Mixture**

**Evaluation Report**

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

## **Disclaimer**

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## Acronyms and Abbreviations

amu	atomic mass units
BCVAA	bis(2-chlorovinyl) arsonous acid
°C	degrees Celsius
CCV	continuing calibration verification
CVAA	2-chlorovinyl arsonous acid
cm	centimeter(s)
EPA	U.S. Environmental Protection Agency
derL-1	common product from reaction of butanethiol with L-1 and CVAA
derL-2	common product from reaction of butanethiol with L-2 and BCVAA
DF200	EasyDecon <sup>®</sup> DF200
GC	gas chromatography
HD	sulfur mustard
HMRC	Hazardous Materials Research Center
hr	hour(s)
HSRP	Homeland Security Research Program
L	Lewisite; when L is used in this report it refers to the synthesized product that is primarily L-1, low levels of L-2, and may contain very low levels of L-3
L-1	2-chlorovinyl dichloroarsine
L-2	bis(2-chlorovinyl) chloroarsine
L-3	tris(2-chlorovinyl) arsine
µg	microgram(s)
µL	microliter
MDL	method detection limit
mg	milligram(s)
min	minute(s)
mL	milliliter(s)
mm	millimeter(s)
MS	mass spectrometry
NHSRC	National Homeland Security Research Center
NIST	National Institute of Standards and Technology
PE	performance evaluation
PTFE	polytetrafluoroethylene
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
RSD	relative standard deviation
TSA	technical systems audit

## Executive Summary

Limited data exist on decontamination approaches that neutralize vesicant properties of Lewisite or chemical agent mixtures containing Lewisite. Recent work conducted under U.S. Environmental Protection Agency's (EPA) Homeland Security Research Program (HSRP) focused on decontamination solutions of neat Lewisite. Agent Yellow, a mixture of Lewisite (L) and sulfur mustard (HD), is a chemical warfare agent mixture and decontamination approaches to neutralize the vesicant properties of both components in this mixture are the focus of this study.

The objective of this evaluation was to develop, demonstrate and apply methods to determine the decontamination efficacies of various readily-available, liquids for the decontamination of Agent Yellow. Agent Yellow, a mixture of the chemical warfare agents Lewisite (L) and sulfur mustard (HD) was applied to  $3.5 \times 1.5$  centimeter pieces (coupons) of four types of materials: sealed concrete, wood flooring, galvanized metal, and glass. Residual Agent Yellow was extracted from the coupons and analyzed for Lewisite (measured after derivatization [derL-1]) and HD using gas chromatography/mass spectrometry (GC/MS). The extractions took place over time, to evaluate persistence, or after decontamination, to evaluate efficacy of various decontaminants.

Persistence testing showed that natural attenuation aids in the removal of Agent Yellow from the materials tested. Measurement of the persistence and decontamination of the Lewisite and HD components of Agent Yellow were made at ambient laboratory conditions (17.8 degrees Celsius ( $^{\circ}\text{C}$ ) to 20.3  $^{\circ}\text{C}$  and 7.5% to 53.5% relative humidity). Measurement of derL-1 includes detection of the L-1 vesicant (blister agent) hydrolysis by-product 2-chlorovinylarsonous acid (CVAA) as both chemicals are derivatized to derL-1. Persistence measurements under these environmental conditions showed that after application of Agent Yellow, less than 20% of the Lewisite (measured as derL-1) was recovered from sealed concrete or glass after 4 hours; however 14% and <3% of the Lewisite, respectively, were recovered after 18 hours. Similarly, 23% and 46% of the HD was recovered from sealed concrete and glass, respectively after six hours at ambient conditions. Little (4% from sealed concrete) or no (from glass) HD was recovered after 18 hours at ambient conditions.

Decontamination efficacy was evaluated for four decontaminants: bleach (full strength), bleach (10 fold dilute), hydrogen peroxide (3%), and EasyDecon<sup>®</sup> DF200 (DF200). A 30 min reaction time was evaluated for all decontaminants and material combinations. Results are summarized in Table ES-1. An additional reaction time (60 min) and a 30 min reaction time with a subsequent reapplication of the decontaminant and additional 30 min reaction time were evaluated for some combinations of decontaminants and materials.

With a 30 min reaction time, efficacy was observed for all four decontaminants against the Lewisite (L-1 and CVAA, measured as derL-1) component of Agent Yellow on all four material

types. While Table ES-1 shows that all four decontaminants were generally efficacious against the HD component of Agent Yellow, efficacies were generally lower for the HD component than for the L-1 component. Efficacies varied by material type as well as decontaminant. Increasing the reaction time to 60 min or reapplying the decontaminant (for bleach [dilute] and hydrogen peroxide [3%]) resulted in lesser amounts of L-1 and HD being recovered. These improvements in efficacy were modest at best.

Bleach (full strength; ~6% sodium hypochlorite), bleach (dilute; about 0.6% sodium hypochlorite), hydrogen peroxide (3%), and DF200 are highly efficacious against the Lewisite component of Agent Yellow and exhibit varying levels of efficacy, depending on material and decontaminant, against the HD component after 30 min reaction times. Bleach (full strength) after a 30 min reaction time generally removed Lewisite to levels at or below the quantitation limit (i.e., 2.0 µg/mL, the lowest value on the calibration curve) and exhibited efficacies for HD of 37% to >95%. The efficacy ranges for bleach (dilute), hydrogen peroxide (3%), and DF200 were comparable to bleach (full strength) for Lewisite, but had lower efficacy ranges for HD.

Qualitative analysis for L-2 (bis[2-chlorovinyl] chloroarsine and its vesicant (blister agent) by-product bis(2-chlorovinyl) arsonous acid (BCVAA; both derivatized to derL-2 prior to analysis)] and the vesicant by-product of HD, bis(beta-chloroethyl)sulfone, showed that these chemicals were generally not extracted from positive control coupons. Small chromatographic peaks consistent with vesicant by-products were detected on some materials after application of each decontaminant. DerL-2 chromatographic peaks ranging from 1% to 24% of the corresponding derL-1 peak area were found on all coupon types after decontamination with bleach (dilute) for both 30 and 60 min reaction times. HD by-product was found on all material types after hydrogen peroxide (3%) decontamination with 30 and 60 min reaction times. Chromatographic peak areas correlated with the HD by-product ranged from <1% to 38% of the corresponding HD peak area across four materials. After the 60 min reaction time the ratio of HD by-product to HD was generally higher than after the 30 min reaction time.

**Table ES-1. Summary of Average % Decontamination Efficacy with a 30 min Reaction Time**

Agent Yellow	Material	Decontaminant			
		Bleach (Full Strength)	Bleach (Dilute)	Hydrogen Peroxide (3%)	DF200
<b>derL-1</b>	Sealed Concrete	>92%	>87%	>86%	>92%
	Wood Flooring	*†	>66%	>83%	>86%
	Galvanized Metal	>79%	>94%	>92%	>87%
	Glass	>94%	>86%	>93%	>95%
Agent Yellow	Material	Decontaminant			
		Bleach (Full Strength)	Bleach (Dilute)	Hydrogen Peroxide (3%)	DF200
<b>HD</b>	Sealed Concrete	>94%	*	*	23%
	Wood Flooring	37%	*	80%	*
	Galvanized Metal	>37%	*	49%	*
	Glass	>95%	35%	42%	57%

Efficacies (E) were calculated as  $E = [(C_o - C_f)/C_o] \cdot 100\%$ , where:  $C_o$  = mean mass per coupon of agent without decontamination (determined from the positive control coupons of each material), and  $C_f$  = mass per coupon on a test coupon with decontamination.

Efficacies shown as “>” had at least one and in most cases all test coupon extracts that had values less than the quantitation limit, i.e., the calibration range (<20.0 µg/coupon).

\*No significant difference ( $p > 0.05$ ) between the mean agent remaining on the positive control coupons and the mean agent remaining on the test coupons, no significant efficacy is observed.

†There was high variability in the positive controls so the  $p = 0.007$ ; efficacy (although not significant) was 85%.

### Impact of the Study:

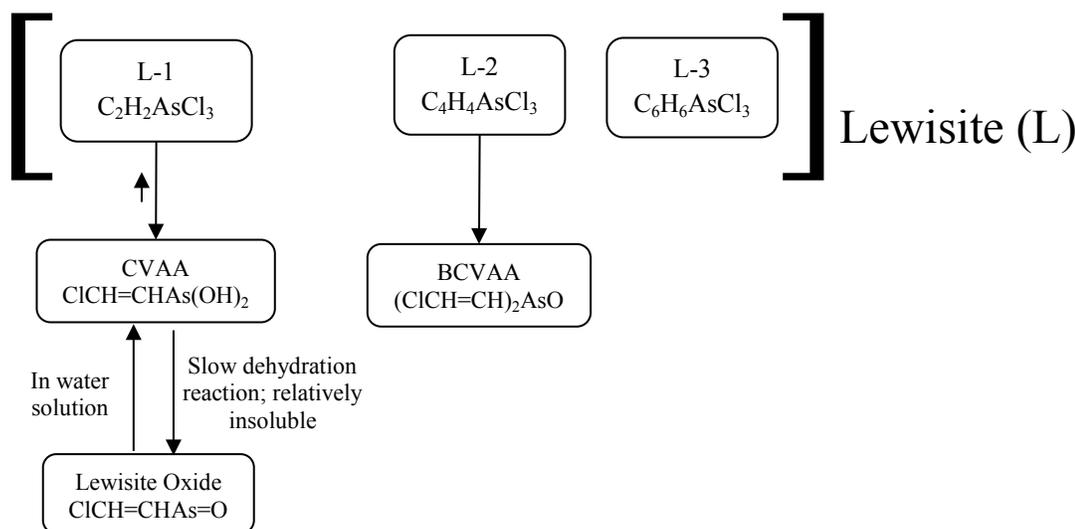
Based on the results obtained in this study, the vesicant properties of Agent Yellow, a mixture of HD and Lewisite can be neutralized by using full strength bleach. Other decontamination products evaluated in this study neutralize the Lewisite component, but would leave significant amounts of the HD component of Agent Yellow on all surfaces tested. Caution should be used in extrapolating from bench testing to field application of these decontamination solutions.

## 1.0 Introduction

Among its responsibilities related to homeland security, the U.S. Environmental Protection Agency (EPA) has the goal of identifying methods and equipment that can be used for decontamination following a terrorist attack using chemical, radiological, or biological agents. The EPA's National Homeland Security Research Center (NHSRC) has been tasked to manage, coordinate, support, and conduct a wide variety of homeland security research and technical assistance efforts. In the interest of expanding our national readiness against highly-ranked threat scenarios, NHSRC, as part of EPA's Homeland Security Research program (HSRP) is conducting tests to evaluate the performance of products, methods, and equipment for decontaminating contaminated materials. EPA has identified a lack of knowledge of simple methods for decontaminating Agent Yellow as an important gap. Agent Yellow is a chemical warfare agent that is a mixture of two blister agents or vesicants under the Chemical Weapons Convention:<sup>1</sup> Lewisite (L) and sulfur mustard (HD, bis[2-chloroethyl] sulfide; CAS 505-60-2). Shown in Figure 1, L is a mixture of up to three compounds that are produced in the synthesis:

- L-1 (2-chlorovinyl dichloroarsine, CAS 541-25-3, Lewisite);
- L-2 (bis[2-chlorovinyl] chloroarsine, CAS 40334-69-8); and
- L-3 (tris[2-chlorovinyl] arsine, CAS 40334-70-1).<sup>2</sup>

L made for weapons in the United Kingdom comprised L-1, L-2, and L-3 in a 90:9:1 ratio.<sup>3</sup> L-3 is not always present in L. (The L used in this study did not contain detectable quantities of L-3.) "Lewisite" is sometimes used to refer only to L-1. In this report, L is used in the broad sense to include L-1 and L-2 (also L-3 if present).



**Figure 1. L (L-1, L-2, and L-3) and degradation by-products.**

Agent Yellow was commonly prepared by the Japanese in World War II as a 1:1 [by volume] mixture of L and HD. Relatively undegraded Agent Yellow found in Yellow shells (the chemical

weapon) comprised, weight/weight percent, HD (43.0%), L1 (50.0%) and L2 (4.6%) of the liquid content of the shells with other chemicals comprising less than 5% of the liquid.<sup>4</sup>

As shown in Figure 1, L-1 rapidly hydrolyzes to 2-chlorovinyl arsinous acid (CVAA) in contact with water and, with excess water and slowly, is transformed to 2-chlorovinyl arsine oxide (Lewisite oxide).<sup>5,6</sup> L-1, CVAA, and Lewisite oxide have similar vesicant properties.<sup>6</sup> Lewisite decontaminants would need to convert the various vesicant compounds to non-vesicant compounds.

HD ( $C_4H_8SCl_2$ ) degradation by oxidation can yield a stable by-product with vesicant properties, bis (beta-chloroethyl) sulfone ( $C_4H_8Cl_2O_2S$ ) (CAS 471-03-4).<sup>7</sup> Munro et al.<sup>6</sup> provides an extensive review of HD degradation products and impurities. Effective decontamination of HD would need to degrade HD without producing this by-product.

### ***1.1 Purpose and Objectives***

The overall purpose of this evaluation was to determine the decontamination efficacies of various, readily-available, liquid-based methods for decontamination of Agent Yellow (including L, HD, and by-products with vesicant properties) from various materials. Specific objectives included:

- Evaluation of the persistence of Agent Yellow on sealed concrete and glass under ambient laboratory conditions;
- Demonstration of efficiency of Agent Yellow extraction using three solvents: acetone, toluene, and hexane;
- Determination of GC/MS method detection limits (MDL) for derL-1 and HD, i.e., bis[2-chloroethyl] sulfide, in a mixture of L and HD using one solvent (hexane). DerL-1 is  $ClCH=CHAs(SC_4H_9)_2$ , the derivatization product of L-1, CVAA, and Lewisite oxide;
- Systematic evaluation of the efficacy of four decontaminants (bleach [full strength], bleach [1:10 dilution], hydrogen peroxide solution [3%], and EasyDecon<sup>®</sup> DF200) for neutralization (conversion to a non-vesicant compound) of L and HD on  $3.5 \times 1.5$  centimeter pieces (coupons) of four materials (sealed concrete, wood flooring, galvanized metal, and glass); and
- Qualitative assessment of coupons for obvious visible damage resulting from application of the decontaminants.

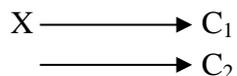
### ***1.2 Test Facility Description***

All testing was performed at the Battelle Hazardous Materials Research Center (HMRC) located on the Battelle site in West Jefferson, Ohio. The HMRC is certified to work with chemical surety material through its Bailment Agreement W911SR-10-H-0001 with the U.S. Department of the Army.

All testing was performed under ambient laboratory conditions. The temperature and relative humidity in the laboratory were not controlled beyond normal heating and air conditioning. The temperature and relative humidity were documented during each day of testing, both prior to and following operations. The temperature in the laboratory during testing ranged from 17.8 degrees Celsius (°C) to 20.3 °C and the relative humidity ranged from 7.5% to 53.5%.

## 2.0 Experimental Methods

A posttest-only control group experimental design was used. In the following representation of the experimental design, “X” is the experimental variable and “C” represents a measurement or observation. The experimental design is represented below in which time (represented by the arrow) passes from left to right:



where coupons are spiked with Agent Yellow and randomly assigned as test coupons or positive control coupons. Decontaminant applied to the test coupons is the experimental variable (symbolized by “X”). Test coupons are decontaminated for a period of time and then extracted and analyzed by GC/MS for derL-1 and HD (Observation 1;  $C_1$ ). Positive control coupons are not preceded by X, i.e., not decontaminated, but are extracted and analyzed for derL-1 and HD (Observation 2;  $C_2$ ) at the same time as the test coupons. The effect of the treatment (efficacy) is reported as the percentage of chemical agent remaining on treated coupons compared to the control coupons:

$$\text{Efficacy} = [(C_2 - C_1) / C_2] \cdot 100\% \quad (1)$$

The higher the efficacy, the greater the effect of the decontamination. If there is no significant difference ( $p > 0.05$ ) between the mean agent remaining on the positive control coupons and the mean agent remaining on the test coupons, no significant efficacy is observed.

In addition to the test and control coupons, laboratory blank coupons (coupons that were neither contaminated with Agent Yellow nor decontaminated) and procedural blank coupons (coupons that were not spiked with Agent Yellow, but were decontaminated along with the test coupons) were extracted and analyzed for derL-1 and HD. To verify the amount of Agent Yellow spiked onto coupons, the same amount as applied to coupons was applied to polytetrafluoroethylene (PTFE) disks and the disks were immediately extracted and analyzed as spike controls.

### 2.1 Chemical Agent and Spiking Coupons

The L and HD (as received from the Army and owned by EPA) without any dilution is referred to as neat L and neat HD. High (>90%) and stable agent purities are normally observed over long periods of time (12 months or greater) following standard Battelle procedures for storage and manipulation of L and HD.

A single batch of Agent Yellow (“neat”) was prepared, sufficient for all testing performed as a mixture of neat L and neat HD without any dilution. The purity of the neat L and neat HD used to prepare Agent Yellow was determined by GC/flame ionization detector (FID). To determine purity, the peak area value of the agent (L-1 or HD) from GC/FID analysis was compared to the

sum of all peaks present in the chromatogram (corrected by a solvent blank analysis result) and calculated as a percentage. The neat L and neat HD sources were found to have purities of 92.2% L-1 (<0.2% L-2; L-3 not detected) and 96.2% HD, respectively, prior to use. Agent Yellow was prepared as a mixture of neat L (63% by weight) and neat HD (37% by weight). Thus, the Agent Yellow was theoretically 58% L-1 and 36% HD by weight. Purity analysis of the Agent Yellow following preparation yielded a value of 95.8%, indicating that L-1 and HD peak areas represented 95.8% of the compounds in the agent.

Neat Agent Yellow was dispensed using a 50 microliter ( $\mu\text{L}$ ) gas-tight syringe (P/N 80920, Hamilton, Reno, NV) equipped with repeating dispenser (P/N PB600-1, Hamilton, Reno, NV) and PTFE needle (P/N 9991326, Integrated Dispensing Solutions, Inc., Agoura Hills, CA). All test and positive control coupons were spiked with 1  $\mu\text{L}$  of neat Agent Yellow. Each  $\mu\text{L}$  of neat Agent Yellow has a nominal mass of 1.6 mg consisting of 1.0 mg of Lewisite and 0.6 mg of HD. The coupons were open to the atmosphere (uncovered in open Petri dishes) within a chemical agent hood during a 30 min weathering period prior to application of decontaminants. Subsequent to the weathering period the decontaminants were applied to the Agent Yellow on the coupons. After an appropriate decontamination reaction time the coupons were transferred into solvent for extraction.

## ***2.2 Test Materials***

Targeted materials included sealed concrete, wood flooring, galvanized metal, and glass (Table 1). Except for concrete, coupons were cut from larger pieces of material to  $3.5 \times 1.5$  centimeters (cm). Concrete coupons were poured into a mold and after setting were coated with sealer. Two materials, glass and wood, were used for persistence testing.

## ***2.3 Description and Application of Decontaminants***

Four decontaminants were evaluated for efficacy against Agent Yellow on coupons:

- Bleach (sodium hypochlorite 5.65% to 6%, #SS290-1, Fisher Scientific)
- Bleach (sodium hypochlorite 5.65% to 6%, #SS290-1, Fisher Scientific) diluted 1:10 with deionized water, (#23-751-610, Fisher Scientific)
- Hydrogen peroxide (3%, # 88597-100ML-F, Fisher Scientific)
- EasyDECON<sup>®</sup> DF200 (DF200, EFT Holdings, Inc.).

**Table 1. Test Materials, Descriptions, Sources, Size, and Preparation**

Material	Description	Manufacturer/ Supplier Name	Coupon Surface Size Length x Width (cm)	Material Preparation
Sealed concrete	Epoxy (Sure Klean® Weather Seal Siloxane PD; PROSOCO, Inc., Lawrence, KS) sealed concrete (5 parts sand, 2 parts concrete); custom preparation	Wysong Concrete, Cincinnati, OH	3.5 × 1.5	Clean with dry air to remove loose dust
Wood flooring material	Pine plywood (bare); thickness 1.0 cm	Lowe's, Columbus, OH	3.5 × 1.5	Clean with dry air to remove loose dust
Galvanized metal ductwork	Industry heating, ventilation, and air conditioning standard; 24 gauge galvanized steel; thickness 0.7 millimeter (mm) (Adept Manufacturing)	Adept Products, Inc., West Jefferson, OH	3.5 × 1.5	Clean with acetone
Glass	Glass (clear window)	Brooks Brothers, West Jefferson, OH	3.5 × 1.5	Clean with dry air to remove loose dust

The decontaminants were applied as a liquid to the test coupons 30 minutes (min) after the Agent Yellow was spiked onto the coupons. The decontaminants were dispensed using a positive displacement pipette (P/N M-250 [50-250 µL] and CP250 tip, Gilson Inc., Middleton, WI). The amount of decontaminant applied to the coupons was 0.06 mL for non-porous materials (sealed concrete, glass, and galvanized metal ductwork) and 0.09 mL for porous wood flooring. These amounts reflect the approximate quantity of liquid decontaminant expected to remain on a coupon of a specific type of material and size after a spray application.<sup>9</sup>

The initial reaction time for the decontaminants was 30 min. The decontamination testing was repeated at a second reaction time (60 min) for combinations of coupons and decontaminant for which the common product of the derivatization of derL-1 or HD was detected after the 30 min reaction time. Application of decontaminant for a reaction time of 30 min followed by a reapplication of the decontaminant for an additional 30 min reaction time was evaluated for bleach (dilute) and hydrogen peroxide (3%) to determine if efficacy was improved.

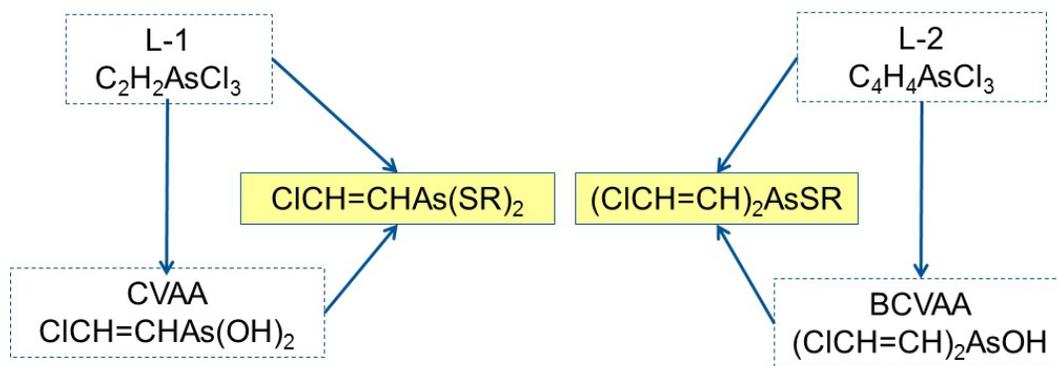
#### **2.4 Extraction of Coupons**

All coupons in the test matrix and the blank coupons were extracted by placing each into a separate 60 milliliter (mL) glass bottle (02-991-701, Fisher Scientific, Pittsburgh, PA) that contained 10 mL of solvent/internal standard (IS), swirled by hand for about 5-10 seconds, and

placing into a sonicator. Extraction bottles were sonicated at 40 to 60 kilohertz for 10 min at ambient laboratory temperature. The IS is included in order to detect instrument drift and loss of analyte in the injection process. The same extraction process was repeated for all coupons used to determine extraction efficiencies, MDL, persistence, and decontamination efficacy. Samples that were not analyzed the same day were stored at or below -20 °C.

## 2.5 Derivatization of L

It is important to determine the presence of L-1, CVAA, and Lewisite oxide post decontamination because these by-products each have harmful vesicant properties, rather than analyzing only for L-1. This determination was accomplished in this work by derivatizing the samples, as shown in Figure 2, and analyzing the samples using gas chromatography (GC)/mass spectrometry (MS) to detect the derivative of L-1 and its hydrolysis by-products (quantitative) (derL-1) and the derivative of L-2 and its hydrolysis by-product (bis(2-chlorovinyl) arsonous acid [BCVAA]) (qualitative) (derL-2). Lewisite oxide, not shown in Figure 2, yields the same product by derivatization as L-1 and CVAA. Derivatizing L by reaction with butanethiol prior to GC/MS analysis also aids detection by creating products that are readily volatilized and with distinctive mass spectral fragmentation. Muir et al. (2005) showed that butanethiol reacted with L-1 and L-2, but did not react with L-3 or HD.<sup>3</sup> L-3 can be detected without derivatization by GC/MS. However, L-3 typically comprises only 1% of American L. No analysis for L-3 was performed as the L used in this work was shown not to contain L-3.<sup>8</sup>



**Figure 2. Derivatization of Lewisite with butanethiol, where R = C<sub>4</sub>H<sub>9</sub>.**

In the presence of water, L-1 is converted to the vesicant CVAA. Derivatization is the preferred approach for detection of both residual L and its vesicant by-product CVAA.<sup>6</sup> Following the method of Muir et al.<sup>3</sup>, 200 μL of 50 milligram (mg) mL<sup>-1</sup> butanethiol were added to 1 mL of each Agent Yellow extract to be analyzed. Each sample/standard received 1% butanethiol per volume. Triethylamine (50 μg) was added to each solution to catalyze the derivatization. Each solution was mixed on a vortex mixer for 10 seconds. After mixing, samples were analyzed the same day by GC/MS or were stored at or below -20 °C until they were thawed for analysis.

## **2.6 Analytical Methods**

Analysis for HD (quantitative) and its vesicant by-product bis(beta-chloroethyl)sulfone (qualitative) was accomplished using GC/MS to identify the sulfone by its mass spectrum.

Quantitative analysis of derL-1 and HD was enabled by preparation of standard solutions of these agents that were analyzed along with test samples. No standard was available for derL-2 or the HD by-product and the results were therefore qualitative, indicating whether or not derL-2 or the by-product is detected.

Blanks, positive control coupons, and decontaminated test coupons were extracted and derivatized according to methods described in Sections 2.4 and 2.5. Aliquots of the sample extracts were analyzed to quantify the amount of derL-1, and HD, and to detect derL-2 and bis (beta-chloroethyl) sulfone, remaining on each coupon using GC/MS (6890 gas chromatograph and 5973 mass selective detector, Agilent Technologies, Santa Clara, CA) operated in the full scan mode for compounds ranging from 40 to 500 atomic mass units (amu). The combined L-1, CVAA, and Lewisite oxide (if present) butanethiol derivatives (derL-1) were detected with quantification ion 164 and qualifier ions 204, and 314. L-2 and BCVAA butanethiol derivatives (derL-2) were detected with quantification ion 164 and qualifier ions 107 and 286. HD was detected with quantification ion 109 and qualifier ions 158, 160 and 63. Bis (beta-chloroethyl) sulfone was detected with quantification ion 63 and qualifier ions 65, 92 and 127. The GC/MS parameters that were used in method demonstration and subsequent decontamination testing are shown in Table 2.

The lowest standard used to establish the calibration curve (quantitation limit) was above, but near, the instrument detection limit of the GC/MS. Samples with results below the lower calibration level are reported as less than the quantitation limit.

**Table 2. Gas Chromatographic/Mass Spectrometry Conditions**

Parameter	Description
Analysis Method	GC/MS
Instrument	Agilent Model 6890 Gas Chromatograph equipped with a 5973 Mass Selective Detector and a Model 7683 Injector with AutoSampler
Data System	MSD ChemStation
Liner Type	4mm Splitless
Column	RTX-5MS, 30.0 meters × 0.25 millimeter, 0.25 micrometer film
Helium Carrier Gas Pressure and Flow	15 pounds per square inch (40 °C) at constant flow (37.7 cm s <sup>-1</sup> )
Temperature Fields	Inlet temperature: 250 °C; Detector temperature: 230 °C
Sample Size	1 µL
Oven Program for Analysis	50 °C (2.0 min), 325 °C (3.0 min) @ 30 °C/min ; total run time 14.5 min
Target Ions, m/z (% relative abundance versus first ion)	derL-1: 164 (quantification ion), 204, 314 derL-2: 164 (quantification ion), 107, 286 HD: 109 (quantification ion), 158, 160, 63 Bis (beta-chloroethyl) sulfone: 63 (quantification ion), 65, 92, 127

## 2.7 Method Development and Demonstration

Method development and demonstration included determining the extraction efficiencies of three solvents; determining the MDL for one solvent that would be used in subsequent testing; and determining the adequacy of extraction alone for quenching decontamination.

### 2.7.1 Extraction Efficiency

Tests were performed in triplicate, as shown in Table 3 to determine Agent Yellow extraction efficiency from four materials (sealed concrete, wood flooring, galvanized metal, and glass) using three solvents (acetone, hexane, and toluene). Neat Agent Yellow (1 µL) was spiked onto the test coupons and immediately extracted. In addition, three spike controls per solvent (a spike of equal amount of Agent Yellow on a PTFE disk followed by immediate extraction) and extraction of a single laboratory blank per material were included in the analysis. Coupons were spiked, extracted, derivatized, and analyzed as described in Sections 2.1, 2.4, 2.5, and 2.6, except that the extractions were immediate, rather than after a 30 min weathering period. Extraction efficiencies were calculated as described in Section 2.11.

**Table 3. DerL-1 and HD Extraction Efficiency Matrix (Repeated for Toluene, Hexane, and Acetone)**

Material	Number of Test Coupons	Number of Spike controls	Number of Laboratory Blank Coupons
Sealed concrete	3	3	1
Wood flooring material	3		1
Galvanized metal ductwork	3		1
Glass	3		1

### 2.7.2 Method Detection Limit

After hexane was selected as the solvent that would be used for extraction in the decontamination investigation, MDL studies for hexane extraction of a mixture of derL-1 and HD on the four materials were performed. Seven coupons of each material type were spiked with a dilute mixture of L and HD in hexane (relative proportions were selected to enable the MDL to be determined for both agents using one solution). The single concentration design estimator recommended by the EPA (40 CFR part 136, Appendix B (1984)) was completed as follows to determine the MDL for derL-1 and HD:

- The coupons were extracted, the extracts derivatized and analyzed for derL-1 and HD using GC/MS as described in Sections 2.4, 2.5, and 2.6.
- The standard deviations of the replicate measurements were calculated.
- The MDL = Student's t-value for n replicates appropriate for a 99% confidence level × standard deviation estimate with n-1.

### 2.7.3 Neutralization of Decontaminant

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 determined the conditions necessary to stop the decontamination reaction (quench) without interfering with the extraction and analysis so that different decontamination reaction times could be evaluated. Two approaches were used. The first approach, shown below, evaluated the hypothesis that hexane extraction alone would be sufficient to quench the reaction. The hypothesis that extraction alone, without additional neutralization, would be sufficient for GC/MS analysis if the amount of derL-1 and HD recovered in Step 1 (decontamination solution present) was each at least 70% of the amount of the respective CWA recovered in Step 2 (no decontamination solution present). Initial method:

1. As a test solution, the amount of decontamination solution to be applied to coupons for decontamination testing (60 microliter [ $\mu\text{L}$ ]) was added (using a positive displacement pipette (P/N M-100 [10-100  $\mu\text{L}$ ] and D-200 [2-200  $\mu\text{L}$ ] tip, Gilson Inc., Middleton, WI)

to a vial containing 10 milliliters (mL) of hexane, IS (10 µg/mL naphthalene-d<sub>8</sub> [176044-1G, Isotec [Sigma-Aldrich], St Louis, MO]), and 1 µL of neat Agent Yellow, shaken for 15 seconds, and allowed to stand for 10 min.

2. As a positive control, water (60 µL) was added to a vial containing 10 mL of solvent and IS (naphthalene-d<sub>8</sub>) and 1 µL of neat Agent Yellow, the vial was shaken for 15 seconds and allowed to stand for 10 min.
3. The extracts from Steps 1 and 2 were stored for at least 24 hours at or below -20 degrees Celsius (°C) and then analyzed using GC/MS.

Because the initial approach did not confirm the hypothesis, a second approach was employed and the addition of sodium thiosulfate as a quench was evaluated. Wood was selected as the most challenging coupon type for the quench evaluation because more decontaminant was applied during testing for wood (a porous material) than for other nonporous coupon materials (90 µL versus 60 µL). Further, wood is the most absorptive of the coupon materials. In a porous material, it may be more difficult for the quench to physically contact (and neutralize) the decontaminant. A second set of coupons was used to test the effectiveness of the quench for the worst case scenario (180 µL total decontaminant in the reapplication test). The decontaminant quench retest procedure follows:

- Positive control coupons were prepared by derivatizing L and HD (5.0 µg/mL final concentration of each) in hexane with IS.
- A candidate decontamination solution (90 µL aliquot) was applied to a wood coupon and two 90 µL aliquots of the decontaminant were applied to a second wood coupon. Both wood coupons were then allowed to sit for 30 min. (This was repeated for each of the four types of decontaminants.)
- Thirty min after the decontaminant was applied, the coupons were individually extracted in hexane with 10 µg/mL naphthalene-d<sub>8</sub> IS as described in Section 2.4.
- Eight 1-mL aliquots of the each wood coupon extracts (coupon spiked with 90 µL and coupon spiked with 180 µL) were placed in vials.
- Quench (0.5 mL of 3M sodium thiosulfate) was added to four of the eight vials.
- 10 µL of a 500 µg/mL solution of HD and L were added to three of the four vials of each condition (quench added and no quench added; final concentration of 5 µg/mL HD and L in each vial).
- All vials were vortexed for 10 seconds.
- Samples from each vial were aliquoted, derivatized and analyzed immediately, and then analyzed again after being stored for 24 hours at room temperature.

**Table 4. Quench Test Matrix**

Additives	Wood (90 µL decontaminant)	Wood (180 µL decontaminant)
No quench addition	Negative Control (no thiosulfate, L, or HD)	Negative Control (no thiosulfate, L, or HD)
No quench addition	Spike 10 µL of a 500 µg/mL solution of HD and L in hexane and vortex (5µg/mL final concentration (triplicate test))	Spike 10 µL of a 500 µg/mL solution of HD and L in hexane and vortex (5µg/mL final concentration (triplicate test))
0.5 mL 3M sodium thiosulfate	Negative Control (no L or HD)	Negative Control (no L or HD)
0.5 mL 3M sodium thiosulfate	Spike 10 µL of a 500 µg/mL solution of HD and L in hexane and vortex (5µg/mL final concentration [triplicate test])	Spike 10 µL of a 500 µg/mL solution of HD and L in hexane and vortex (5µg/mL final concentration [triplicate test])

#### 2.7.4 Confirm Derivatization Does Not Interfere with HD Analysis

Tests were performed, summarized in Table 5, to confirm that the addition of triethylamine or derivatization mixture (adding triethylamine and butanethiol), as described in Section 2.5, does not interfere with the HD analysis. To perform the test, 1 µL of neat HD was spiked into 10 mL of acetone. One-milliliter aliquots were placed into each of nine GC vials. IS consistent with testing was added to three vials; triethylamine was added to three vials; and triethylamine and butanethiol (consistent with the derivatization in Section 2.5) were added to three vials. Samples were taken from each vial and analyzed for IS and HD as described in Section 2.6. Results were considered acceptable if differences among the means of the treatments were within 30%.

**Table 5. Matrix to Test Impact of Derivatization on HD and IS Analysis**

Vial Containing HD and IS	Additive
Negative Control (triplicate test)	No addition
Triethylamine Enhancement Test (triplicate test)	Triethylamine
Derivatization Test (triplicate test)	Triethylamine + butanethiol

## 2.8 Persistence Testing

Persistence was evaluated at ambient conditions in the laboratory for Agent Yellow spiked onto sealed concrete and glass as described in Section 2.1. These tests were included to assess the natural attenuation of the agent on materials over time (up to 18 hours (hr) post contamination). The persistence testing generally followed the same set-up and procedures (spike, extraction, derivatization, and analysis) used for the decontamination efficacy testing with two exceptions: no decontaminant was applied to any coupon, and coupons were covered (lids were placed on the Petri dishes holding the coupons) during the Agent Yellow residence time. The temperature and relative humidity during the persistence testing were in the range of 18.5 °C to 20.6 °C and 8.3% to 13.7%, respectively.

The test matrix for Agent Yellow persistence is shown in Table 6. On the first day of testing, coupons were extracted immediately after spiking (“0” hours) and at one, two, and four hours after the coupons were spiked. On a second day of persistence testing, coupons were spiked with Agent Yellow and extracted at 0, 6 and 18 hours after spiking. All coupons from the persistence tests were extracted, the L derivatized, and the samples analyzed using GC/MS to quantify residual derL-1 and HD. In addition to the test coupons, one laboratory blank coupon of each material type was extracted and analyzed along with the test coupons on each day of persistence testing.

**Table 6. Persistence Test Matrix**

Lab Day	Coupon Type	Time Intervals, hours			
		0	1	2	4
1	Sealed Concrete	3 coupons	3 coupons	3 coupons	3 coupons
	Glass	3 coupons	3 coupons	3 coupons	3 coupons
Lab Day	Coupon Type	Time Intervals, hours			
		0	6	18	
2	Sealed Concrete	3 coupons	3 coupons	3 coupons	
	Glass	3 coupons	3 coupons	3 coupons	

## 2.9 Decontamination Testing

The Agent Yellow decontamination test matrix is shown in Table 7. For each combination of time, material and decontamination method, five test coupons (spiked with neat Agent Yellow and decontaminated), three positive control coupons (spiked with neat Agent Yellow, not decontaminated) and two procedural control coupons (not spiked with Agent Yellow, decontaminated) were included. Coupons were spiked, extracted, derivatized, and analyzed as

described in Sections 2.1, 2.4, 2.5, and 2.6. One blank (negative control) coupon of each material type was extracted and analyzed each day of testing. The two reaction times that were evaluated were 30 min and 60 min. In addition, sequential 30 min reapplication of the decontaminant was evaluated for bleach (dilute) and hydrogen peroxide (3%). Quantitative GC/MS analysis was used for derL-1 and HD; qualitative GC/MS analysis was used to detect derL-2 and bis (beta-chloroethyl) sulfone.

As shown in the last row in Table 7, decontamination of neat HD on two materials and with two decontaminants was also evaluated. The materials selected were glass and wood and the decontaminants applied were bleach (dilute) and hydrogen peroxide (3%).

**Table 7. Agent Yellow Decontamination Test Matrix**

Decontaminant	Reaction Time	Coupons for each Combination of Decontaminant and Material
Repeated for each of the four decontaminants and all four coupon materials	30 min	5 test coupons; 3 positive control coupons, two procedural blank coupons; one blank coupon included for each day of testing
Repeated for each of the four decontaminants and all four coupon materials, except full-strength bleach on glass and sealed concrete were excluded*	60 min	5 test coupons; 3 positive control coupons, two procedural blank coupons; one blank coupon included for each day of testing
Repeated for bleach (dilute) and hydrogen peroxide (3%) on all four coupons types	30 min; then reapplication after the first 30 min of reaction time; total reaction time 60 min	5 test coupons; 3 positive control coupons, two procedural blank coupons; one blank coupon included for each day of testing
Glass and wood were spiked with 1 $\mu$ L of neat HD and decontaminated with bleach (dilute) and hydrogen peroxide (3%)	30 min	5 test coupons; 3 positive control coupons, two procedural blank coupons; one blank coupon included for each day of testing

\*No detectable derL-1 or HD remained on glass and sealed concrete after the 30 min decontamination with full-strength bleach, so these material and decontaminant combinations were excluded from testing with a 60 min reaction time.

### ***2.10 Observation of Surface Damage***

The impact of decontamination on the building materials was assessed visually. Independent of the agent work, one procedural blank of each material type was rinsed with deionized water and allowed to dry. The procedural blank was visually inspected and compared to laboratory blank coupons not exposed to the decontamination treatment to look for obvious changes in color, reflectivity, or apparent roughness of the coupon surfaces. Observations were documented and photographs of pre- and post-decontamination coupons were taken.

### **2.11 Extraction Efficiency**

Extraction efficiency was calculated using a series of equations as follows. Chemical agent concentration in a coupon extract or solution sample was determined by the GC analysis software (ChemStation) using the obtained sample area value.

GC concentration results ( $\mu\text{g/mL}$ ) are converted to total mass by multiplying by extract volume:

$$M_m = C \times E_v \quad (2)$$

where:

$M_m$  = measured mass of chemical agent (microgram[s],  $\mu\text{g}$ )

$C$  = GC concentration ( $\mu\text{g/mL}$ ), see Equation 1

$E_v$  = volume of extract (mL).

Extraction efficiency was then defined as:

$$\text{Extraction Efficiency} = \left( \frac{M_m \text{ of Chemical Agent on Material}}{M_m \text{ of Chemical Agent on PTFE}} \right) \times 100\% \quad (3)$$

where:

$M_m$  = measured mass of chemical agent ( $\mu\text{g}$ )

“Material” = coupons of materials used in the evaluation that are spiked with chemical agent, extracted, and analyzed

“PTFE” = PTFE disks used in the evaluation as spike controls that are spiked with chemical agent, extracted, and analyzed

Extraction efficiency = percent recovery of chemical agent from coupons.

### **2.12 Decontamination Efficacy**

Decontamination efficacy was determined by measuring the amount of residual derL-1 and HD on test coupons and comparing with positive control coupons (spiked with Agent Yellow, not decontaminated) analyzed after the same elapsed time after spiking as the test coupons, i.e., 30 min weathering time plus reaction time. For derL-1 and HD, efficacy in percent was calculated for each individual test coupon as shown in Equation 4. The primary efficacy results from the coupon testing are provided in a matrix table in which each entry shows the mean and percent relative standard deviation of efficacy results for derL-1 and HD on each of the materials.

$$E = [(C_o - C_f)/C_o] \cdot 100\% \quad (4)$$

where:

E = efficacy

C<sub>o</sub> = mean concentration of agent without decontamination (determined from the positive control coupons of each material)

C<sub>f</sub> = concentration on a test coupon with decontamination.

A Student's t-test was used to compare the amount of derL-1 and HD recovered from test coupons to the amount of agent recovered from positive control coupons; p-values  $\leq 0.05$  were considered statistically significant. A separate t-test was performed for each material and for both derL-1 and HD. If there is no significant difference ( $p > 0.05$ ) between the mean agent remaining on the positive control coupons and the mean agent remaining on the test coupons, no significant efficacy is observed.

Qualitative analysis of derL-2 was reported as the ratios of the areas of the chromatograph peaks for derL-2 to derL-1. The presence of HD by-product was also noted.

### 3.0 Test Results

#### 3.1 Method Development and Demonstration Results

##### 3.1.1 Extraction Efficiency

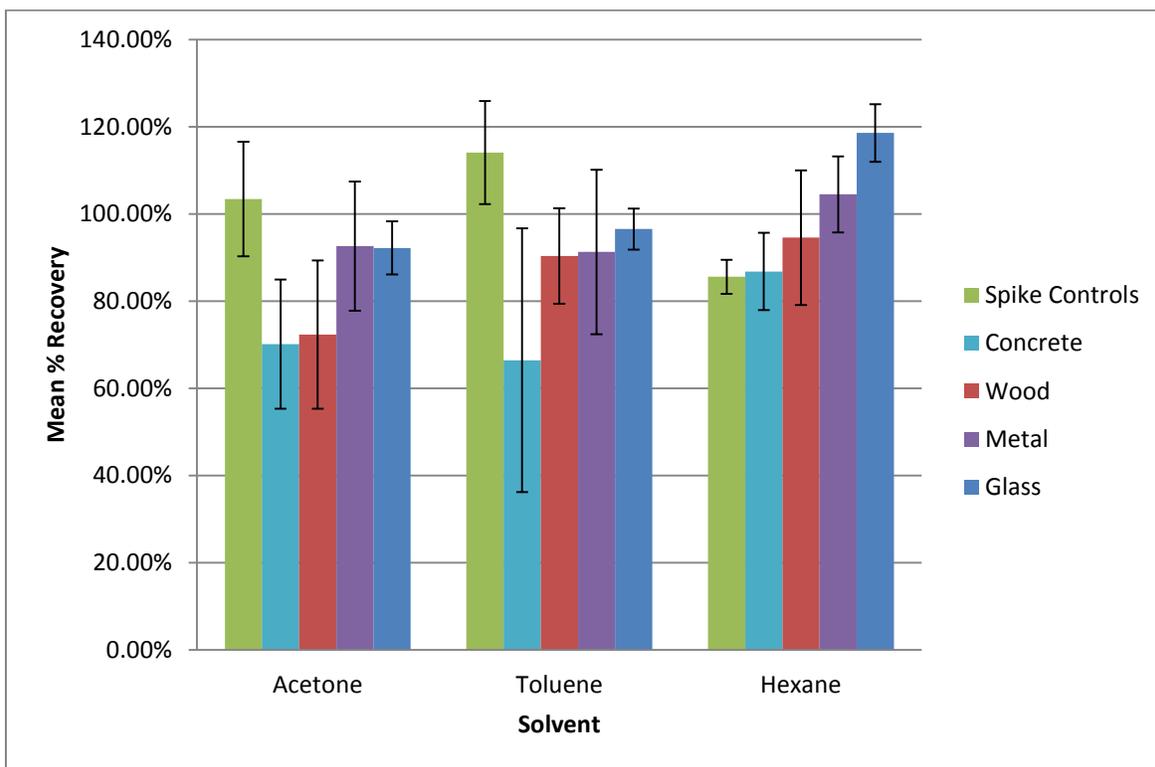
The results of extraction efficiency testing for Agent Yellow with acetone, hexane, and toluene are shown in Table 8. Recoveries of derL-1 for the various solvent material combinations ranged from 66% to 119%. In all cases the percent relative standard deviation was 19% or lower except for derL-1 recovery from concrete using toluene; 1 of 3 concrete samples extracted with toluene showed a 44% recovery while the other two samples showed recoveries >70%. Recoveries of HD for the various solvent material combinations ranged from 78% to 122%. In all cases the percent relative standard deviation (RSD) was at or below 15%. Note that extraction efficiencies shown for PTFE spike compare the recovery from the PTFE disk to the theoretical mass applied. The efficacy for materials used in testing compare the recoveries from materials to recoveries from the PTFE disk (consistent with Equation 3).

**Table 8. DerL-1 and HD Extraction Efficiencies with Various Solvents**

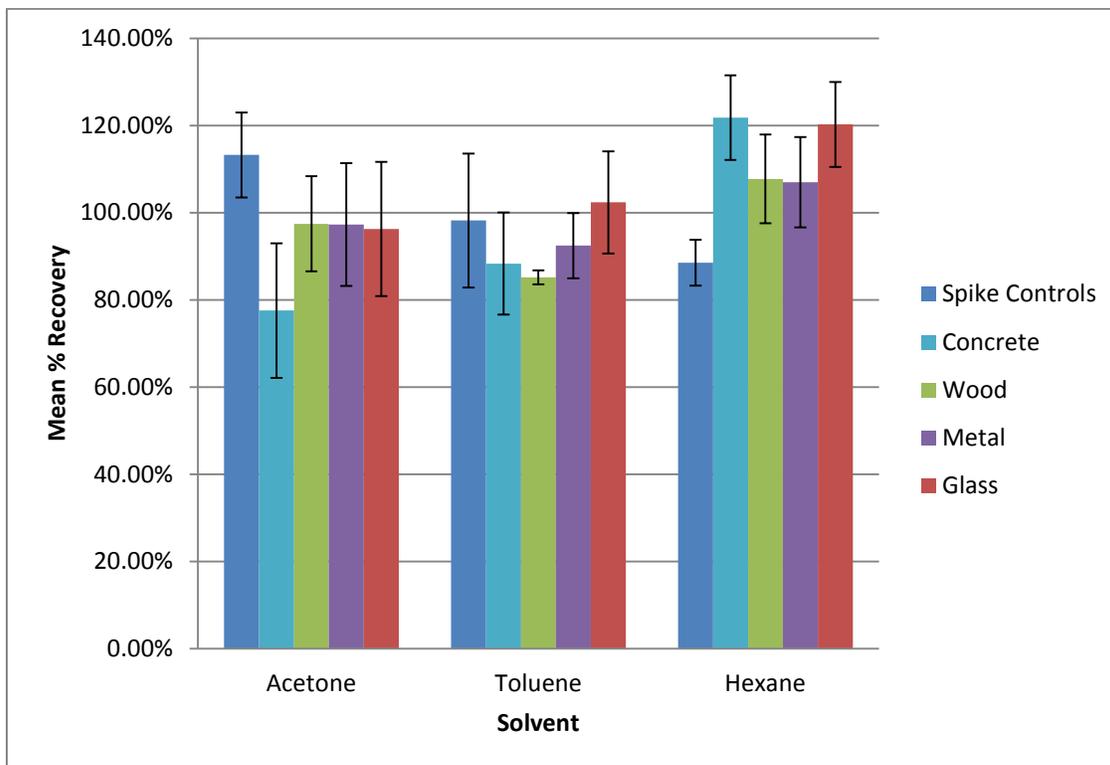
Solvent	Coupon Material	derL-1 Mean, $\mu\text{g}$	derL-1 %RSD	derL-1 Extraction Efficiency, %	HD Mean, $\mu\text{g}$	HD %RSD	HD Extraction Efficiency, %
Acetone	PTFE Spike	1200	13	100*	680	10	110*
	Concrete	750	15	70	530	15	78
	Wood	770	17	72	670	11	97
	Metal	980	15	93	660	14	97
	Glass	980	6	92	660	3	96
Hexane	Spike	880	4	86*	530	5	89*
	Concrete	760	9	87	650	10	120
	Wood	830	15	95	580	10	110
	Metal	920	9	100	570	10	110
	Glass	1000	7	120	640	8	120
Toluene	Spike	1200	12	110*	590	15	98*
	Concrete	780	30	66	520	12	88
	Wood	1100	11	90	500	2	85
	Metal	1700	19	91	550	8	92
	Glass	1100	5	97	600	10	100

\*Compared to the theoretical mass of agent applied to the disk of 1.0 mg of Lewisite and 0.6 mg of HD.

Figures 3 and 4 provide graphs showing a comparison of recovery efficiencies by material type. Recoveries were generally lower for wood and concrete than for glass and metal for all three solvents. Recoveries were at least 85% from all materials (including the PTFE disks for spike controls) using hexane. Based on these data, hexane was selected as the extraction solvent for the decontamination testing.



**Figure 3. DerL-1 mean recoveries by solvent and material (error bars show % relative standard deviation).**



**Figure 4. HD mean recoveries by solvent and material (error bars show % relative standard deviation).**

### 3.1.2 Method Detection Limit

MDL studies for hexane extraction of a mixture of derL-1 and HD are summarized in Table 9. For  $n = 7$  coupons,  $t(n-1, 1-\alpha=0.99) = 2.998$ . Therefore:

$$\text{MDL} = 2.998 \times \text{standard deviation estimate with } n-1. \quad (5)$$

For both derL-1 and for HD in hexane, MDL was lowest for wood (0.3 and 0.4  $\mu\text{g}/\text{coupon}$ , respectively). For derL-1, the highest MDL was for sealed concrete (0.9  $\mu\text{g}/\text{coupon}$ ). For HD, the highest MDL was for metal (1.2  $\mu\text{g}/\text{coupon}$ ).

**Table 9. MDL for derL-1 and HD in Hexane**

Sample Source	Target	SD, µg/coupon	MDL, µg/coupon
Extracted from Sealed Concrete	derL-1	0.3	0.9
	HD	0.2	0.5
Extracted from Wood	derL-1	0.1	0.3
	HD	0.1	0.4
Extracted from Metal	derL-1	0.1	0.4
	HD	0.4	1.2
Extracted from Glass	derL-1	0.2	0.5
	HD	0.2	0.5

### 3.1.3 Neutralization of the Decontaminant

The initial approach to evaluate the need for neutralization yielded anomalous results, e.g., HD recovery in 5% bleach extract was greater than 200% higher than HD recovery from the positive control condition. The results also suggested that extraction alone might not be a sufficient quench for hydrogen peroxide (3%) or for DF200. A revised test procedure, described in Section 2.7.3, was used to both reevaluate the sufficiency of extraction alone to quench decontamination and to evaluate the addition of sodium thiosulfate as a quench.

The results of the revised test (Table 10) showed recoveries of derL-1 and HD from hexane containing decontaminants was 82% or more of the recoveries from hexane in the absence of decontaminants. Similar results were observed when the extracts were stored for 24 hours at room temperature and analyzed (data not shown). The addition of sodium thiosulfate was not effective as a quench. Little or no derL-1 was found in extracts to which thiosulfate had been added suggesting that thiosulfate degraded L. Thiosulfate did not impact the amount of HD recovered from extracts. These results indicated that extraction alone was sufficient to terminate decontamination.

**Table 10. Hexane Extraction and Thiosulfate Quench Test Results**

Additives to Hexane Containing L and HD	Bleach (full strength), % Recovery vs. Positive control coupons		Bleach (dilute) % Recovery vs. Positive control coupons		Hydrogen Peroxide (3%)% Recovery vs. Positive control coupons		DF200 % Recovery vs. Positive control coupons	
	derL-1	HD	derL-1	HD	derL-1	HD	derL-1	HD
90 µL decontaminant	91	88	89	87	85	86	100	92
90 µL decontaminant plus thiosulfate	<37	86	<37	86	<37	87	<47	96
180 µL decontaminant	90	87	89	87	82	85	110	100
180 µL decontaminant plus thiosulfate	<37	88	<40	87	<37	84	<40	100

Efficacies shown as “<” had at least one and in most cases all coupon extracts that were below the quantitation limit; quantitation limit (2.0 µg/mL) substituted into calculation of the mean and reported as “<”.

#### 3.1.4 Confirm Derivatization Does Not Interfere with HD Analysis

Tests were performed, summarized in Table 11, to confirm that the addition of triethylamine or derivatization mixture (adding triethylamine and butanethiol), as described in Section 2.5, does not interfere with the HD analysis. To perform the test, 1 µL of neat HD was spiked into 10 mL of acetone. One-milliliter aliquots were placed into each of nine GC vials. IS consistent with testing was added to three vials; triethylamine and IS were added to three vials; and triethylamine, IS and butanethiol (consistent with the derivatization in Section 2.5) were added to three vials. Samples were taken from each vial and analyzed for IS and HD as described in Section 2.6. The means of the results from the treatments differed by less than 8%. The results with triethylamine or with triethylamine and butanethiol added were not significantly different from the controls.

**Table 11. Matrix to Test Impact of Derivatization on HD and IS Analysis**

Vial Containing HD (1 $\mu$ L) in 10 mL acetone and IS (naphthalene-d <sub>8</sub> [10 $\mu$ g/mL]) plus:	Mean HD, $\mu$ g/mL (%RSD)	% HD Difference Compared to Control	IS Area (%RSD)	% IS Difference Compared to Control (Student's t-test, n=3)
200 $\mu$ L naphthalene-d <sub>8</sub> 10 $\mu$ g/mL (Control)	100 (9.3)	--	419,857 (9)	--
200 $\mu$ L naphthalene-d <sub>8</sub> 10 $\mu$ g/mL and 250 $\mu$ g/mL triethylamine	94 (13)	7.4% (p=0.73)	389,573 (15)	7.2% (p=0.63)
200 $\mu$ L naphthalene-d <sub>8</sub> 10 $\mu$ g/mL and 250 $\mu$ g/mL triethylamine and 1 mg/mL butanethiol	94 (10.8)	7.8% (p=0.34)	386,282 (11)	8.0% (p=0.4)

### 3.2 Persistence Testing Results

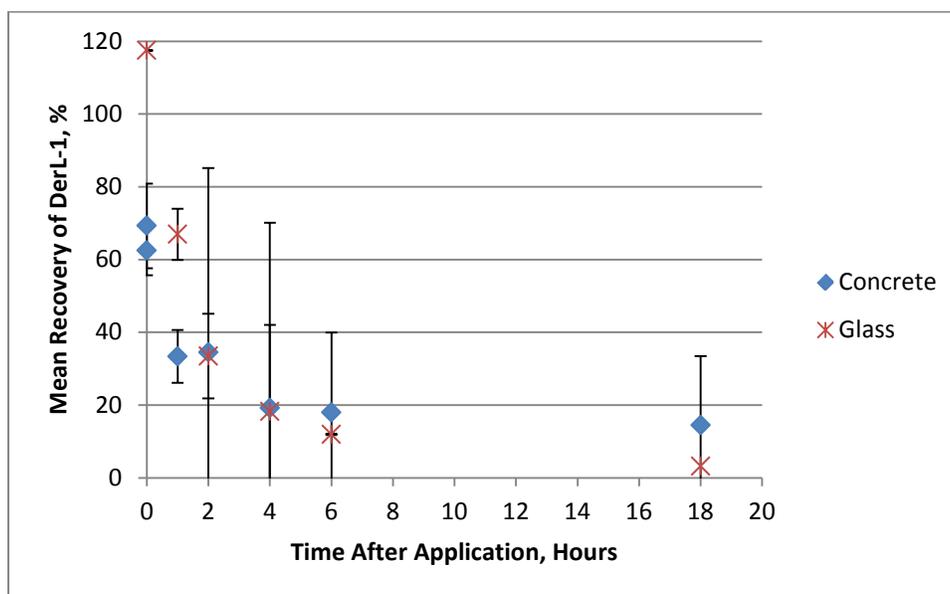
Persistence was evaluated on sealed concrete and glass as described in Section 2.8. The test results for Agent Yellow persistence are shown in Table 12. The percentages are in comparison to recoveries from spike control disks (Agent Yellow spiked onto PTFE disks). Extraction efficiencies are higher from glass than from the PTFE disks, resulting in time zero recoveries that are greater than 100%. Persistence testing occurred on two separate days. Zero hour controls were included on both days and results are similar. For derL-1, mean zero hour recoveries (extracted immediately after spiking) were 62% on Day 1 and 69% on Day 2 for concrete. Shown in Figure 5, derL-1 recoveries were somewhat higher from glass than from concrete during the first hour, but were very similar at and after two hours. Recoveries of derL-1 from both materials were about 20% after four hours. Shown in Figure 6, HD recoveries from concrete parallels that of glass, but somewhat more HD is recovered from glass than from concrete during the first six hours. Recoveries of HD from concrete and glass were minimal or non-detected at 18 hours.

**Table 12. Persistence Testing Results**

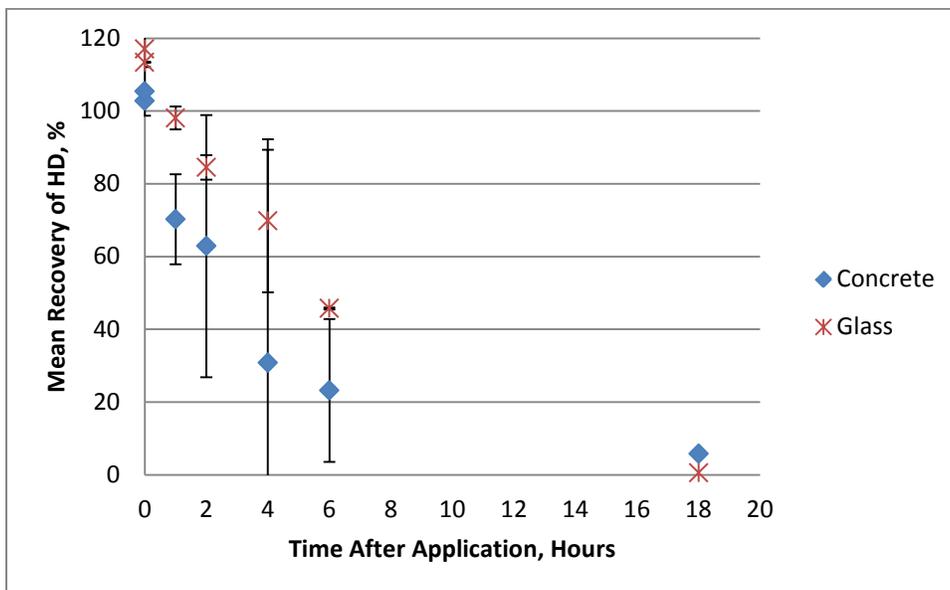
Agent and Test Day	Hours	Mean % of Spike controls	% RSD	Mean % of Spike controls	% RSD
Concrete			Glass		
DerL-1 (Day 1)	0	62	7	130	2
	1	33	7	67	7
	2	34	51	33	12
	4	19	51	18	24
DerL-1 (Day 2)	0	69	12	120	3
	6	18	22	12	18
	18	14	19	<3†	NA†
HD (Day 1)	0	100	1	120	4
	1	70	12	98	3
	2	63	36	85	3
	4	31	62	70	20
HD (Day 2)	0	110	7	110	3
	6	23	20	46	24
	18	<6†	NA†	<2*	NA*

\*Values below the quantitation limit, <20 µg/coupon.

† Some values below quantitation limit.

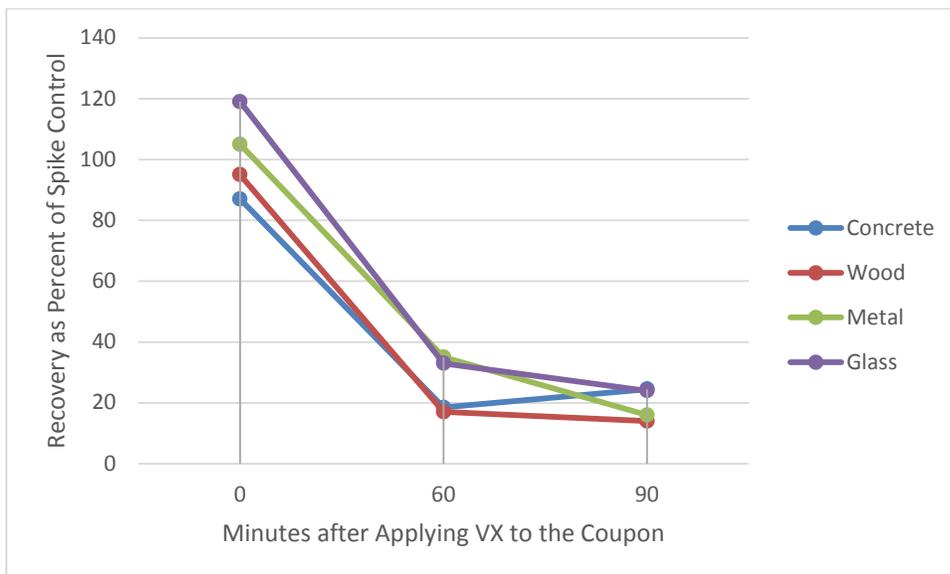


**Figure 5. Persistence of derL-1 on concrete and glass over time (error bars show % relative standard deviation).**

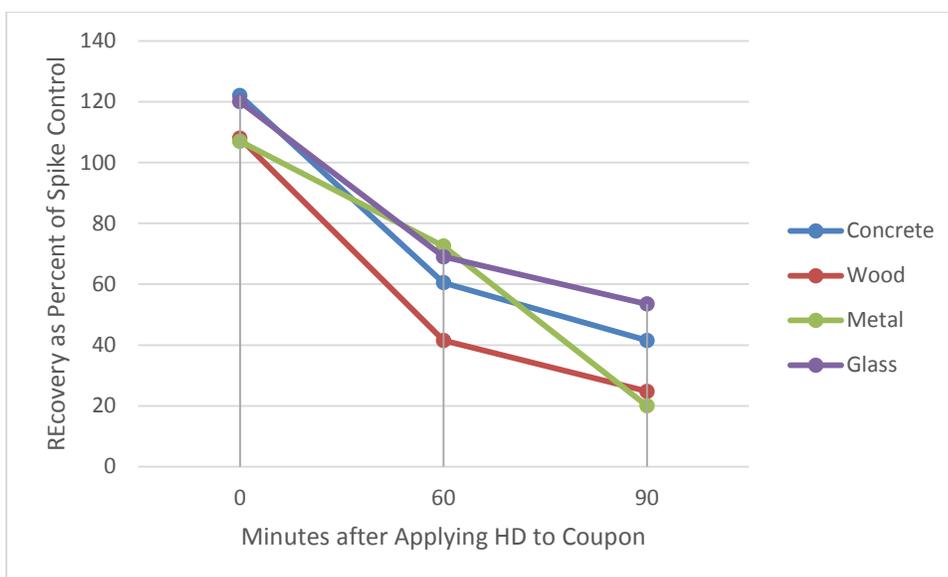


**Figure 6. Persistence of HD on concrete and glass over time (error bars show % relative standard deviation).**

During decontamination testing, the amount of VX and HD remaining on the coupons further demonstrates persistence. Figures 7 and 8 show the average recoveries (compared to the spike controls) from the extraction efficiency testing (with hexane) and the recoveries from positive control coupons (compared to the spike controls) from all of the hydrogen peroxide (3%) and bleach (dilute) testing. Persistence on wood and metal appears to follow a similar trend to persistence on glass and concrete.



**Figure 7. Average percent recovery of derL-1 from positive control coupons compared to spike controls (100%) from all bleach (dilute) and hydrogen peroxide (3%) decontamination tests.**



**Figure 8. Average percent recovery of HD from positive control coupons compared to spike controls (100%) from all bleach (dilute) and hydrogen peroxide (3%) decontamination tests.**

### ***3.3 Decontamination Testing Results***

The results of decontamination testing after 30 min and 60 min reaction times with bleach (full strength), bleach (dilute), hydrogen peroxide (3%), and DF200 are shown in Tables 13, 14, 15, and 16, respectively. Sequential 30 min reapplication of the decontaminant was evaluated for bleach (dilute) and hydrogen peroxide (3%). Quantitative GC/MS analysis was used to analyze for derL-1 and HD. Qualitative GC/MS analysis was used to detect derL-2 and bis (beta-chloroethyl) sulfone. Test results were defined as “efficacious” when the amount of agent recovered from the test coupons was below the quantification limit. Efficacy results were deemed “moderately efficacious” when the mean efficacy was at least 60% with quantifiable amounts remaining on the test coupons. “Low efficacy” was defined for test results where the efficacy values were less than 60% and amounts recovered from the test coupons were above the quantification limit.

For all testing, no derL-1 or HD was measured on any of the laboratory blanks or procedural control blanks. On some procedural blanks small peak areas consistent with derL-1 or HD were sometimes observed but were below the quantitation limits.

### 3.3.1 Efficacy Results Using Bleach (Full Strength)

Bleach (full strength) was efficacious against the L component of Agent Yellow decontaminated from concrete, metal, and glass, as determined by measuring the decrease in derL-1. Efficacies, shown in Table 13, ranged from >79 (metal) to >94 (glass) after a 30 min reaction time. Recoveries from all coupon materials were at or below the limits of detection for derL-1 after a 30 min reaction time.

Bleach (full strength) bleach was generally efficacious against the HD component of Agent Yellow decontaminated from all four materials. Efficacy was material dependent with the lowest efficacies after 30 min reaction time (37%) observed on wood and metal. The highest efficacies after a 30 min reaction time (>94%) were observed on concrete and glass. Increasing the bleach (full strength) reaction time from 30 min to 60 min did not significantly improve removal of HD or corresponding efficacies. (In these results and subsequent results a negative mean percent efficacy is sometimes reported. This indicates that the amount of chemical agent recovered from the test coupons after decontamination was greater than the amount of chemical agent recovered from the positive control coupons. However, in no case was this figure significant. This should be interpreted as no significant difference between agent recovered from test and positive control coupons.)

The mass of HD recovered from the positive control metal coupons was unexpectedly low in the 30 min testing. The reason for this result is not known, but was observed again on metal during testing with bleach (dilute) and hydrogen peroxide (3%).

**Table 13. Bleach (Full Strength) Efficacy Results**

Analyte	Reaction Time, min	Mean Positive Control Coupons Total Mass of derL-1 or HD Recovered, µg (% RSD)	Mean Test Coupon Total Mass of derL-1 or HD Recovered, µg (% RSD)	Mean% Efficacy (p-value)
<b>Concrete</b>				
derL-1	30	270 (50)	<20 (*)	>92 (<0.05)
HD	30	330 (38)	<20 (*)	>94 (<0.05)
<b>Wood</b>				
derL-1	30	140 (62)	21 (9)	85 (0.07)
	60	99 (8)	55 (53)	45 (<0.05)
HD	30	210 (22)	140 (25)	37 (<0.05)
	60	120 (19)	130 (29)	-1.5 (0.47)
<b>Metal</b>				
derL-1	30	94 (33)	<20 (*)	>79 (<0.05)
	60	180 (53)	22 (20)	88 (0.05)
HD	30	32 (32)	<20 (*)	>37 (<0.05)
	60	260 (57)	36 (87)	86 (<0.05)
<b>Glass</b>				
derL-1	30	320 (23)	<20 (*)	>94 (<0.05)
HD	30	410 (21)	<20 (*)	>95 (<0.05)

\*Not calculated because one or more values are below the quantitation limit, <20 µg/coupon).

### 3.3.2 Efficacy Results Using Bleach (Dilute)

Bleach (dilute), shown in Table 14, was moderately to fully efficacious (72% to 94%) against the L component of Agent Yellow decontaminated from concrete, metal, and glass, as determined by measuring the decrease in derL-1. Wood exhibited a lower efficacy, but little derL-1 was recovered from wood after treatment or from positive control coupons. Low efficacy was observed for dilute bleach (16% to 35%) against the HD component of Agent Yellow decontaminated from concrete, wood, metal, and glass with a 30 min reaction time. Efficacy was material dependent with the lowest efficacies observed on wood and metal.

**Table 14. Bleach (Dilute) Efficacy Results**

Analyte	Reaction Time, min	Mean Positive control coupons Total Mass of derL-1 or HD Recovered, µg (% RSD)	Mean Test Coupon Total Mass of derL-1 or HD Recovered, µg (% RSD)	Mean% Efficacy (p-value)
<b>Concrete</b>				
derL-1	30	160 (63)	<20 (*)	>87 (<0.05)
	60	140 (25)	<20 (*)	>86 (<0.05)
	30 + 30	330 (20)	<20 (*)	>94 (<0.05)
HD	30	350 (55)	230 (40)	33 (0.14)
	60	160 (31)	110 (51)	30 (0.14)
	30 + 30	410 (11)	160 (42)	62 (<0.05)
<b>Wood</b>				
derL-1	30	170 (23)	<59 (*)	>66 (<0.05)
	60	150 (21)	<41 (*)	>72 (<0.05)
	30 + 30	140 (24)	<24 (*)	>83 (<0.05)
HD	30	260 (14)	220 (40)	16 (0.24)
	60	170 (26)	140 (37)	18 (0.21)
	30 + 30	160 (19)	120 (61)	27 (0.2)
<b>Metal</b>				
derL-1	30	350 (6.6)	<21 (*)	>94 (<0.05)
	60	160 (24)	<38 (*)	>76 (<0.05)
	30 + 30	120 (14)	<21 (*)	>83 (<0.05)
HD	30	420 (12)	340 (28)	19 (0.10)
	60	260 (27)	170 (4)	35 (0.08)
	30 + 30	43 (23)	120 (27)	-180 (<0.05)
<b>Glass</b>				
derL-1	30	300 (21)	41 (70)	86 (<0.05)
	60	290 (6.5)	80 (34)	72 (<0.05)
	30 + 30	230 (5.7)	<20 (*)	>91 (<0.05)
HD	30	510 (10)	340 (15)	35 (<0.05)
	60	460 (3.0)	180 (53)	62 (<0.05)
	30 + 30	380 (6.0)	170 (30)	54 (<0.05)

\*Not calculated because one or more values are below the quantitation limit, <20 µg/coupon).

Repeating the application of the dilute bleach generally appeared to increase efficacy more than just increasing the reaction time. However, this does not always hold for HD, e.g., HD on glass and metal.

Lower amounts of HD were recovered from all coupon types after reapplication of bleach (dilute) compared to the single 30 min application. HD recovery results after reapplication of bleach (dilute) were similar to the 60 min reaction time. The mass recovered from the positive control metal coupons was unexpectedly low in the 30 min reapplication testing (30 + 30). In

spite of low recoveries from metal after decontamination, efficacy was not observed because of the unexpectedly low recoveries from the positive control coupons, i.e., there was little difference between the recoveries from positive control or test metal coupons. Rust coloring on the metal where the Agent Yellow was applied suggests corrosion may be related to the low recoveries from the positive control coupons.

An additional decontamination test with bleach (dilute) was performed by spiking wood and glass coupons with 1 µl of neat HD (no L) and repeating the decontamination test with a 30 min reaction time. Results are shown in Table 15. The efficacies were 49% on wood and 30% on glass. These efficacy values are similar to those observed for the HD component of Agent yellow, namely, 16% on wood and 35% on glass.

**Table 15. Decontamination of Neat HD using Bleach (dilute) with a 30 min Reaction Time**

Analyte	Reaction Time, min	Mean Positive control coupons Total Mass of derL-1 or HD Recovered, µg (% RSD)	Mean Test Coupon Total Mass of derL-1 or HD Recovered, µg (% RSD)	Mean% Efficacy (p-value)
<b>Wood</b>				
HD	30	650 (41)	330 (44)	49 (<0.05)
<b>Glass</b>				
HD	30	1200 (5.6)	830 (17)	30 (<0.05)

### 3.3.3 Efficacy Results Using Hydrogen Peroxide (3%)

Hydrogen peroxide (3%) results are shown in Table 16. Hydrogen peroxide (3%) was efficacious (83% to 93%) against the L component of Agent Yellow decontaminated from all four materials, as determined by measuring the decrease in derL-1 after a 30 min reaction time. In all cases, after decontamination with hydrogen peroxide (3%) derL-1 extracted was below the limit of detection.

Hydrogen peroxide (3%) was low to moderately efficacious (19% to 80%) against the HD component of Agent Yellow decontaminated from all four materials after a 30 min reaction time. Efficacy was material dependent with a moderate efficacy observed only on wood (80%); other efficacies were below 60%; at 60 min recoveries were below the quantitation limits for one to all five of the wood and metal coupons.

**Table 16. Hydrogen Peroxide (3%) Efficacy Results**

Analyte	Reaction Time, min	Mean Positive control coupons Total Mass of derL-1 or HD Recovered, µg (% RSD)	Mean Test Coupon Total Mass of derL-1 or HD Recovered, µg (% RSD)	Mean% Efficacy (p-value)
<b>Concrete</b>				
derL-1	30	150 (12)	<20 (*)	>86 (<0.05)
	60	180 (59)	<20 (*)	>89 (<0.05)
	30 + 30	390 (42)	<20 (*)	>95 (<0.05)
HD	30	310 (24)	260 (15)	19 (0.09)
	60	230 (46)	83 (68)	63 (<0.05)
	30 + 30	370 (26)	140 (34)	61 (<0.05)
<b>Wood</b>				
derL-1	30	120 (13)	<20 (*)	>83 (<0.05)
	60	96 (16)	<20 (*)	>79 (<0.05)
	30 + 30	190 (12)	<20 (*)	>89 (<0.05)
HD	30	190 (22)	38 (8)	80 (<0.05)
	60	110 (14)	<50 (*)	>55 (<0.05)
	30 + 30	240 (31)	<20 (*)	>91(<0.05)
<b>Metal</b>				
derL-1	30	250 (8)	<20 (*)	>92 (<0.05)
	60	250 (14)	<20 (*)	>92 (<0.05)
	30 + 30	110 (19)	<20 (*)	>81 (<0.05)
HD	30	380 (7)	190 (36)	49 (<0.05)
	60	380 (11)	110 (26)	71 (<0.05)
	30 + 30	<42 (*)	<43 (*)	-2.50 (0.5)
<b>Glass</b>				
derL-1	30	270 (37)	<20 (*)	>93 (<0.05)
	60	240 (17)	<20 (*)	>92 (<0.05)
	30 + 30	200 (7)	<20 (*)	>90 (<0.05)
HD	30	420 (16)	240 (17)	42 (<0.05)
	60	400 (6.2)	120 (54)	70 (<0.05)
	30 + 30	360 (6.7)	190 (43)	47(<0.05)

\*Not calculated because one or more values are below the quantitation limit, <20 µg/coupon.

Lower amounts of HD were recovered from test coupons of all material types after reapplication of hydrogen peroxide (3%) compared to the single 30 min application. HD recovery results after

reapplication of hydrogen peroxide (3%) were similar to the 60 min reaction time. Shown in Table 16, the mass of HD recovered from the positive control metal coupons was unexpectedly low in the 30 min reapplication testing (30 + 30). In spite of low recoveries from metal after decontamination, efficacy was not observed because of the unexpectedly low recoveries from the positive control coupons, i.e., there was little difference between the recoveries from positive control or test metal coupons. Rust coloring on the metal where the Agent Yellow was applied suggests corrosion that may be related to the low recoveries from the positive control coupons.

No generalizations can be made regarding the improved efficacy of repeated application of the hydrogen peroxide (3%) or the longer reaction times.

An additional decontamination test with hydrogen peroxide (3%) was performed by spiking wood and glass coupons with 1 µl of neat HD (no L) and repeating the decontamination test with a 30 min reaction time. Results are shown in Table 17. The efficacy was statistically significant, but low (21% on wood and 10% on glass.)

**Table 17. Decontamination of Neat HD using Hydrogen Peroxide (3%) with a 30 min Reaction Time**

Analyte	Reaction Time, min	Mean Positive control coupons Total Mass of derL-1 or HD Recovered, µg (% RSD)	Mean Test Coupon Total Mass of derL-1 or HD Recovered, µg (% RSD)	Mean% Efficacy (p-value)
<b>Wood</b>				
HD	30	460 (26)	320 (21)	21% (<0.05)
<b>Glass</b>				
HD	30	1100 (5)	1000 (3.7)	10% (<0.05)

### 3.3.3.1 *Efficacy Results Using DF200*

DF200, shown in Table 18, was efficacious (86% to 95%) against the L component of Agent Yellow decontaminated from all four materials, as determined by measuring the decrease in derL-1 after a 30 min reaction time. In all cases, after decontamination with DF200 derL-1 extracted was below the limit of detection. Because no derL-1 was detected after a 30 min application of DF200, extending the reaction time to 60 min yielded no improvement in efficacy.

DF200 exhibited low efficacy (23% to 57%) against the HD component of Agent Yellow decontaminated from all materials except metal after a 30 min reaction time. No significant efficacy was observed for decontamination of HD on metal by application of DF200 after the 30 min and 60 min reaction times. Extending the reaction time for DF200 from 30 min to 60 min resulted in little or no additional efficacy on the other three materials.

**Table 18. DF200 Efficacy Results**

Analyte	Reaction Time, min	Mean Positive control coupons Total Mass of derL-1 or HD Recovered, µg (% RSD)	Mean Test Coupon Total Mass of derL-1 or HD Recovered, µg (% RSD)	Mean% Efficacy (p-value)
<b>Concrete</b>				
derL-1	30	250 (15)	<20 (*)	>92 (<0.05)
	60	130 (15)	<20 (*)	>84 (<0.05)
HD	30	380 (5)	290 (25)	23 (0.05)
	60	270 (19)	240 (520)	12 (0.3)
<b>Wood</b>				
derL-1	30	160 (28)	<22 (*)	>86 (<0.05)
	60	99 (15)	<20 (*)	>80 (*)
HD	30	220 (33)	120 8	46 (0.07)
	60	140 (12)	110 (38)	23 (0.13)
<b>Metal</b>				
derL-1	30	160 (42)	<20 (*)	>87 (*)
	60	110 (60)	<20 (*)	>81 (*)
HD	30	90 (120)	150 (42)	-68 (0.17)
	60	>150 (*)	200 (29)	-32 (0.22)
<b>Glass</b>				
derL-1	30	370 (35)	<20 (*)	>95 (*)
	60	310 (17)	<20 (*)	>94 (*)
HD	30	520 (1)	230 (29)	57 (<0.05)
	60	480 (7)	170 (51)	64 (<0.05)

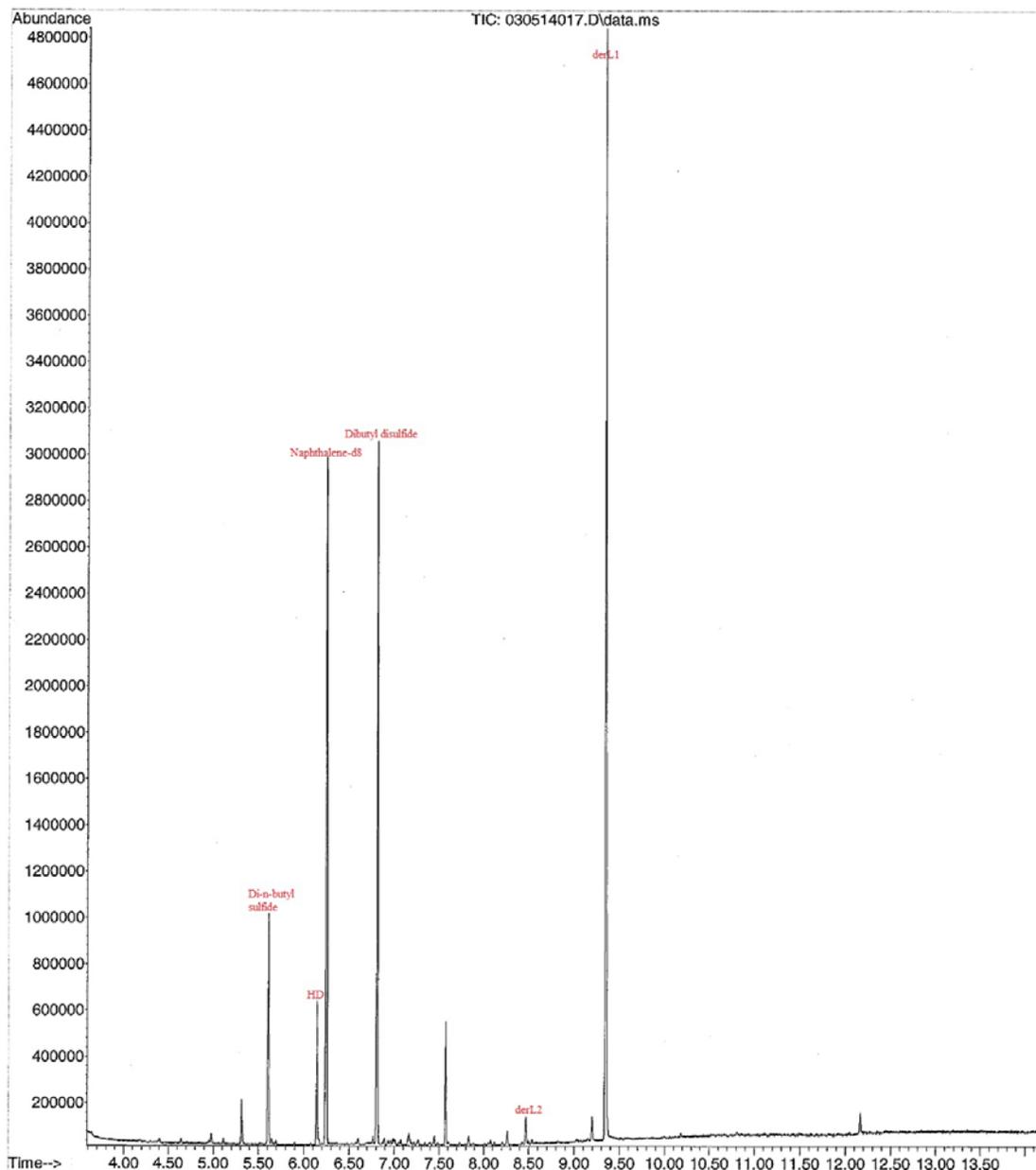
\*Not calculated because one or more values are below the quantitation limit, <20 µg/coupon.

### 3.4 Qualitative Evaluation of By-products

Qualitative analysis for derL-2 and bis(beta-chloroethyl)sulfone (CAS 471-03-4), a vesicant by-product of HD (qualitative) was accomplished using GC/MS. Results of the analyses are summarized in Tables 19 and 20. The qualitative analysis of derL-2 and HD are reported as detection of peaks and the ratios of the areas of the chromatograph peaks for derL-2 to derL-1 and the ratio of the areas of the chromatographic peaks of bis(beta-chloroethyl)sulfone (CAS 471-03-4) to HD.

On some metal and glass positive control coupons, small peaks were observed indicating the presence of derL-2 (see Figure 9); none was detected on any concrete or wood positive control coupons. After decontamination, significant ratios of derL-2 to derL-1 peaks were observed on one or more types of materials. DerL-2 was always detected on metal after decontamination and was found on all coupon types after decontamination with dilute bleach. In all cases, the peaks

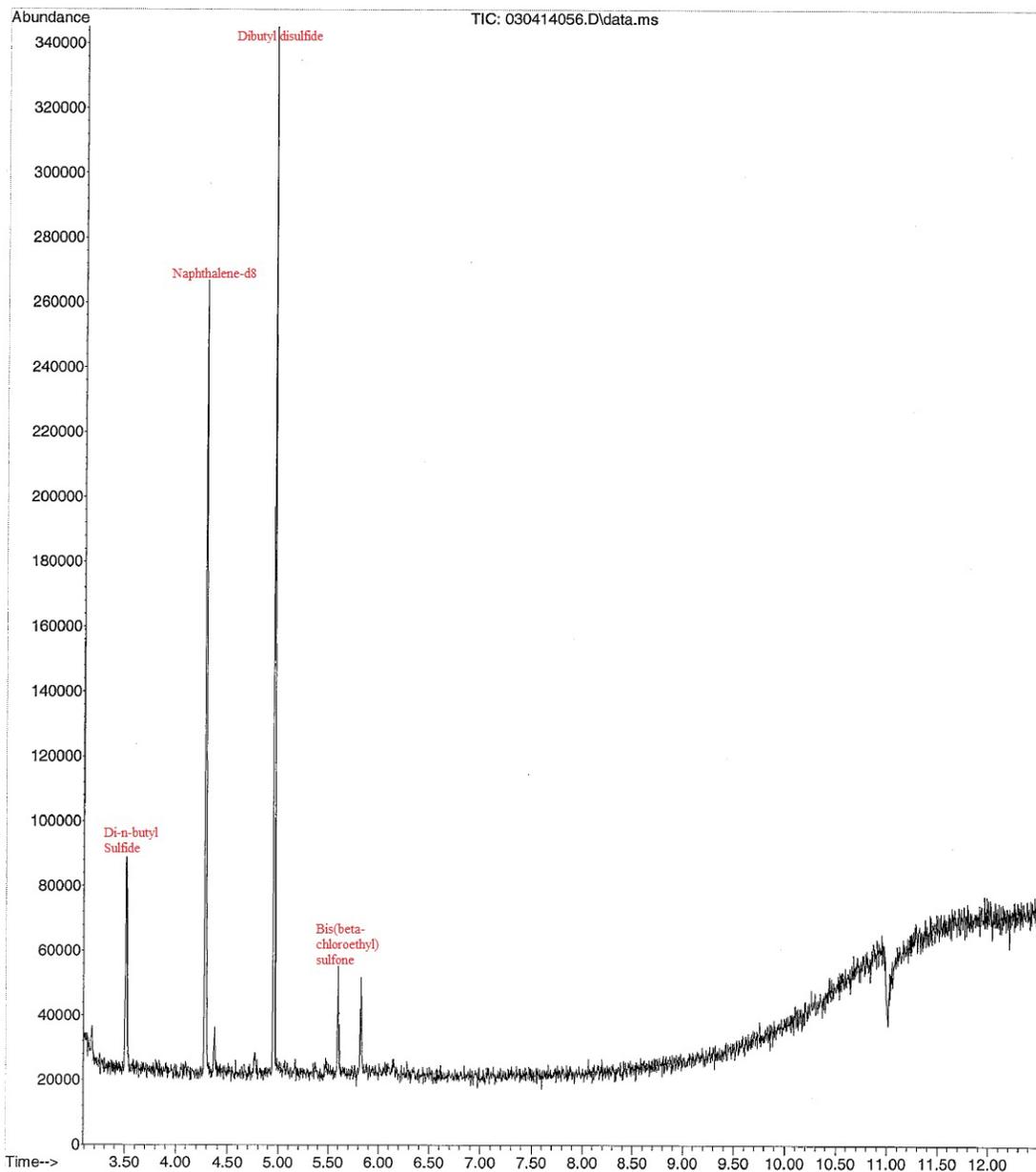
were small or very small compared to the peaks observed for derL-1 on the positive control coupons.



**Figure 9. Chromatogram showing peaks for derL-1, derL-2, and HD.**

The chromatographic peak representative of the HD by-product bis (beta chloroethyl) sulfone (see Figure 10) was found on one of 24 wood positive control coupons, one of 24 metal positive control coupons, and one of 24 glass positive control coupons, but not on any concrete positive control coupons. Chromatographic peaks indicating the presence of the HD by-product were observed on one or more coupon types after decontamination with all four products. After hydrogen peroxide (3%) decontamination the by-product was detected on all coupon types and the ratio of the by-product to residual HD was high (2% on concrete, 16% on wood, 20% on

metal, and 38% on glass after 60 min reaction time). In all cases, the peaks were small or very small compared to the peaks observed for HD on the positive control coupons.



**Figure 10. Chromatogram showing peaks for the HD by-product bis (beta chloroethyl) sulfone.**

**Table 19. Summary of Results for Qualitative Analysis of DerL-2**

	Bleach (full strength)		Bleach (diluted)		Hydrogen Peroxide (3%)		DF200	
	Positive control coupons, n=3	DerL-2/derL-1 (%) or positive / total test coupons, n=5	Positive control coupons, n=3	DerL-2/derL-1 (%) or positive/ total test coupons, n=5	Positive control coupons, n=3	DerL-2/derL-1 (%) or positive/ total test coupons, n=5	Positive control coupons, n=3	DerL-2/derL-1 (%) or positive / total test coupons, n=5
Concrete	30: ND	ND	30:ND	24%	30:ND	1/5	30:ND	5/5; for all coupons but one the derL-2 area exceeded the derL-1 area
			60:ND	8%	60:ND	ND	60:ND	ND
Wood	30:ND	2%	30:ND	1%	30:ND	ND	30:ND	2/5
	60:ND	1%	60:ND	2%	60:ND	ND	60:ND	1/5
Metal	30:1%	5%	30:ND	11%	30:ND	9%	30:ND	1%
	60:ND	3%	60:ND	4%	60:ND	2%	60:1/3	3/5
Glass	30:1/3	ND	30:ND	11%	30:2/3	ND	30:ND	1%
			60:ND	1%	60:ND	3/5	60:ND	24%

Notes: Two reaction times are show 30 min, indicated by “30”, and 60 min, indicated by “60”. “ND” indicates that no peaks indicative of derL-2 were noted on any coupon in that group, e.g., in the first column, concrete, 30 min reaction time positive control coupons. A percentage shown indicates the ratio of the derL-2/derL-1 converted to percentage. If the ratio was <1%, the number of coupons positive for derL-2 out of total coupons in the group is shown, e.g., 2/5 indicates that derL-2 was detected on two of five coupons.

**Table 20. Summary of Results for Qualitative Analysis of HD By-product (Sulfone)**

Material	Bleach (full strength)		Bleach (diluted)		Hydrogen Peroxide (3%)		DF200	
	Positive control coupons, n=3	Sulfone/HD (%) or positive/ total test coupons, n=5	Positive control coupons, n=3	Sulfone/HD (%) or positive/ total test coupons, n=5	Positive control coupons, n=3	Sulfone/HD (%) or positive/ total test coupons, n=5	Positive control coupons, n=3	Sulfone/HD (%) or positive/ total test coupons, n=5
Concrete	30:ND	1/5	30:ND	ND	30:ND	3%	30:ND	1/5
			60:ND	ND	60:ND	2%	60:ND	1%
Wood	30:1/3	2%	30:ND	ND	30:ND	4/5	30:ND	ND
	60:ND	ND	60:ND	ND	60:ND	16%	60:ND	ND
Metal	30:1/3	1/5	30:ND	ND	30:1/3	12%	30:ND	8%
	60:ND	5/5 and much higher area on each coupon than HD	60:ND	1%	60:ND	20%	60:ND	1/5
Glass	30:ND	4/5	30:ND	1/5	30:ND	5%	30:ND	1%
	60:ND	ND	60:ND	4%	60:ND	38%	60:1/3	1%

Notes: Two reaction times are show 30 min, indicated by “30”, and 60 min, indicated by “60”. “ND” indicates that no peaks indicative of the HD by-product were noted on any coupon in that group, e.g., in the first column, concrete, 30 min reaction time positive control coupons. A percentage shown indicates the ratio of the HD by-product converted to percentage. If the ratio was <1%, the number of coupons positive for the HD by-product out of total coupons in the group is shown, e.g., 2/5 indicates that the HD by-product was detected on two of five coupons.

### 3.5 Observations of Damage to Coupons

Example photographs before and after the decontamination treatment are shown in Figure 11. During the surface damage test, bleach (full strength) showed slight discoloration (lightening) of the wood (Figure 11b). Except for the bleach (full strength) on wood, the decontamination treatment resulted in no obvious visible change to any of the coupons (Figures 11a, c, and d). During the efficacy testing, Agent Yellow droplets would lead to rust-colored corrosion of metal coupons where the drop was applied (caused by the Agent Yellow droplet rather than any of the decontaminants).



a. Bleach (dilute) on concrete; left is before, right is after application.



b. Bleach (full strength) on wood; left is before, right is after application.



c. DF200 on metal; left is before, right is after application.



d. Bleach (dilute) on glass; left is before, right is after application.

**Figure 11. Photographs of coupons before and after decontamination treatment.**

## 4.0 Quality Assurance/Quality Control

### 4.1 Control of Monitoring and Measuring Devices

Quality control (QC) requirements and results are shown in Table 21. All results were acceptable.

One parameter in the test/Quality Assurance (QA) plan, “Agent (derL-1 and HD) on Glass Positive Controls,  $\mu\text{g}/\text{mL}$ ,” to verify chemical agent amounts spiked onto coupons had limited usefulness because the glass positive control coupons were not extracted immediately. Rather, they controlled for agent losses, e.g., to evaporation, during the time required for decontamination. Extraction of chemical agent from PTFE disks at time zero, the spike controls, is the basis for determining extraction efficiencies from other materials. The spike control values, rather than the positive control values, should indicate that the mass of agent extracted from the disk (compared to the mass applied) was within an acceptable range. The spike controls using PTFE disks were all in the range of 70% to 120% with %RSD <30% (data not shown).

While the spike controls are more appropriate for evaluating adequacy of recoveries, extraction of L (derL-1) and HD from glass test coupons gave higher recoveries (derL-1 at 119% and HD at 120%) than have been observed from PTFE disks. The derL-1 recovery %RSD for the glass positive control coupons after 30 min weathering were all <30% except during the hydrogen peroxide (3%) test that had a %RSD of 37% and the DF200 test that had a %RSD of 35%.

**Table 21. Quality Control Requirements**

Parameter	Measurement Method	Data Quality Indicators	Results and Corrective Action
Temperature, degrees Celsius (°C)	Thermometer	Compare to calibrated National Institute of Standards and Technology (NIST)-traceable thermometer once before testing, agree $\pm 1$ °C	Accuracy of thermometer was within $\pm 1$ °C limits.
Relative humidity, %	Hygrometer	Compare to calibrated NIST-traceable hygrometer once before testing, agree $\pm 10\%$ (full scale)	Accuracy of hygrometer was acceptable.
Time, sec	Timer/data logger	Compare once before testing; agree $\pm 2$ sec/hour	Accuracy of laboratory clock was acceptable.
Volume, $\mu\text{L}$	Calibrated pipette and repeating dispenser/syringe	Pipettes and repeating dispenser/syringe will be checked for accuracy and repeatability before use by determining the mass of water delivered. The pipette will be acceptable if the range of observed masses for five droplets is $\pm 10\%$ of expected.	1-10 $\mu\text{L}$ pipette – < 3% average error; 50-250 $\mu\text{L}$ pipette – < 1% average error; 100-1000 $\mu\text{L}$ pipette – < 1% average error; 50 $\mu\text{L}$ syringe – < 3% average error
Agent (derL-1 and HD) on Glass Positive Control Coupons, $\mu\text{g}/\text{mL}$	Extraction, GC/MS	The mean percent recovery for a known quantity of each analyte added to a test coupon or an IS used to gauge recovery must fall within the range of 70% to 120% and have a coefficient of variation of <30% between replicates	Recoveries of agent were acceptable and, with two exceptions discussed in the text, coefficient of variation was with acceptable limits. Variance was noted. Because the extractions were occurring 30 min after application rather than immediately after application, and the recoveries from the spike controls were within the target range and variation, no changes were made. See discussion in text.
Agent on Laboratory Blank Coupons, $\mu\text{g}/\text{mL}$	Extraction, GC/MS	Laboratory blanks (coupons without applied agent that are not decontaminated) should have less than 1% of the amount of analyte compared to that found on positive control coupons	No measurable agent detected on laboratory blank coupons.
Agent on Procedural Blank, $\mu\text{g}/\text{mL}$	Extraction, GC/MS	Procedural blanks (coupons without applied agent that are decontaminated) should have less than 5% of the amount compared to that found on positive control coupons	No measurable agent detected on procedural blank coupons.

## 4.2 Equipment Calibrations

The instrumentation used for the analyses are identified in Section 2.6. The required analytical equipment was maintained and operated according to the quality requirements and documentation of the HMRC. All equipment was calibrated at the time of use and at the frequency specified in Table 22.

**Table 22. Equipment Calibration Schedule**

Equipment	Frequency
Calibrated pipette and repeating dispenser/syringe	Prior to testing and every six months thereafter
Calibrated Hygrometer/Thermometer	Prior to testing and annually thereafter
GC/MS	Beginning of each batch of test samples (calibration curve) and a calibration verification standard every six samples and at the end of a batch of samples

Neat L and neat HD were used to prepare stock solutions. The L and HD stocks (concentrations corrected for percent purity) were used to create calibration standards encompassing the appropriate analysis range. L calibration standards were derivatized prior to use. The GC was maintained in calibration such that the coefficient of determination ( $r^2$ ) from the regression analysis of the standards was more than 0.98. In addition, the percent bias for the low standard was less than 25%, and the percent bias for the remaining standards was less than 15%. The GC/MS was tuned initially and as needed following manufacturer's guidelines. A tune check was performed before each analytical run using decafluorotriphenylphosphine (DFTPP). A 12-hour tune time was not employed.

Five-point high and low, overlapping calibration curves for derL1 and HD were used with an overall lower calibration level of 2  $\mu\text{g}/\text{mL}$  and upper level of 150  $\mu\text{g}/\text{mL}$ . Any sample exceeding the upper calibration limit was diluted to a concentration within the calibration range and reanalyzed. Table 23 provides the high and low calibration curve standard levels used during sample analysis.

Except as noted in the deviations, one continuing calibration verification (CCV) check standard was analyzed at the beginning and end of each run and after every five samples. Each analytical run included CCV standard at two different concentrations, with the low standard set to the same level as the lowest calibration standard and the second set near the midpoint of the curve (5.0  $\mu\text{g}/\text{mL}$  and 50.0  $\mu\text{g}/\text{mL}$  for the high curve and 2.0  $\mu\text{g}/\text{mL}$  and 5.0  $\mu\text{g}/\text{mL}$  for the low curve). The two concentrations were alternated throughout the run. The

percent bias for the low calibration check standard was less than 35%, and the percent bias for the remaining calibration check standards were less than 20%.

**Table 23. Calibration Levels**

	High Curve	Low Curve
Level 1	5.0 µg/mL	2.0 µg/mL
Level 2	10.0 µg/mL	2.5 µg/mL
Level 3	25.0 µg/mL	4.0 µg/mL
Level 4	50.0 µg/mL	5.0 µg/mL
Level 5	150.0 µg/mL	10.0 µg/mL

Standards do not exist for derL-2 or BCAA so only a qualitative analysis of these species was performed. The peak area values for derL-2 and BCAA detected in each sample were reported as well as a peak area ratio to the corresponding derL-1 and HD values, respectively.

#### ***4.3 Performance Evaluation Audits***

A performance evaluation (PE) audit was conducted, summarized in Table 24. Acceptable values were: volume ( $\pm 10\%$ ), time ( $\pm 1$  sec/min), chemical mass ( $\geq 85\%$ ), IS ( $\pm 10\%$ ), temperature ( $\pm 1$  °C), and relative humidity ( $\pm 10\%$ ).

**Table 24. PE Results**

Parameter	Audit Procedure	Results
Volume	Pipette used for dispensing chemical agent will be checked for accuracy and repeatability one time before use by determining the mass of water delivered	Pipettes < 4% Syringe < 6%
Time	Compare time to independent clock one time before use	0.0 sec/min
Chemical Mass	Use GC/MS to determine mass of agent delivered to PTFE spike control disks and compare to target application level one time	DerL-1 and HD were above the acceptance of $\geq 85\%$ : DerL-1: 103% (range 88% to 115%) HD: 113% (range 101% to 122%)
Internal Standard	Use GC/MS to measure from a secondary source(Supelco Product # 48715-U, Lot LC04085, Exp. 11/30/2016) and compare to the primary source (Isotec [Aldrich] Product # 176044-1G, Lot TV1320, Exp. 5/20/2015) one time	2% relative percent difference
Temperature	Compare against calibrated National Institute of Standards and Technology (NIST)-traceable thermometer one time before use	< 1 °C
Relative Humidity	Compare against calibrated NIST-traceable hygrometer one time before use	< 1%

#### **4.4 Data Quality Audit**

The QA Manager audited at least 10% of the evaluation data and traced the data from initial acquisition, through reduction and statistical comparisons, to final reporting. All data analysis calculations were checked. Only minor calculation issues were noted with the data that did not have a significant bearing on the reported data. These issues were corrected.

#### **4.5 QA/QC Reporting**

QA/QC procedures were performed in accordance with the QAPP for this study. Two deviations that were not covered by two amendment to the initial QAPP were related to a longer time (by 7 min) between completion of the 10-min sonication and taking of an aliquot into GC vials during the MDL study and one occasion where all required CCV standards were inadvertently omitted from the repeat (low calibration) run for a decontamination test for process blanks and laboratory blanks (no test coupons). Both deviations are expected to have a negligible impact on the results of this study.

## 5.0 Summary

The objective of this evaluation was to develop, demonstrate and apply methods to determine the efficacies of various readily-available, liquid-based methods for the decontamination of Agent Yellow, a mixture of L and HD, from materials. In addition, the persistence of Agent Yellow on four building materials was determined.

Method development was used to determine extraction efficiencies for L and HD extracted from four materials (sealed concrete, wood flooring, galvanized metal, and glass). Three solvents were evaluated: acetone, hexane, and toluene. Efficiencies varied by material. Recoveries of L (measured as derivatized L, derL-1) across the three solvents was lowest from concrete: 66% with toluene, 70% with acetone, and 87% with hexane. Hexane overall had the highest efficiencies (glass and metal at >100% (relative to PTFE recovery), wood at 95%). Recoveries of HD from all material/solvent combinations were 78% (concrete extracted with acetone) or higher. From hexane, recoveries were >100% (107% to 122%) relative to recoveries from PTFE. Based on the results, hexane was selected for extractions in subsequent testing.

The GC/MS method MDL for derL-1 in hexane ranged from 0.03 µg/mL from wood to 0.09 µg/mL from concrete. The GC/MS MDL for HD in hexane ranged from 0.04 µg/mL from wood to 0.12 µg/mL from metal.

Extraction with hexane alone was shown to provide adequate neutralization of the decontaminants (with both 90 and 180 µL of decontaminant included) with 82% to 108% of the derL-1 measured in hexane to which decontaminant was added. Similarly, extraction with hexane alone was shown to provide adequate neutralization of the decontaminants with 85% to 101% of the HD measured in hexane to which decontaminant was added compared to HD in hexane to which no decontaminants were added. (Note that addition of thiosulfate as a quench had little or no effect on HD recovery, but reduced measured amounts of derL-1 to <47% of the positive control coupons with both 90 and 180 µL of decontaminant included.)

The addition of triethylamine and butanethiol for derivatization of the Lewisite was shown not to interfere with the measurement of HD by GC/MS. Measured concentrations in the presence of triethylamine or triethylamine and butanethiol were 94% and 94%, respectively compared to controls.

Measurement of the persistence of the L and HD components of Agent Yellow at ambient laboratory conditions showed that after application of Agent Yellow, less than 20% of the L (measured as derL-1) was recovered from concrete or glass after 4 hours; however 14% of the L was recovered from concrete and <3% of the L was recovered from glass after 18 hours. However, only one of three glass coupon had a measurable level of L after 18 hours.

Decontamination efficacy was evaluated for four decontaminants: bleach (full strength; ~6% sodium hypochlorite), bleach (dilute, ~0.6% sodium hypochlorite), hydrogen peroxide (3%), and DF200. A 30 min reaction time was evaluated for all decontaminants and material combinations. An additional reaction time (60 min) and a 30 min reaction time with a subsequent reapplication of the decontaminant and additional 30 min reapplication) were evaluated for some combinations of decontaminants and materials. Results are summarized in Tables 25, 26, and 27.

With a 30 min reaction time, high efficacy was observed for all four decontaminants against the L (derL-1) component of Agent Yellow on all four material types. Efficacies were generally lower for the HD component than for the derL-1 component with full strength bleach and hydrogen peroxide (3%) more efficacious than diluted bleach or DF200. Efficacies varied by material type as well as decontaminant.

An additional test applying bleach (dilute) or hydrogen peroxide (3%) with a 30 min reaction time against HD only on wood and glass was performed. Dilute bleach demonstrated efficacy of 49% on wood and 30% on glass. Hydrogen peroxide (3%) demonstrated low levels (but statistically significant) of efficacy (21% on wood and 10% on glass).

**Table 25. Summary of Average % Decontamination Efficacy with a 30 min Reaction Time**

Agent Yellow	Material	Decontaminant			
		Bleach (Full Strength)	Bleach (Dilute)	Hydrogen Peroxide (3%)	DF200
<b>derL-1</b>	Sealed Concrete	>92%	>87%	>86%	>92%
	Wood Flooring	*†	>66%	>83%	>86%
	Galvanized Metal	>79%	>94%	>92%	>87%
	Glass	>94%	86%	>93%	>95%
Agent Yellow	Material	Decontaminant			
		Bleach (Full Strength)	Bleach (Dilute)	Hydrogen Peroxide (3%)	DF200
<b>HD</b>	Sealed Concrete	>94%	*	*	23%
	Wood Flooring	37%	*	80%	*
	Galvanized Metal	>37%	*	49%	*
	Glass	>95%	35%	42%	57%

Efficacies shown as “>” had at least one and in most cases all coupon extracts that were below the limit of detection.

\* No significant difference ( $p > 0.05$ ) between the mean agent remaining on the positive control coupons and the mean agent remaining on the test coupons, no significant efficacy is observed.

† There was high variability in the positive controls so the  $p = 0.007$ ; efficacy (although not significant) was 85%.

In the 60 min reaction time testing, derL-1 was below the quantitation limits for all extracts decontaminated with hydrogen peroxide (3%) and DF 200 as well as concrete extract after

decontamination with bleach (dilute). Natural attenuation of agent from positive control coupons occurs during the 60 min period during which the decontamination process is occurring on the test coupons. Thus, in spite of these non-detected recoveries, artificially lower efficiencies were observed after 60 min compared to 30 min. The lower efficacies compared to 30 min reaction times reflect bias arising from the declining derL-1 recoveries from the positive control coupons. The lower recoveries from positive control coupons are likely due to the longer period of evaporation. Because of this bias, the reduction in recovered Agent Yellow as a result of the decontamination process (including both the decontaminant and natural attenuation) is likely more informative than the efficacy value for longer reaction times. No operational significance should be derived from these artificially declining efficacy values. (Because no derL-1 or HD had been recovered after 30 min reaction time with bleach (full strength), testing with concrete and glass at 60 min was not evaluated.)

**Table 26. Summary of Average % Decontamination Efficacy with a 60 min Reaction Time**

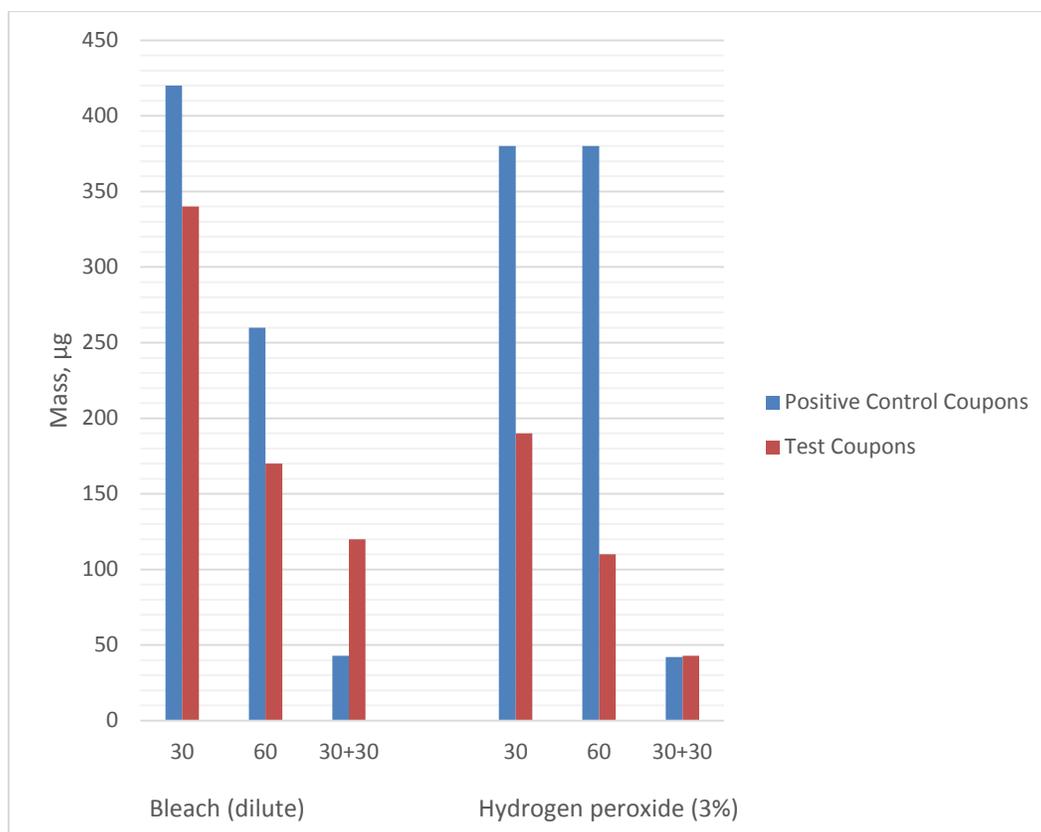
<u>Efficacy for derL-1</u>				
Material	Bleach (Full Strength)	Bleach (Dilute)	Hydrogen Peroxide (3%)	DF200
Concrete	Not tested	>86%	>89%	>84%
Wood	45%	>72%	>79%	>80%
Metal	88%	>76%	>92%	>81%
Glass	Not tested	72%	>92%	>94%
<u>Efficacy for HD</u>				
Material	Bleach (Full Strength)	Bleach (Dilute)	Hydrogen Peroxide (3%)	DF200
Concrete	Not tested	*	63%	*
Wood	*	*	>55%	*
Metal	86%	*	71%	*
Glass	Not tested	62%	70%	64%

Efficacies shown as “>” had at least one and in most cases all coupon extracts that were below the limit of detection. Green color indicates % efficacy was greater with a 60 min reaction time compared to the 30 min reaction time.

\* No significant difference ( $p > 0.05$ ) between the mean agent remaining on the positive control coupons and the mean agent remaining on the test coupons, no significant efficacy is observed.

The 30 min reaction time followed by reapplication for 30 min was only evaluated for two decontaminants, bleach (dilute) and hydrogen peroxide (3%). Lower amounts of HD were recovered from all coupon types after reapplication of bleach (dilute) or hydrogen peroxide (3%) compared to the single 30 min application. HD recovery results after reapplication of bleach (dilute) or hydrogen peroxide (3%) were similar to the corresponding 60 min reaction times for these decontaminants. In spite of low recoveries from metal after decontamination (shown in Figure 12), efficacy was not observed because of the unexpectedly low recoveries from the

positive control coupons, i.e., there was little difference between the recoveries from positive control or test metal coupons. Rust coloring on the metal where the Agent Yellow was applied suggests corrosion that may be related to the low recoveries from the positive control coupons.



**Figure 12. Comparison of the mass of HD recovered from metal positive control coupons and test coupons after a 30 min reaction time (30), 60 min reaction time (60), and 30 min reaction time with reapplication for additional 30 min (30+30) of bleach (dilute) or hydrogen peroxide (3%).**

Qualitative analysis for vesicant by-products for L [L-2 (bis[2-chlorovinyl] chloroarsine and bis(2-chlorovinyl) arsinous acid (both derivatized to derL-2 prior to analysis)] and the vesicant by-product of HD, bis(beta-chloroethyl)sulfone (BCVAA) showed that there were generally no chromatographic peaks observed in positive control coupon extracts that correspond to these chemicals. Small chromatographic peaks consistent with vesicant by-products were detected on some materials after application of each decontaminant. Peak areas correlated with derL-2 were found on all coupon types after decontamination with dilute bleach for both 30 and 60 min reaction times and ranged from 1% to 24% of the corresponding derL-1 peak area. BCVAA was found on all coupon types after hydrogen peroxide (3%) decontamination with 30 and 60 min reaction times.

**Table 27. Summary of Average % Decontamination Efficacy with a 30 min Reaction Time with Reapplication and Subsequent additional 30 min Reaction Time**

<u>Efficacy for derL-1</u>		
Material	Bleach (Dilute)	Hydrogen Peroxide (3%)
Concrete	>94%	>95%
Wood	>83%	>89%
Metal	>83%	>81%
Glass	>91%	>90%
<u>Efficacy for HD</u>		
Material	Bleach (Dilute)	Hydrogen Peroxide (3%)
Concrete	62%	61%
Wood	27%	>91%
Metal	-176%*	-2.5%* (not significant)
Glass	54%	47%

Efficacies shown as “>” had at least one and in most cases all coupon extracts that were below the quantitation limit, <20 µg/coupon.

\* HD recovered from positive control coupons was unexpectedly very low.

In summary, all four methods of decontamination that were tested (bleach [full strength], bleach [dilute], hydrogen peroxide [3%], and DF200 are highly efficacious against the L component of Agent Yellow and exhibit varying levels of efficacy, depending on material and decontaminant, against the HD component after 30 min reaction times. Bleach (full strength) generally removed L below the levels of detection and exhibited efficacies for HD of 37% to >95%. The efficacy ranges for bleach (dilute), hydrogen peroxide (3%), and DF200 were comparable to bleach (full strength) for L, but had lower ranges of efficacy for HD.

Application of decontamination solutions on surfaces as described in this report is one approach than would be part of a remediation strategy. Volumetric decontamination is another approach for larger areas with lower contamination levels. Further research efforts would be required to determine its functionality towards neutralization of these vesicant chemical agents.

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