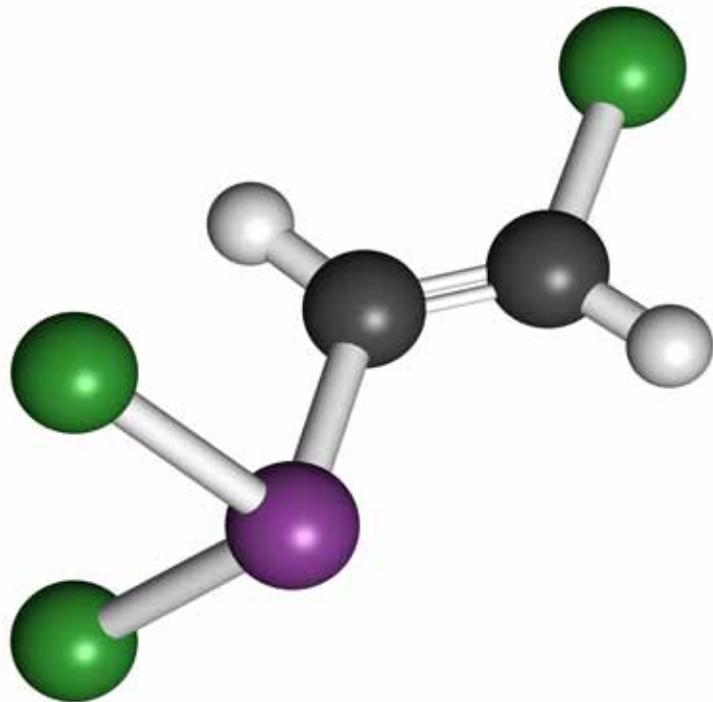


Assessment and Evaluation Report

Decontamination of Lewisite using Liquid Solutions: Neutralization and Arsenic Removal



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National Homeland Security Research Center
Office of Research and Development
U.S. Environmental Protection Agency
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Disclaimer

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Acronyms and Abbreviations

amu	atomic mass units
BBRC	Battelle Biomedical Research Center
BCVAA	bis(2-chlorovinyl) arsonous acid
°C	degrees Celsius
CCV	continuing calibration verification
COR	Contracting Officer Representative
CVAA	2-chlorovinyl arsonous acid
CVAO	2-chlorovinyl arsine oxide (Lewisite oxide)
CVAOA	2-chlorovinyl arsonic 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 DF200
DI	deionized
g	gram(s)
GC	gas chromatography
GFAA	graphite furnace atomic absorption
HPLC	high-performance liquid chromatography
kHz	kilohertz
L-1	2-chlorovinyl dichloroarsine, Lewisite
L-2	bis(2-chlorovinyl) chloroarsine
L-3	tris(2-chlorovinyl) arsine
LC	liquid chromatography
m	meter(s)
µg	microgram(s)
µm	micrometer(s)
µL	microliter(s)
MDL	method detection limit
mg	milligram(s)
min	minute(s)
mL	milliliter(s)
mm	millimeter(s)
MS	mass spectrometry
ng	nanogram(s)
NHSRC	National Homeland Security Research Center

NIST	National Institute of Standards and Technology
NRT	National Response Team
PE	performance evaluation
ppm	part(s) per million
QA	quality assurance
QC	quality control
RSD	relative standard deviation
SAM	Selected Analytical Methods
SIM	selected ion monitoring
TSA	technical systems audit

Executive Summary

Lewisite is an arsenical, vesicant, chemical warfare agent. During construction of the National Response Team Quick Reference Guides, U.S. Environmental Protection Agency (EPA) scientists discovered a lack of data on the decontamination of Lewisite-contaminated surfaces. The objective of this evaluation was to determine the neutralization efficacies of various Lewisite decontamination methods. Lewisite comprises three organo-arsenic vesicants: L-1 (2-chlorovinyl dichloroarsine, Lewisite), L-2 (bis-[2-chlorovinyl]-chloroarsine), and L-3 (tris-[2-chlorovinyl]-arsine). L-1, L-2, and L-3 typically constitute approximately 90 %, 9 %, and 1 %, respectively, of Lewisite. It is known that in the presence of water, L-1 rapidly hydrolyzes to 2-chlorovinyl arsonous acid (CVAA) which is a vesicant. Because decontamination of Lewisite generates arsenical compounds, residual risk may be associated with the decontamination products. Therefore, an additional objective was to evaluate the amount of residual arsenic remaining on building materials after decontamination and wiping with gauze wetted with water or the commercial lead removal product Hygenall[®] LeadOff Surface Decontamination Spray Cleaner (LeadOff).

Decontamination efficacy was evaluated for four decontaminants: water, hydrogen peroxide (3 %), bleach (8.7 % hypochlorite), and EasyDecon DF200 (DF200). Results are summarized in Table ES-1. In the presence of water, a significant decrease in the amount of L-1 occurred that may be attributed to conversion to CVAA. Derivatization during analysis that converts L-1 and CVAA to a common product, derL-1, was used. Significant amounts of derL-1 (i.e. L-1 or CVAA) were recovered relative to the L-1, indicating most of the L-1 had been converted to CVAA. DerL-1 (i.e., L-1 or CVAA) remained on glass and wood- even after a 60 minute (min) contact period with water. Water exhibited the lowest efficacy of the four methods tested at 30 min and no additional efficacy was observed with a longer (60 min) reaction time.

With hydrogen peroxide applied to either glass or wood, neither L-1 nor CVAA (i.e., no derL-1) was detected after a 30- or 60- min reaction time. A small amount of derL-1 was detected in the derivatized extract from wood (but not on glass) after a 30- min reaction time with both bleach and DF200. After a 60- min reaction time with bleach or DF200, no derL-1 (i.e., no L-1 or CVAA) was detected on wood. Hydrogen peroxide, bleach, and DF200 all showed significant efficacy against Lewisite (measured as derL-1).

Table ES-2 summarizes the qualitative results from decontamination of L-2. While analysis showed that the amounts of L-1 recovered from test coupons decontaminated with water were lower compared to the amounts recovered from positive controls, the relative amount of L-2 (qualitative) recovered from the test coupons was not reduced by the 30- min reaction time with water. The L-2 remained on the test coupons in the presence of water, while most of the L-1 was no longer detected. DerL-2 (the common product when derivatizing both L-2 and bis[2-chlorovinyl] arsonous acid) was detected in the derivatized extracts from all glass and some wood coupons after a 60- min contact with water, but was not detected after decontamination with hydrogen peroxide, bleach, or DF200 for 30- or 60- min.

Table ES-1. Summary of L-1 and DerL-1 Efficacy Results.

Form of Agent Analyzed and Decontaminant	Efficacy on Building Materials	
	30 min	60 min
L-1 conversion by water	Glass	Not tested
	Wood ^a	
L-1 conversion by hydrogen peroxide (3 %)	Glass	Not tested
	Wood ^a	
DerL-1 conversion by water	Glass	Glass
	Wood	Wood
DerL-1 conversion by hydrogen peroxide (3 %)	Glass	Glass
	Wood	Wood
DerL-1 conversion by bleach (8.7 % hypochlorite)	Glass	Not tested
	Wood	Wood
DerL-1 conversion by DF200	Glass	Not tested
	Wood	Wood

^a: Insufficient amount of L-1 recovered from positive controls after 30 min to assess efficacy.

Key:

-  Efficacy less than 87 % for agent in specified form, e.g., L-1 or derL-1.
-  Agent detected on some of the test coupons in specified form with efficacy greater than 87 %.
-  No agent detected in specified form and efficacy greater than 94 %.

Table ES-2. Summary of L-2 and DerL-2 Efficacy Results.

Form of Agent Analyzed and Decontaminant	Detection on Building Materials	
	30 min	60 min
L-2 conversion by water	Glass	Not Tested
	Wood	Not Tested
L-2 conversion by hydrogen peroxide (3 %)	Glass	Not Tested
	Wood	Not Tested
DerL-2 conversion by water	Glass	Glass
	Wood	Wood (Detected on Two of Five Coupons)
DerL-2 conversion by hydrogen peroxide (3 %)	Glass	Glass
	Wood	Wood
DerL-2 conversion by bleach (8.7 % hypochlorite)	Glass	Not Tested
	Wood	Wood
DerL-2 conversion by DF200	Glass	Not Tested
	Wood	Wood

Key:

-  Detected in specified form, e.g., L-2 or derL-2.
-  No agent detected in specified form.

Qualitative analysis was performed to detect a potential oxidation product of CVAA, 2-chlorovinyl arsonic acid (CVAOA). After decontamination by hydrogen peroxide for 30 or 60 minutes, detectable amounts of CVAOA were observed, except that after 60- min contact with water, CVAOA was no longer detected on glass or wood. The dynamics of CVAOA formation and degradation were not obvious from these results.

Removal of residual arsenic from coupons after decontamination with water and hydrogen peroxide was evaluated by wiping the coupons with a wetted gauze pad after spraying with either water or LeadOff followed by analysis of residual arsenic on the coupons. Wiping with gauze after spraying with either water or LeadOff was efficacious in removing arsenic from glass. LeadOff removal efficiencies from glass were 93 % -98 %, which were slightly better than using water (84 % -92 %). Removal of arsenic from wood by wiping with wetted gauze after spraying with water or LeadOff was ineffective. The arsenic was assumed to have soaked into the wood where it was not readily removed by wiping.

Based on results obtained in this study, the vesicant properties can be neutralized by using bleach, hydrogen peroxide, or hydrogen peroxide containing products such as DF200. Water only converts the main L-1 component of Lewisite into a different chemical with significant

vesicant properties and is therefore not recommended. Caution should be used in extrapolating from bench testing to field application of these decontamination solutions.

1.0 Introduction

Protecting human health and the environment from the release of hazardous materials is the mission of the U.S. Environmental Protection Agency (EPA). During construction of the National Response Team (NRT) Quick Reference Guides, EPA's National Homeland Security Research Center (NHSRC) scientists discovered there was a lack of data on the persistence of Lewisite and on decontamination of Lewisite-contaminated surfaces. (For NRT Guides, see: [NRT Quick Reference Guides for Chemicals](#)). Figure 1 highlights Lewisite constituents and potential decontamination products that are discussed in this report. As manufactured, Lewisite (L) comprises three compounds: L-1 (2-chlorovinyl dichloroarsine, Lewisite); L-2 (bis[2-chlorovinyl] chloroarsine); and L-3 (tris[2-chlorovinyl] arsine).¹ Lewisite, including L-1, L-2, and L-3, is a Schedule 1 organo-arsenic vesicant under the Chemical Weapons Convention.² In the presence of water, Lewisite rapidly hydrolyzes to 2-chlorovinyl arsonous acid (CVAA) and, with excess water, is rapidly converted to 2-chlorovinyl arsine oxide (Lewisite oxide or CVAO).^{3,4} L-1, CVAA, and CVAO are vesicants.⁴ Lewisite has only low solubility in water and is more volatile than CVAA, which is water soluble,⁴ thus complicating extraction and analysis of Lewisite and degradation products. Further, oxidation of CVAA during decontamination may generate 2-chlorovinyl arsonic acid (CVAOA).⁴ No single method is known for analysis of the three constituents of Lewisite and the hydrolysis and oxidation products.

While Lewisite released into the environment may not persist, various inorganic arsenic decontamination products may persist.⁴ Arsenic, as an element, is expected to remain in degradation products that arise naturally or by applying decontaminants. The residual arsenic may be toxic and needs to be removed to release a contaminated site for reoccupation and use.

Efficacious Lewisite decontaminants would be expected to neutralize the three different compounds that comprise Lewisite (L-1, L-2, and L-3), converting them to non-vesicant compounds. Observing efficacy against Lewisite (L-1, L-2, and L-3) is not sufficient to ensure that products with vesicant properties have not been generated. Additional or alternative testing was, therefore, used to detect such products. In addition, the decontamination strategy must also consider how to remove the remaining toxic arsenical compounds after Lewisite degradation and remove arsenic in totality if materials are to be reused.

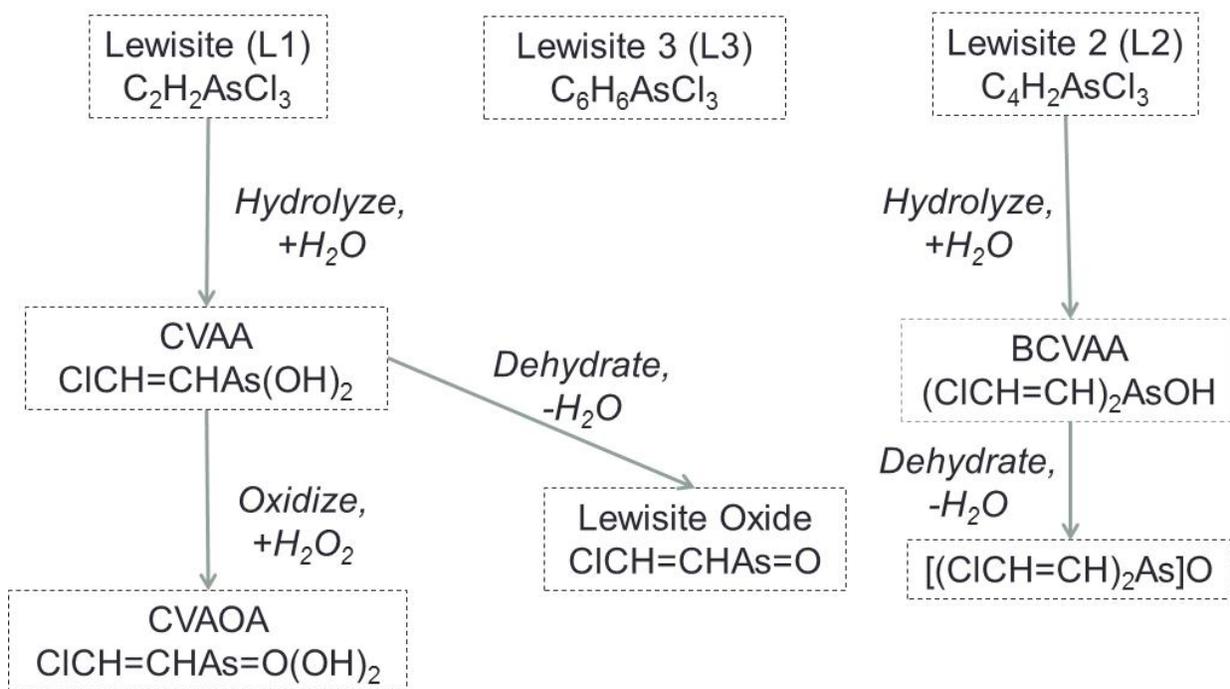


Figure 1. Lewisite and common degradation products for L-1 and L-2.

1.1 Purpose

The overall purpose of this evaluation was to determine the neutralization efficacies of various readily-available, liquid-based methods for Lewisite decontamination (including arsenic removal) from surfaces.

A review of the literature, as part of this effort, showed that methods for evaluating residual Lewisite and decontamination products were limited. Therefore, prior to performing decontamination and removal experiments, method development for the analysis of Lewisite and degradation products was required. The method development and subsequent evaluation of decontamination and arsenic removal technologies are presented here.

1.2 Project Objectives

Specific project objectives to achieve the overall purpose included:

- systematic evaluation of the efficacy of four surface decontaminants for neutralization of Lewisite (conversion to a non-vesicant compound) on two surfaces (glass and wood), and
- evaluation of the efficacy of subsequent arsenic removal by scrubbing the surface with water and with Hygenall[®] LeadOff Surface Decontamination Spray Cleaner (LeadOff).

1.3 Test Facility Description

All testing was performed at the Battelle Biomedical Research Center (BBRC) located on the Battelle site in West Jefferson, Ohio. Battelle is certified to work with chemical surety materials through its contract with the Defense Threat Reduction Agency (contract number: W81XWH-11-D-0002).

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 at least once during each day of testing. The temperature in the laboratory during testing ranged from 19.0 °C – 20.7 °C and the relative humidity ranged from 45 % to 62 %.

2.0 Experimental Methods

Decontamination testing was conducted on a bench-scale to evaluate the efficacy of four decontamination methods against surfaces contaminated with Lewisite. Four types of building materials were included in method development that included determination of extraction efficiencies of alternative methods. Four decontaminants were evaluated for efficacy against Lewisite (L-1 and L-2) applied to two materials. Not all combinations of building materials and decontamination methods were tested as a result of initial testing results.

A post-test-only control group experimental design was used for the determination of the decontamination efficacy against L-1 and L-2. Test coupons were decontaminated (experimental variable) and then extracted and analyzed for L-1 and L-2 (Observation 1; O_1). Positive control coupons were not decontaminated but were extracted and analyzed for L-1 and L-2 (Observation 2; O_2) along with the test coupons. The effect of the treatment (efficacy) was reported as the percentage of L-1 or L-2 remaining on treated coupons compared to the control coupons:

$$\text{Efficacy} = [(O_2 - O_1) / O_2] \cdot 100\% \quad (1)$$

The higher the efficacy, the greater the effect of the decontamination.

In addition to the test and control coupons, laboratory blank coupons (coupons that were neither contaminated with Lewisite nor decontaminated) and procedural blank coupons (coupons that were not spiked with Lewisite, but decontaminated along with the test coupons) were extracted and analyzed for L-1 and L-2. To verify the amount of Lewisite spiked onto coupons, the same amount as applied to coupons was directly pipetted into the extraction solvent and analyzed as a spike control.

Likewise, a post-test-only control group experimental design was used for the determination of the removal of arsenic subsequent to the application of two Lewisite decontaminants. Coupons were spiked with Lewisite, decontaminated, and randomly assigned as test coupons or positive control coupons for the arsenic removal test. Arsenic removal test coupons were wiped with water or with Leadoff (experimental variable) and then extracted and analyzed for arsenic (Observation 1; O_1). Arsenic removal positive control coupons were not wiped with water or Leadoff but were extracted and analyzed for arsenic (Observation 2; O_2). The effect of the treatment (efficacy) was reported as the percentage of arsenic remaining on treated coupons compared to the control coupons and was calculated using Equation 1. The higher the efficacy, the greater the arsenic removal.

2.1 Chemical Agent and Spiking Coupons

The neat Lewisite that was used was supplied by the U.S. Army and owned by the EPA. Because no standards exist for Lewisite, relative composition of L-1 (CAS 541-25-3), L-2 (CAS 40334-69-8), and L-3 (CAS 40334-70-1) in the agent was determined using gas chromatography/mass spectrometry (GC/MS). The Lewisite used in this evaluation was determined to be 92 % L-1 and 3 % L-2; no L-3 was detected. The impurities constituting the remainder of the area under the chromatographic peaks were not identified.

All test and positive control coupons were spiked with 1 microliter (μL ; nominally 1.88 milligrams [mg]) of neat Lewisite onto a coupon surface of about 5.25 square centimeters (cm^2). This area represented a contamination level of approximately 4 grams (g)/meter² (m^2). Each microliter contained approximately 0.68 mg of arsenic. A positive displacement pipette (P/N M-10 [1-10 μL] and CP10 tip, Gilson Inc, Middleton, WI) was used to apply the Lewisite to the test and positive control coupons.

2.2 Test Materials

Four types of building materials were included in method development that included determination of extraction efficiencies of alternative methods. These materials included sealed concrete, wood flooring, galvanized metal, and glass (Table 1). Except for sealed concrete, coupons were cut from larger pieces of material to 3.5 centimeters (cm) \times 1.5 cm. Concrete coupons were poured into a mold and coated with sealer (Sure Klean[®] Weather Seal Siloxane PD). Two materials, glass and wood, were selected by the EPA for subsequent use in the evaluation of the decontaminants based on the ability to extract sufficient Lewisite from these surfaces.

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

Material	Description	Manufacturer/ Supplier Name	Coupon Surface Size Length (cm) x Width (cm)	Material Preparation
Sealed concrete	Epoxy-sealed concrete (5 parts sand; 2 parts concrete); custom preparation	Wysong Concrete, Cincinnati, OH	3.5 \times 1.5	Clean with dry air to remove loose dust
Wood flooring material	Fir plywood (bare); thickness 0.9 cm	Lowe's, Columbus, OH	3.5 \times 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 millimeters (mm) (Adept Manufacturing)	Adept Products, Inc., West Jefferson, OH	3.5 \times 1.5	Clean with acetone
Glass	Glass (clear window)	Brooks Brothers, West Jefferson, OH	3.5 \times 1.5	Clean with dry air to remove loose dust

2.3 Description and Application of Lewisite Decontaminants

Four decontaminants were evaluated for efficacy against Lewisite (L-1 and L-2) as applied to the materials. The first three of four decontamination solutions are, in general, readily available from local retail stores:

- Deionized (DI) water (#23-751-610, Fisher Scientific)
- Bleach (sodium hypochlorite 5-10 %, Clorox® Regular Concentrated bleach (#003-07-0755, Target)
- Hydrogen peroxide (3 %, #3819132, Fisher Scientific)
- EasyDECON DF 200 (DF200, EFT Holdings, Inc.) applied as a liquid.

The decontaminants were applied as a liquid to the test coupons 30 min after the Lewisite was spiked onto the coupons. The initial reaction time for the decontaminants was 30 min. The decontamination testing was repeated at a second reaction time (60 min) for selected combinations of coupons and decontaminant to determine whether extended interaction times of the decontaminant with Lewisite would enhance the decontamination efficacy. Each decontaminant was applied as a single droplet (using a positive displacement pipette (P/N M-100 [100 uL] and D-200 [2-200 uL] tip, Gilson Inc., Middleton, WI). Decontamination volumes ranged from 60 to 90 µL for decontamination of glass and wood, respectively.

2.4 Description and Application of Arsenic Removal Technologies

Two technologies were evaluated for efficacy in removing residual arsenic on glass and wood after decontamination with two of the four decontaminants, namely water or hydrogen peroxide:

- Deionized water (#23-751-610, Fisher Scientific), and
- LeadOff (#HN21131QCS, The LeadOff Store, Newington NH 03801).

The LeadOff manufacturer's instructions are: (1) apply generously; (2) allow to sit 5-10 seconds; (3) wipe with a damp cloth. Based on these instructions, the following approach was used for both deionized water and the LeadOff cleaner:

1. Apply generously with mist setting of spray bottle until entire coupon surface appears visually "wet".
2. Allow treated coupon to sit for ~5-10 seconds.
3. Wet a 5 cm × 5 cm gauze pad (#22-362-178, Fisher) with 2 mL DI water and fold in half.
4. Grasp wetted gauze with disposable forceps and wipe the coupon once from one end to the other using the folded end of the gauze pad.
5. Repeat Step 4 twice with a freshly wetted gauze pad for a total of three wipes.
6. Extract the coupon for arsenic using the Certifier 6⁵ method.

2.5 Extraction of Coupons

At the time of this study, the recommended methods for analysis of Lewisite using the EPA's Selected Analytical Methods for Environmental Remediation and Recovery (SAM)⁶ database only relate to measurement of total arsenic. These methods do not address the change in vesicant properties of this agent during decontamination and were not considered here.

After the appropriate period of contact with the decontaminant, the test coupons were transferred individually into separate 40 mL glass bottles (05-719-120, Fisher Scientific, Pittsburgh, PA) that contained 10 mL of solvent that was added to the vial using a 0.10-110 mL bottle-top dispenser (unknown model number, Barnstead), then sonicating at 50-60 kilohertz (kHz) for 10 min. Solvent selection was based on the outcome of the extraction efficiency studies as part of the method development. The same extraction process was repeated for all samples until each test coupon, positive control coupon, and procedural blank coupon had been extracted and an aliquot taken for analysis. Extraction removes L-1, L-2, and L-3 from the aqueous decontaminating conditions thus halting further decontamination processes in the extract. After the extraction was completed, a 1-mL sample was transferred to a GC vial (P/N HP-5181-8801, VWR [Agilent Technologies], West Chester, PA) using a 1 mL positive displacement pipette (P/N M1000 [100-1000 μ L] and CP1000 tip, Gilson Inc., Middleton, WI) and sealed. Samples that were not analyzed the same day were stored at ≤ -70 °C.

In a similar manner, 10 mL acetone was used to extract a replicate set of coupons for liquid chromatography/MS (LC/MS) analysis for the degradation product CVAOA.

2.6 Derivatization of Subset of Extracts in the Test Matrix

Extracts were analyzed for L-1 and L-2 by GC/MS or derivatized prior to detection by GC/MS for detection of decontamination byproducts. The derivatization process of the extract is described in this section.

2.6.1 Derivatized Lewisite

Based on the results from method development and demonstration, analysis of derivatized Lewisite was used as the primary metric of decontamination efficacy. The derivative of L-1 and its hydrolysis byproduct, CVAA, yield the same product (derL-1). Similarly, the derivative of L-2 and its hydrolysis byproduct, bis(2-chlorovinyl) arsonous acid (BCVAA), yield a common product, derL-2, that can be distinguished from derL-1 using GC/MS.

The derivatization followed the method of Muir et al.⁷ Lewisite derivatives were formed by adding 200 μ L of 1 mg mL⁻¹ butanethiol to a 1 mL aliquot of each Lewisite extract (no coupon material present) to be analyzed. Triethylamine (50 micrograms [μ g]) was added to the solution to catalyze the derivatization. The solution was mixed on a vortex mixer for 10 seconds.

2.7 Analyzing for Lewisite and Degradation Products

Four analysis techniques were investigated but only three were evaluated to analyze the extracts for Lewisite and degradation products: GC/MS with cool on-column injection system;

derivatization of the extract followed by GC/MS; and LC/MS. These methods are described in the following sections.

2.7.1 GC/MS with Cool On-Column Injection System

Method development was required for quantitative analysis of L-1 and qualitative analysis of L-2 and L-3 by GC/MS. Replicate sets of samples were analyzed using an Agilent® 6890N Series GC interfaced to a 5973 network quadrupole mass-selective detector (Palo Alto, CA).

Chromatographic separation of the analytes was conducted using a Restek Rtx-5 fused silica capillary column (Bellefonte, PA), 30 m x 0.25 mm x 0.25 micrometers (µm). The GC/MS conditions used for separation of L-1, L-2, and L-3 from co-extractives are outlined in Table 4.

A conventional application of GC/MS, using the same parameters as in Table 2 but using splitless injection (rather than cool on-column injection) was evaluated for analysis of neat Lewisite. L-1 and L-2 were detected (no L-3 was observed), but with poor sensitivity (no data shown; development funded by other Federal Agency). A revised method was therefore evaluated using a cool on-column injection system, shown in Figure 2, and using the parameters described in Table 2.

Table 2. GC/MS Conditions for Lewisite Analysis

Parameter	Description
Instrument	Agilent Model 6890 Gas Chromatograph equipped with a 5973 Mass Selective Detector and a Model 7683 Injector with AutoSampler.
Column	Restek Rtx-MS5 fused silica capillary column (30 m, 0.25 mm inside diameter, 0.25 µm film thickness)
Injection Temperature	Track oven temperature ± 3 degrees Celsius (°C)
Injection Mode	Cool on-column
Injection Volume	2 µL
Oven Program	40 °C Initial temperature (hold 2 min) 250 °C @ 25 °C/min (hold 0 min) 300 °C Post temperature (hold 0 min)
MS Transfer Line Temperature	250 °C
MS Source Temperature	230 °C
Electron Multiplier Voltage	~2200 V
Mode	Selective ion Monitoring (SIM)

Table 3 outlines the ion masses that were used to quantitate L-1, and identify L-2 and L-3 using the SIM mode.

Table 3. Ions Monitored for Target Chemicals Using GC/MS for Lewisite

Analyte	SIM Ions, <i>m/z</i>
L-1	206, 208, 145
L-2	87, 145, 51, 210
L-3	136, 77, 145

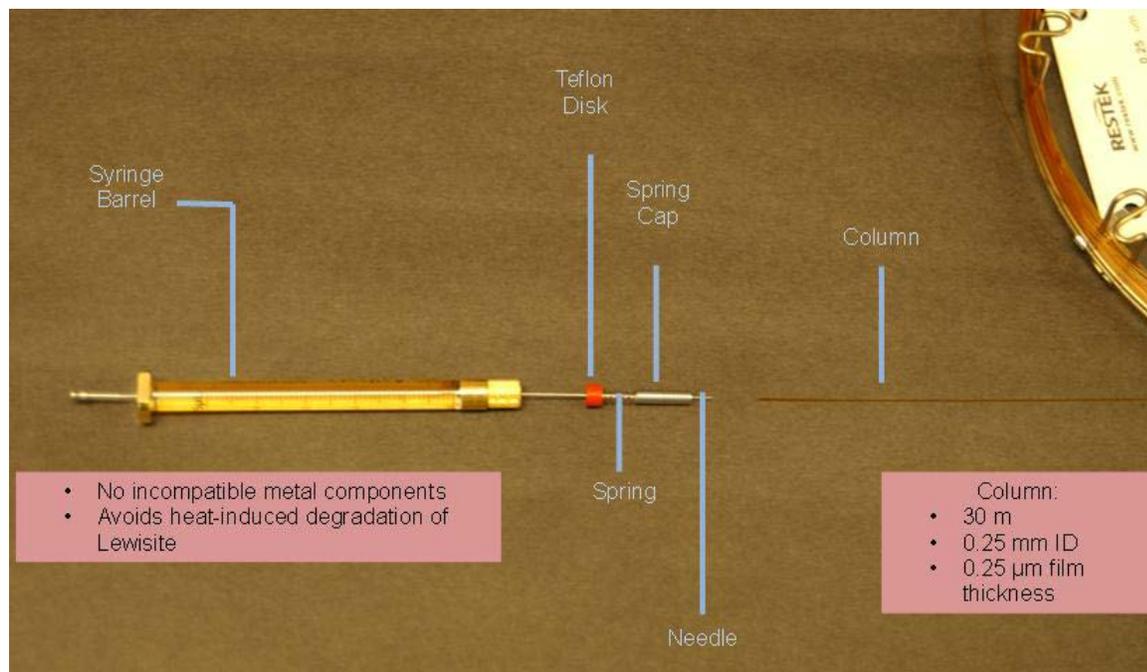


Figure 2. Details of the on-column inlet system.

2.7.2 GC/MS with Derivatization of Lewisite

Method development was required to demonstrate a GC/MS method for analyzing for derL-1 (the common product of derivatizing both L-1 and CVAA) and derL-2 (the common product of derivatizing both L-2 and bis[2-chlorovinyl] arsonous acid).

GC/MS detection was also demonstrated for analyzing derL-1 (the common product of derivatizing L-1 and CVAA) and derL-2 (the common product of derivatizing both L-2 and bis[2-chlorovinyl] arsonous acid). The GC/MS conditions (oven temperature, oven temperature program rate, injection port/detector temperatures, and column flow rates) used for separation of the derivatized Lewisite products are outlined in Table 4. The method of analysis for derL-1 was a procedure similar to that of Hanaoka et al.⁸

The mass selective detector was operated in the full-scan mode for compounds ranging from 40 to 400 amu. Table 5 outlines the target ion masses that were used to identify derL-1 and derL-2.

Table 4. GC/MS Conditions for Derivatized Lewisite Analysis

Parameter	Description
Instrument	Agilent Model 6890 Gas Chromatograph equipped with a 5973 Mass Selective Detector and a Model 7683 Injector with AutoSampler.
Column	Restek Rtx-MS5 fused silica capillary column (30 m, 0.25 mm inside diameter, 0.25 µm film thickness)
Injection Temperature	Track oven temperature ± 3 °C
Injection Mode	Splitless
Injection Volume	1µL
Oven Program	40 °C Initial Temp (hold 2 min) 250 °C @ 15 °C/min (hold 0 min) 300 °C Post Temp (hold 0 min)
MS Transfer Line Temperature	250 °C
MS Source Temperature	230 °C
Electron Multiplier Voltage	~1400 V
Mode	Scan (40-400 atomic mass units [amu])

Table 5. Target Ions Monitored in GC/MS Analysis of Derivatized Lewisite

Analyte	Target Ions, m/z
L-1 (derivatized)	164, 204, 314
L-2 (derivatized)	107, 164, 286

2.7.3 LC/MS for CVAOA analysis

Method development and demonstration included demonstrating an LC/MS method to analyze for the degradation product CVAOA.

LC/MS was used to analyze CVAOA a Lewisite oxidation product that is not detectable by GC/MS. Replicate sets of samples extracted in acetone were analyzed using high performance liquid chromatography (HPLC) coupled to a tandem mass spectrometer (HPLC-MS/MS). A Shimadzu 20 XR series HPLC (Columbia, MD) coupled to an AB SCIEX Triple Quad™ 5500 mass spectrometer with the TurboIonSpray® probe installed (Framingham, MA) was used for CVAOA analysis. Analyst® software was used for data acquisition, instrument control, and data analysis. The method demonstrated followed Battelle’s existing Standard Operating Procedure,

summarized in Table 6, to analyze for CVAOA. Table 7 shows the ion transitions that were used to identify CVAOA.

A single CVAOA standard was included in the LC/MS analyses. This standard was not used to create a set of calibration standards as this was beyond the scope of the intended semi-quantitative analysis of CVAOA by LC/MS. The use of the single standard enabled the results to be reported as greater than or less than the value of the standard.

Table 6. LC/MS Conditions

LC/MS		
Mass Spectrometer	AB SCIEX Triple Quad™5500 with the TurboIonSpray® probe, Positive Ion Mode	
HPLC	Shimadzu 20 XR Series	
Data Acquisition Software	Analyst 1.5.1	
Analytical Column	Phenomenex Prodigy ODS-3 150 x 2 mm, 5 µm (Torrance, CA)	
Guard Column	Phenomenex Security Guard C18, 2.1 x 4 mm (Torrance, CA)	
Column Temperature	30 °C	
Mobile Phase	A: 98:2 H ₂ O/Acetonitrile	
	B: 0.2 % formic acid in 80:20 acetonitrile/isopropanol	
Mobile Phase Gradient	Time (min)	%B
	0	0
	1.0	0
	13.0	25
	15.0	100
	19.0	100
	21.0	0
	25.0	0
Flow Rate	0.2 mL/min	
Injection Volume	50 µL	
Run Time	25 min	

Table 7. Pertinent Parameters for CVAOA Using LC/MS

Analyte	MS/MS transitions monitored
CVAOA	187.0 > 109.0 (primary) 187.0 > 61.0, 187.0 > 91.0, 187.0 > 123.0, 187.0 > 169.0 (all confirmation)

2.8 Method Development and Demonstration

Additional method development and demonstration included:

- Determining extraction efficiency of L-1 and derL-1 from coupons of each of the four material types using three alternative solvents;
- Determining method detection limits for derL-1 by GC/MS;
- Determining extraction efficiencies of arsenic using two alternative methods: modified EPA Method 200.9⁹ and Certifier 6⁵;
- Determining method detection limits for arsenic by graphite furnace atomic absorption (GFAA) spectroscopy.

2.8.1 Extraction Efficiency for L-1 and derL-1

Three solvents (toluene, hexane, and acetone) were selected for extraction efficiency testing for extracting L-1 from the coupon materials used in this evaluation. Extraction of L-1 with each solvent was evaluated for all four building materials (sealed concrete, wood flooring material, galvanized metal ductwork, and glass). The extraction efficiency matrix is shown in Table 8. Neat Lewisite (1 μ L) was spiked onto the test coupons, as described in Section 2.1, and immediately extracted. The test coupons and laboratory blank coupons were extracted as described in Section 2.5. The positive solution controls were prepared by directly injecting 1 μ L of neat Lewisite into the same type of vials containing solvent.

Replicate sets of samples were analyzed for L-1, L-2, and L-3 as described in Section 2.7.1. The extraction efficiency was also measured for derL-1 as described in Section 2.7.2 for the selected extraction solvent for L-1. Samples that were not analyzed the same day were stored at ≤ -70 °C.

Table 8. L-1 Extraction Efficiency Matrix

Solvent	Material	Number of Test Coupons	Number of Solution Spike Controls	Number of Laboratory Blank Coupons
Toluene	Sealed concrete	3	1	1
	Wood flooring material	3		1
	Galvanized metal ductwork	3		1
	Glass	3		1
Hexane	Sealed concrete	3	1	1
	Wood flooring material	3		1
	Galvanized metal ductwork	3		1
	Glass	3		1
Acetone	Sealed concrete	3	1	1
	Wood flooring material	3		1
	Galvanized metal ductwork	3		1
	Glass	3		1
	Total Coupons	36		12

2.8.2 Method Detection Limit for Derivatized Lewisite

A method detection limit (MDL) study was performed for derL-1 for all four materials. For MDL determination, seven samples of each material were spiked with 20 μL of a 4.2 mg/mL L-1 solution in hexane, allowed to sit undisturbed for approximately five minutes, and extracted as described in Section 2.3.2. The hexane extract was derivatized as described in Section 2.5.1. The mass of derL-1 in the extract was determined by GC/MS. The MDL was calculated following the single concentration design estimator (40 CFR Part 136, Appendix B [1984]) as follows:

$$\text{MDL} = t(n-1, 1-\alpha = 0.99) \times \text{SD} \quad (2)$$

where:

$t(n-1, 1-\alpha = 0.99)$ = the Student's t-value for a 99 % confidence level and standard deviation estimate with n-1 degrees of freedom.

SD = standard deviation of the replicate measurements.

2.8.3 Extraction Efficiencies for Arsenic

Two extraction methods for total arsenic from the same coupon materials were demonstrated during method development, as shown in Table 9. The method for analysis for total arsenic was GFAA spectrometry.⁶ Coupons for determining extraction efficiency were spiked with 1 μL of

arsenic reference standard solution (1000 parts per million [ppm] arsenic in 7 % nitric acid; #SA449-500, Fisher Scientific). Extraction efficiency tests were also performed in the presence of selected Lewisite decontamination methods (water and hydrogen peroxide) from glass and wood to ensure that the decontamination method did not interfere with the arsenic extraction analysis. The two extraction methods that were demonstrated were modified from EPA Method 200.9⁹ (aqueous solutions of nitric acid and hydrochloric acid are added to the sample and refluxed at approximately 95 °C) and modified from the method of Certifier 6 described in De La Calle et al. (2010)⁵ (aqueous solutions of nitric acid are added to the sample and microwaved). Analysis was performed following the Certifier 6⁵ method.

Table 9. Evaluation of Total Arsenic Extraction Efficiency

Solvent	Material	Test Coupons	Number of Solution Controls	Laboratory Blank Coupons
Nitric Acid + Hydrochloric Acid⁶	Sealed concrete	3	1	1
	Wood flooring material	3		1
	Galvanized metal ductwork	3		1
	Glass	3		1
Nitric Acid⁷	Sealed concrete	3	1	1
	Wood flooring material	3		1
	Galvanized metal ductwork	3		1
	Glass	3		1
	Total Coupons	24		8

2.8.4 Method Detection Limit for Arsenic

A MDL study was performed for total arsenic measurement for all four materials. To determine the MDL, seven samples of each material were spiked with 1 µL of the 1000 ppm arsenic reference solution, allowed to sit undisturbed for approximately five minutes, and extracted and analyzed as described in Section 2.8.3. The MDL was calculated according to Equation 2.

2.9 Test Matrices

The overall testing sequence for Lewisite decontamination is diagrammed in Figure 3. The test matrix is shown in Table 10. For each combination of time, material and decontamination method, five test coupons (spiked with neat Lewisite, decontaminated for Lewisite), three positive control coupons (spiked with neat Lewisite, not decontaminated for Lewisite) and two procedural control coupons (not spiked with neat Lewisite, decontaminated for Lewisite) were included. 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, for water and hydrogen peroxide 30 min reaction times, additional coupons were included for subsequent analysis for residual arsenic. These included, for each combination of decontaminant and materials, five test coupons (spiked with neat Lewisite, decontaminated for Lewisite, and sprayed/wiped for arsenic removal), three positive control coupons (spiked with neat Lewisite, decontaminated for Lewisite, but not sprayed/wiped for arsenic removal), and two laboratory blank coupons (not spiked with neat Lewisite, decontaminated for Lewisite, and sprayed/wiped for arsenic removal).

No controls that were not wiped were included in the quality assurance project plan. To remedy this oversight, an additional set of coupons was prepared and decontaminated for Lewisite on a different day than the day the arsenic test coupons were prepared. The additional set of coupons included the positive controls for the Lewisite test. These coupons were not wiped prior to extraction and analysis of the arsenic.

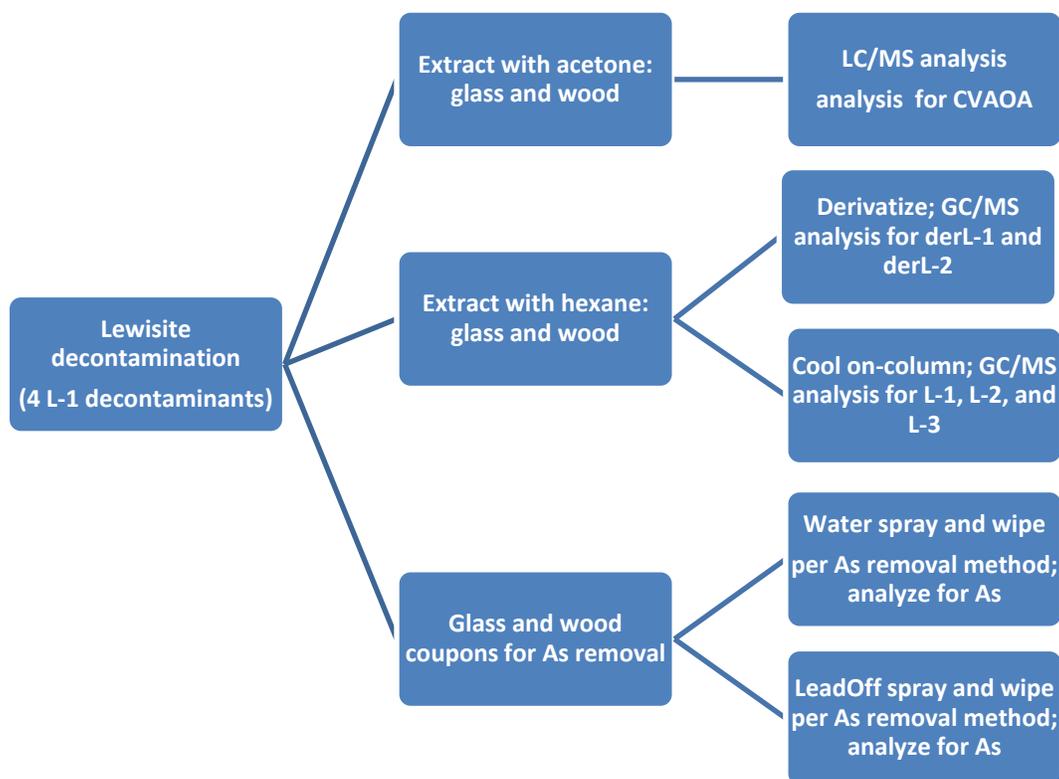


Figure 3. Testing sequence for Lewisite and arsenic removal efficacy evaluation.

2.10 Observation of Surface Damage

The possible 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 and photographs of pre- and post-decontamination coupons are included in Section 3.4. No visual changes to the building materials were observed during testing.

Table 10. Test Matrix for Lewisite Decontamination

Test Day	Decontaminant	Material	30 Min Reaction Time	60 Min Reaction Time
1	Water	Wood flooring material	5 test, 3 positive controls, 2 procedural blanks [Ten additional coupons included for use as test, positive control, and laboratory blank coupons in arsenic removal testing.]	5 test, 3 positive controls, 2 procedural blanks
		Glass	5 test, 3 positive controls, 2 procedural blanks [Ten additional coupons included for use as test, positive control, and laboratory blank coupons in arsenic removal testing.]	5 test, 3 positive controls, 2 procedural blanks
	Hydrogen Peroxide	Wood flooring material	5 test, 3 positive controls, 2 procedural blanks [Ten additional coupons included for use as test, positive control, and laboratory blank coupons in arsenic removal testing.]	5 test, 3 positive controls, 2 procedural blanks
		Glass	5 test, 3 positive controls, 2 procedural blanks [Ten additional coupons included for use as test, positive control, and laboratory blank coupons in arsenic removal testing.]	5 test, 3 positive controls, 2 procedural blanks
2	Bleach	Wood flooring material	5 test, 3 positive controls, 2 procedural blanks	5 test, 3 positive controls, 2 procedural blanks
		Glass	5 test, 3 positive controls, 2 procedural blanks	5 test, 3 positive controls, 2 procedural blanks
	DF200	Wood flooring material	5 test, 3 positive controls, 2 procedural blanks	5 test, 3 positive controls, 2 procedural blanks
		Glass	5 test, 3 positive controls, 2 procedural blanks	5 test, 3 positive controls, 2 procedural blanks

2.11 Extraction Efficiency

Extraction efficiency was calculated using a series of equations as follows. Chemical agent concentration in a coupon extract or positive control solution sample was determined by Equation 3:

$$A_s = a \cdot C_s^2 + b \cdot C_s + c \quad (3)$$

where:

A_s = Area of the target analyte peak in the sample

C_s = Concentration ($\mu\text{g/mL}$) of the target analyte in the sample

a, b, c = Constants.

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

$$M_m = C \times E_v \quad (4)$$

where:

M_m = measured mass of chemical agent (μg)

C = GC concentration ($\mu\text{g/mL}$), see Equation 3

E_v = volume of extract (mL).

Extraction efficiency is then defined as:

$$\text{Extraction Efficiency} = \left(\frac{M_m \text{ of Chemical Agent on Coupon}}{M_m \text{ of Chemical Agent Spiked in Solvent}} \right) \times 100\% \quad (5)$$

where:

M_m = measured mass (μg)

Extraction efficiency = percent recovery of chemical agent from coupons.

The primary assessment of efficacy relies upon comparing the concentration of the target agent, i.e., L-1 or derL-1, on the test coupons, before and after the application of the decontaminant. The purity of derL-1 and the ratio of derL-2 to derL-1 was determined prior to determining extraction efficiency. (No L-3 was detected by qualitative analysis.) Efficacy in percent was calculated as follows:

$$E = (C_o - C_f) / C_o \cdot 100\% \quad (6)$$

where:

E = efficacy

C_o = mean concentration of agent without decontamination (determined from the positive control coupons of each surface material)

C_f = concentration on a test coupon with decontamination.

A separate efficacy calculation was performed for each of the surface materials for L-1 and/or derL-1. For each material, a mean and percent relative standard deviation (%RSD) of efficacy results were reported. Thus, the primary efficacy results from the coupon testing were placed in a matrix table in which each entry shows the mean and %RSD of efficacy results for L-1 or derL-1

on each of the surface materials. The ratios of L-2 and derL-1 to L-1 and derL-1, respectively, were also reported.

A Student's t-test was used to compare the amount of L-1 or derL-1 recovered from test coupons to the amount of agent recovered from positive control coupons; p-values ≤ 0.05 were considered statistically significant.

Similar efficacy determination calculations were performed to compare the mass of arsenic remaining on the coupons after decontamination and subsequent arsenic removal by wiping with water or LeadOff.

3.0 Test Results

3.1 Method Development and Demonstration Results

3.1.1 Extraction Efficiency for L-1 Measured with GC/MS using a Cool On-Column Inlet

As shown in Figure 4, the efficiency with which L-1 was extracted from test coupons depends on the type of surface material onto which the Lewisite was applied and the solvent that was used to extract the Lewisite. For all three solvents (acetone, hexane, and toluene), the highest extraction efficiency was observed from glass, ranging from 65 % (hexane) to 100 % (toluene). The lowest extraction efficiencies were observed from sealed concrete, ranging from 0 % (acetone) to 4 % (toluene). For sealed concrete, only one of the three coupons returned a quantifiable amount of L1.

Low extraction efficiencies may have alternative causes. First, extraction efficiencies may be low due to adsorption or absorption of the L-1 by the material or sealant. Second, extraction efficiencies may be low due to a chemical reaction that converts L-1 to other compounds, e.g., CVAA or CVAOA, that were not detected using the cool on-column GC/MS method. Hexane was selected as the extraction solvent of choice for all GC/MS analysis of L1, L2, derL-1, and derL-2 based on better extraction of L-1 from wood.

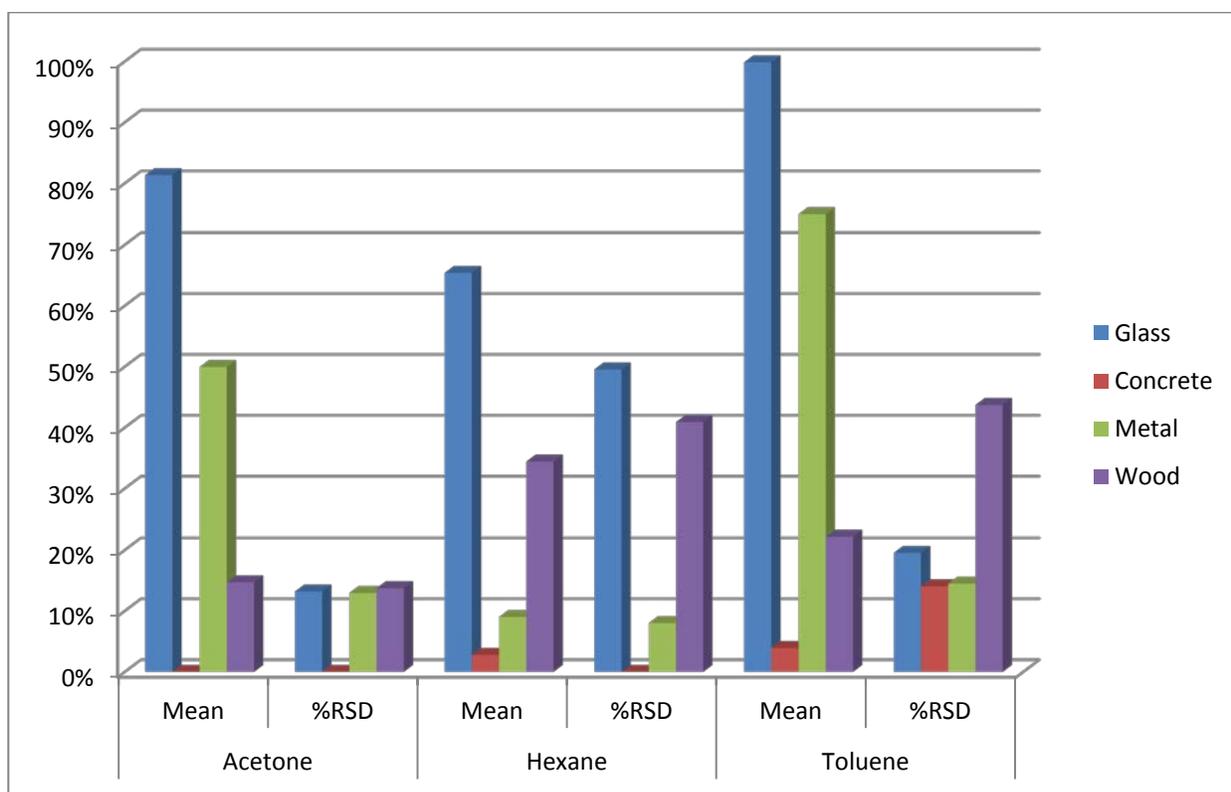


Figure 4. Results for L-1 extraction efficiencies and %RSD (n = 3) by cool on-column GC/MS.

3.1.2 Extraction Efficiency and MDL for derL-1 Analyzed with GC/MS

As shown in Table 11, the extraction efficiency with which L-1 and CVAA (measured as derL-1) were extracted with hexane from glass and wood test coupons was demonstrated to meet data quality objectives (recoveries within the range of 40 % to 120 % of the mass applied to the coupons). Recoveries from wood were much lower and more variable than from glass. The MDL from solution, glass, and wood were all 1.7 µg/mL or less. These MDLs are at least a factor of 100 lower than the nominal mass of Lewisite (~1800 µg) extracted in 10 mL solvent. Therefore, decontamination efficacies as high as 99 % can be determined without additional extraction sample concentration steps.

Table 11. Hexane Extraction Efficiencies and Method Detection Limits for derL-1

Sample Source	Average % Recovered, n=10	%RSD	MDL, µg/mL
Solution (no extraction)	78	7	1.3
Extracted from Glass	77	6	1.1
Extracted from Wood	43	17	1.7

3.1.3 Extraction Efficiency and MDL for Arsenic

As shown in Table 12, the efficiency with which arsenic, applied as arsenic trioxide, was extracted from test coupons ranged from 85 % (metal) to 128 % (galvanized metal) using the EPA Method 200.9.⁹ The efficiency with which arsenic, applied as arsenic trioxide was extracted from the test coupons ranged from 90 % (wood) to 100 % (galvanized metal) using Certifier 6⁵ method. Background levels of arsenic were detected in some materials, as indicated in Table 12 and shown in Table 13. Additional extraction efficiency tests in the presence of two decontaminants (water and hydrogen peroxide) showed no impact on the observed extraction efficiency values (data not shown).

Table 12. Extraction Efficiencies (% Arsenic Applied that was Recovered) from Test Coupons and Solution Controls

Solvent	Material	Test Coupons, n=3	Solution Controls, n=3
Nitric Acid + Hydrochloric Acid (EPA 200.9)⁹	Sealed concrete*	110 %	93 %
	Wood flooring material*	85 %	
	Galvanized metal ductwork	128 %	
	Glass	105 %	
Nitric Acid (Certifier 6)⁵	Sealed concrete*	97 %	96 %
	Wood flooring material	90 %	
	Galvanized metal ductwork*	100 %	
	Glass	92 %	

*Background levels of arsenic were extracted from negative controls; see Table 13.

Table 13. Background Levels of Arsenic Detected in Coupon Materials

Sample Type	Certifier 6 Statistics, µg		EPA 200.9 Statistics, µg	
Negative Sealed Concrete Controls	Mean=	6.6	Mean=	13.8
	StDev=	0.270	StDev=	0.43
	%RSD=	4 %	%RSD=	3 %
Negative Glass Controls	Mean=	<0.25	Mean=	<0.25
	StDev=	n/a	StDev=	n/a
	%RSD=	n/a	%RSD=	n/a
Negative Metal Controls	Mean=	14.2	Mean=	<0.25
	StDev=	0.89	StDev=	n/a
	%RSD=	6 %	%RSD=	n/a
Negative Wood Controls	Mean=	<0.25	Mean=	1.90
	StDev=	n/a	StDev=	0.15
	%RSD=	n/a	%RSD=	7.7 %

The nominal amount of arsenic spiked onto the coupons (as 1 µL Lewisite) is 680 µg. Arsenic background levels across all materials are, therefore, always less than 2 % from expected.

As shown in Table 14, determining the extraction efficiencies using the Certifier 6⁵ method to extract arsenic from sealed concrete, wood, and glass samples spiked with arsenic trioxide met data quality objectives (recoveries within the range of 40 % to 120 % of the mass of arsenic applied to the coupons). MDLs from solution, glass, wood, and sealed concrete were all 3 ng/mL or less. No MDL results were determined for metal because of high background levels of arsenic extracted from the metal.

Table 14. Determination of Arsenic Method Detection Limits Using the Certifier 6 Method

Sample Source	Average % Recovered, n=7 coupons	%RSD	MDL, ng/mL
Solution (no extraction)	101	2.6	0.45
Extracted from Glass	112	13.0	1.5
Extracted from Wood	90	10	3.0
Extracted from Sealed Concrete	91	6.8	1.2
Extracted from Metal	*	*	*

*No results obtained due to high background levels of arsenic.

3.2 Decontamination Results

3.2.1 Solution Test

A solution test was used to determine the efficacy of the decontaminants in the absence of coupon materials. Sixty microliters of decontamination solution were added to a vial containing 1 µL of neat Lewisite (test solutions). The test solutions and positive controls (Lewisite that was not decontaminated) were analyzed for L-1 and (a derivatized sample) for derL-1 after a 15 min reaction time. The results of a 15 minute solution test are shown in Table 15 where efficacy indicates how much of the applied mass was not recovered, e.g., was decontaminated. Efficacies for the 15 min reaction time with water were 97 %, measured as L-1, and 72 %, measured as derL-1. Most of the L-1 positive control was observed to be present by both the cool on-column GC/MS measurement of L-1 (76 %) and by GC/MS measurement of derL-1 (91 %). Contact with water for 15 minutes reduced the measured L-1 and derL-1 by 97 % (only 3 % of the mass applied measured in the extract) and 78 % (only 22 % of the mass applied was measured in the extract), respectively. These results suggest that the L-1 in contact with water was converted to CVAA as well as to unidentified degradation products. (CVAA was detected as derL-1, but not in the L-1 measurement).

No L-1 (for hydrogen peroxide) or derL-1 (for hydrogen peroxide solution, bleach solution, or DF 200 solution) were detected after 15 min contact. (Because hydrolysis in water effectively converted all L-1 to CVAA or other degradation product, analysis for L-1 was not performed for bleach and DF200 decontamination.)

Table 15. Solution Decontamination Test (15 min Reaction Time)

Reaction Time, min	Decontamination Method	Nominal Mass of Lewisite Applied, µg	Mean Total Calculated Mass of L-1, µg (%RSD)	Mean Efficacy, % (L-1)	Mean Total Calculated Mass of der L-1, µg (%RSD)	Mean Efficacy, % (derL-1)
No Exposure	Positive Control	1,880	1,427 (11)	24	1,717 (5)	9
15	Water Test	1,880	40 (15)	97	480 (20)	72
15	Hydrogen Peroxide Test	1,880	<25	>98	<25	>98
15	Bleach Test	1,880	--	--	<25	>98
15	DF200 Test	1,880	--	--	<25	>98

-- No test performed.

Based on peak area, no efficacy was observed for water against L-2, measured as L-2 or derL-2 in the solution test. In contrast, no L-2 or derL-2 were detected in the hydrogen peroxide, bleach, or DF200 solution tests.

3.2.2 Efficiency Results Using Water to Decontaminate Lewisite on Glass or Wood

Water (deionized) was evaluated as a decontamination method for Lewisite as L-1 on glass or wood. The mean mass of L-1 recovered from coupons and corresponding calculated decontamination efficacies after a 30 min reaction time are summarized in Table 16. After 60 min (sum of 30 min delay time between application and start of decontamination plus 30 min decontamination reaction time), 24 % of L-1 was recovered from the glass positive controls. Very low levels of L-1 were recovered from glass (92 % mean efficacy) after a 30 min reaction time with water. No L-1 was recovered from wood, both from positive control coupons and from test coupons after the 30 min reaction time with water.

Table 16. Water Decontamination Efficacy (measuring L-1)

Reaction Time, min	Material	Mass of Lewisite applied, µg	Mean Positive Controls Total Mass of L-1, µg (%RSD)	Mean Test Coupons Total Mass of L-1, µg (%RSD)	Mean Positive Controls Recovery, %	Mean Test Coupons Recovery, %	Mean Efficacy, % (p value)
30	Glass	1,580	373 (9)	28	24	2	92 (<0.05)
30	Wood	1,580	<25 [†]	<25 [†]	<2	<2	*

*Cannot be determined because no L-1 was recovered from positive control coupons.

[†]The value reported was based on the lowest calibration standard for a particular data set.

Water (deionized) was evaluated as a decontamination method for Lewisite analyzed as derL-1 that includes both derivatized L-1 and CVAA extracted from glass or wood after decontamination. The mean mass of derL-1 recovered from coupons and corresponding calculated decontamination efficacies after a 30 min or 60 min reaction time are summarized in Table 17. Significant efficacy was observed at both 30 min and 60 min reaction times on both glass (mean efficacy 31 % and 53 %, respectively) and wood (mean efficacy 81 % and 86 %, respectively). Greater efficacy was observed at the longer reaction time for both materials. However, the mass of derL-1 recovered from glass and wood was much greater than the mass of L-1 without derivatization, suggesting that L-1 was converted to CVAA that was detected after derivatization.

Table 17. Water Decontamination Efficacy (measuring derL-1)

Reaction time, min	Material	derL-1 Mass of Lewisite applied, µg	Mean Positive Controls Total Mass of derL-1, µg (%RSD)	Mean Test Coupons Total Mass of Der L-1, µg (%RSD)	Mean Positive Controls Recovery, %	Mean Test Coupons Recovery, %	Mean Efficacy, % (p value)
30	Glass	1,490	797 (3)	546 (9)	53	37	31 (<0.05)
30	Wood	1,490	800 (8)	154 (23)	54	10	81 (<0.05)
60	Glass	1,610	737 (5)	344 (17)	46	21	53 (<0.05)
60	Wood	1,610	660 (10)	92 (44)	41	6	86 (<0.05)

L-2 was detected on glass, but not on wood after 30 min reaction time with water.

DerL-2 was not detected on glass or wood positive control or test coupons after a 30 min reaction time with water.

No L-1, derL-1, L-2, or derL-2 was found on any laboratory blank or procedural blank coupon.

CVAOA was detected (<25 µg) on two glass positive control coupons and not detected on the third. CVAOA was detected (<25 µg) on all five glass test coupons at 30 minutes. CVAOA was detected (>25 µg) on all three wood positive control coupons and on four of five wood test coupons after a 30 min reaction time with water; CVAOA was detected at <25 µg on the fifth wood test coupon. CVAOA was not detected on glass or wood coupons after a 60 min reaction time with water and was only detected at <250 µg on one of the three wood positive control coupons.

3.2.3 Efficiency Results Using Hydrogen Peroxide to Decontaminate Lewisite on Glass or Wood

Hydrogen peroxide (3 %) was evaluated as a decontamination method for Lewisite as L-1 on glass and wood. The mean mass of L-1 recovered from coupons and corresponding calculated decontamination efficacies after a 30 min reaction time are summarized in Table 18. L-1 was not recovered after a 30 min reaction time with hydrogen peroxide. Mean efficacy was >95 %. No L-1 was found on any laboratory blank or procedural blank coupon.

Table 18. Hydrogen Peroxide Decontamination Efficacy Measured as L-1

Reaction time, min	Material	Mass of Lewisite applied, µg	Mean Positive Controls Total Mass of L-1, µg (%RSD)	Mean Test Coupons Total Mass of L-1, µg (%RSD)	Mean Positive Controls Recovery, %	Mean Test Coupons Recovery, %	Mean Efficacy, % (p value)
30	Glass	1,580	473 (19)	<25	30	<2	>94 (<0.05)
30	Wood	1,580	<100 [†]	<100 [†]	<6	<6	*

*Cannot be determined.

[†]The value reported was based on the lowest calibration standard for a particular data set.

No L-2 was detected on wood test coupons in the hydrogen peroxide 30 min or 60 min reaction time decontamination tests. (No derL-2 was detected on the wood positive controls.)

Hydrogen peroxide (3 %) was evaluated as a decontamination method for Lewisite analyzed as derL-1 that includes both derivatized L-1 and CVAA extracted from glass or wood after decontamination. The mean mass of derL-1 recovered from coupons and corresponding calculated decontamination efficacies after a 30 min or 60 min reaction time are summarized in Table 19. Significant efficacy was observed with 30 min reaction time on both glass (derL-1 not detected; >96 % mean efficacy) and wood (derL-1 not detected on positive controls or test coupons).

After the 30 min and 60 min reaction times with hydrogen peroxide, chromatographic peaks were not detected for derL-2 in the glass extraction sample.

No derL-1 was found on any laboratory blank or procedural blank coupon.

In the 30 min hydrogen peroxide decontamination testing, qualitative analysis showed the product CVAOA was detected at <25 µg on the glass positive control coupons. CVAOA recovered from coupons was >25 µg on all three wood positive control coupons and >25 µg on all glass and wood test coupons after a 30 min contact with hydrogen peroxide. In the 60 min hydrogen peroxide decontamination testing, CVAOA was not detected on the glass and wood positive control coupons. CVAOA was >250 µg on four of five glass test coupons, detected on

one glass coupon at <250 µg, and detected at <250 µg on all wood test coupons after 60 min decontamination with hydrogen peroxide.

Table 19. Hydrogen Peroxide Decontamination Efficacy (measuring derL-1)

Reaction time, min	Material	derL-1 Mass of Lewisite applied, µg	Mean Positive Controls Total Mass of derL-1, µg (%RSD)	Mean Test Coupons Total Mass of derL-1, µg (%RSD)	Mean Positive Controls Recovery, %	Mean Test Coupons Recovery, %	Mean Efficacy, % (p value)
30	Glass	1,490	637 (11)	<25	43	<2	>96 (<0.05)
30	Wood	1,490	470 (18)	<25	32	<2	>95 (<0.05)
60	Glass	1,610	490 (7)	<25	30	<2	>95 (<0.05)
60	Wood	1,610	387 (20)	<25	24	<2	>94 (<0.05)

3.2.4 Efficiency Results Using Bleach to Decontaminate Lewisite on Glass or Wood

Bleach (8.7 % sodium hypochlorite solution by redox titration) was evaluated as a decontamination method for Lewisite analyzed as derL-1 that includes both derivatized L-1 and CVAA extracted from glass or wood after decontamination. The mean mass of derL-1 recovered from coupons and corresponding calculated decontamination efficacies after a 30 min or 60 min reaction time are summarized in Table 20. Significant efficacy was observed with 30 min reaction time on both glass (derL-1 not detected; >96 % mean efficacy) and wood (94 % mean efficacy) and for the 60 min reaction time on wood (derL-1 not detected; >97 % mean efficacy). Glass was not included in the 60 min reaction time evaluation because no derL-1 was detected after the 30 min reaction time.

No derL-2 was detected on positive control or test coupons after bleach decontamination of glass and wood after a 30 reaction time or wood after a 60 min reaction time.

No derL-1 or derL-2 was found on any laboratory blank or procedural blank coupon.

Since no derL-1 was detected in any of the test coupon abstracts following bleach decontamination, no efforts were made to measure the L1 or L2 amount by cool on-column GC/MS.

Table 20. Bleach Decontamination Efficacy (measuring derL-1)

Reaction time, min	Material	derL-1 Mass of Lewisite applied, µg	Mean Positive Controls Total Mass of derL-1, µg (%RSD)	Mean Test Coupons Total Mass of derL-1, µg (%RSD)	Mean Positive Controls Recovery, %	Mean Test Coupons Recovery, %	Mean Efficacy, % (p value)
30	Glass	1,570	670 (24)	<25	43	<2	>96 (<0.05)
30	Wood	1,570	593 (22)	36 (104)	38	2	94 (<0.05)
60	Wood	1,420	723 (4)	<25	51	<2	>97 (<0.05)

CVAOA was detected at <250 µg for glass and wood positive control coupons after 30 min decontamination with bleach. CVAOA was also detected at <250 µg for all five wood test coupons and four of five glass test coupons for the 30 min reaction time. No CVAOA was found after 60 min on wood positive control coupons. After 60 min of bleach decontamination <250 µg CVAOA was present on one wood test coupon; no CVAOA was detected on four coupons. Analysis for CVAOA after the 60 min of bleach decontamination of glass was not conducted.

3.2.5 Efficiency Results Using DF200 to Decontaminate Lewisite on Glass or Wood

DF200 was evaluated as a decontamination method for Lewisite extracted from glass or wood after decontamination and analyzed as derL-1. The mean mass of derL-1 recovered from coupons and corresponding calculated decontamination efficacies after a 30 min or 60 min reaction time are summarized in Table 21. Significant efficacy was observed with 30 min reaction time on both glass (derL-1 not detected; >96 % mean efficacy) and wood (87 % mean efficacy) and for the 60 min reaction time on wood (derL-1 not detected; >97 % mean efficacy). Glass was not included in the 60 min reaction time evaluation because no L-1 was detected after the 30 min reaction time.

No derL-1 was found on any laboratory blank or procedural blank coupon.

No derL-2 was detected on positive control or test coupons after DF200 decontamination of glass and wood after a 30 reaction time or wood after a 60 min reaction time.

Since no derL-1 was detected in any of the test coupon abstracts following DF200 decontamination, no efforts were made to measure the L1 or L2 amount by cool on-column GC/MS.

Table 21. DF200 Decontamination Efficacy (measuring derL-1)

Reaction Time, min	Material	derL-1 Mass of Lewisite applied, µg	Mean Positive Controls Total Mass of derL-1, µg (%RSD)	Mean Test Coupons Total Mass of derL-1, µg (%RSD)	Mean Positive Controls Recovery, %	Mean Test Coupons Recovery, %	Mean Efficacy, % (p value)
30	Glass	1,570	593 (18)	<25	38	<2	>96 (<0.05)
30	Wood	1,570	770 (6)	102 (61)	49	7	87 (<0.05)
60	Wood	1,420	743 (9)	<25	52	<2	>97 (<0.05)

In the 30 min DF200 decontamination testing, qualitative analysis showed the by-product CVAOA was <250 µg/mL on the glass and wood positive control and test coupons, except that one glass test coupon had CVAOA >250 µg/mL. In the 60 min DF200 decontamination testing, qualitative analysis showed the product CVAOA was not detected on the wood positive control coupons, but was detected at <250 µg/mL on four of five test coupons. (No glass coupons were included in the 60 min DF200 decontamination testing.)

3.3 Arsenic Removal Results

Coupons that were spiked with Lewisite and decontaminated with water or with hydrogen peroxide were subsequently sprayed with either water or with LeadOff and wiped with a wetted gauze pad (2 inch x 2 inch gauze sponge). As shown in Table 22, arsenic extracted with nitric acid and analyzed with GFAA from coupons was substantially reduced on glass after spraying with water (85 % – 92 % mean efficacies) or LeadOff (92 %-98 % mean efficacies) and wiping. Neither removal procedure (water nor LeadOff) was efficacious at removing arsenic from wood.

High variability in the amounts of residual arsenic on the un-wiped coupons was noted and in some cases with wood, the amount of arsenic recovered after wiping was greater than the amount of arsenic detected before wiping. The cause of this anomaly was not investigated, but the presence of residual arsenic may have been related to conditions that varied between the day the unwiped coupons were prepared and decontaminated and the day the wiped coupons were prepared and decontaminated.

Table 22. Arsenic Removal Efficiencies Using Water and LeadOff

Decontaminant, Material, and Coupon Type from Lewisite Decontamination Test	No Removal Total Mass of Arsenic, µg (%RSD)	Water Wiping Total Mass of Arsenic, µg (%RSD)	Water Efficacy, %	LeadOff Wiping Total Mass of Arsenic, µg (%RSD)	LeadOff Efficacy, %
Water, Glass Positive Control	251 (17)	29 (14)	88	16 (80)	94
Water, Glass Test Coupon	425 (11)	66 (53)	85	13 (20)	97
Water, Wood Positive Control	263 (55)	448 (10)	0*	452 (19)	0*
Water, Wood Test Coupon	308 (35)	461 (5)	0*	419 (13)	0*
Hydrogen Peroxide, Glass Positive Control	251 (17)	29 (70)	90	21 (78)	92
Hydrogen Peroxide, Glass Test Coupon	417 (22)	35 (52)	92	6.5 (76)	98
Hydrogen Peroxide, Wood Positive Control	263 (55)	527 (17)	0*	444 (17)	0*
Hydrogen Peroxide, Wood Test Coupon	421 (21)	499 (18)	0*	518 (4)	0*

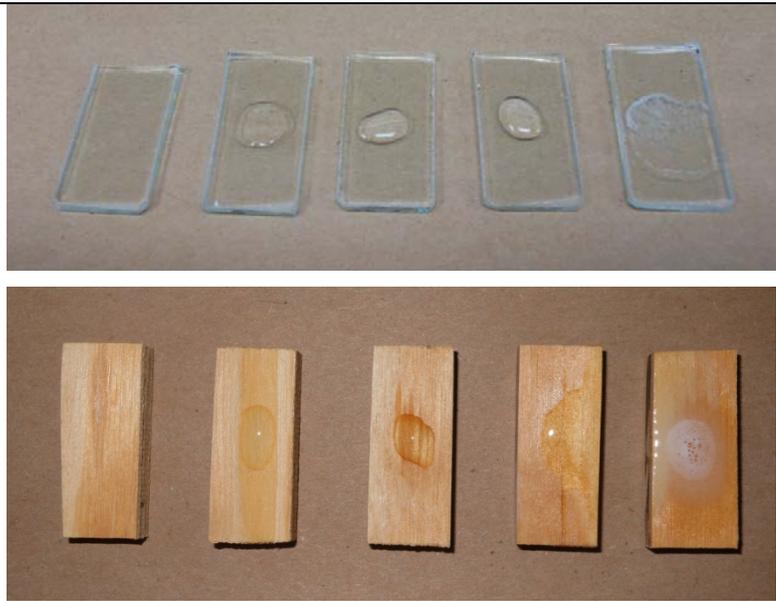
*Recovery after wiping was greater than recovery before wiping.

3.4 Observations of Damage to Coupons

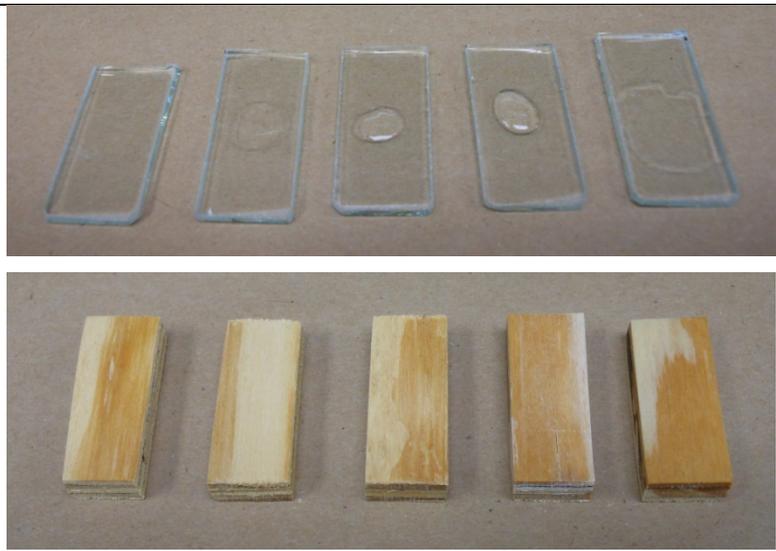
The decontamination treatment resulted in no obvious visible change to any of the coupons. Example photographs before and after the decontamination treatment are shown in Figure 5.

Blank **Water** **Hydrogen Peroxide** **Bleach** **DF200**
↓ ↓ ↓ ↓ ↓

0 min



30 min



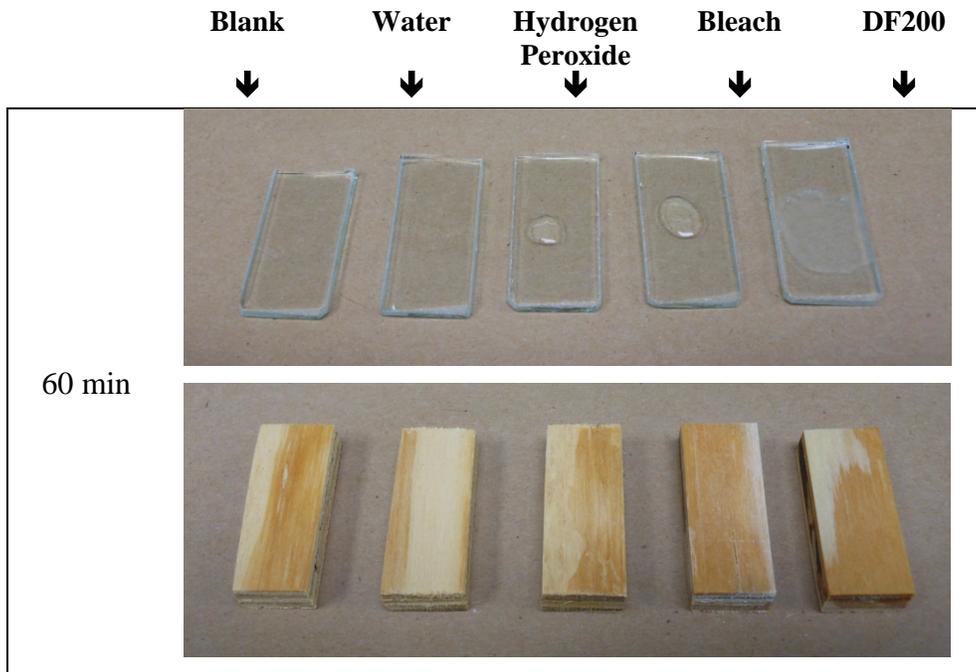


Figure 5. Photographs of coupons before and after decontamination treatment.

4.0 Quality Assurance/Quality Control

4.1 Control of Monitoring and Measuring Devices

Quality control (QC) requirements and results are shown in Table 23.

Table 23. Data Quality Objectives and Results for the Test Measurements

Parameter	Measurement Method	QC Requirement	Results
Temperature, °C	NIST*-traceable thermometer	Compare against calibrated thermometer once before testing, agree ± 1 °C	Accuracy of thermometer was acceptable
Relative Humidity, %	NIST*-traceable hygrometer	Compare against calibrated hygrometer once before testing, agree ± 10 % (full scale)	Accuracy of thermometer was acceptable
Time, sec	Timer/data logger	Compare once before testing; agree ± 2 sec/hour	Accuracy of laboratory clock was acceptable
Volume, μ L	Calibrated pipette	Check pipettes for accuracy and repeatability one time before use by determining the mass of water delivered. The pipette was acceptable if the range of observed masses for five droplets was ± 10 % of expected.	Received with a calibration certificate from manufacturer
Agent on Positive Control, μ g/mL	Extraction, GC	The mean percent recovery for a known quantity of each analyte added to a test coupon used to gauge recovery must fall within the range of 40 %-120 % and have a coefficient of variation of <30 % between replicates	All were within the acceptable range for percent recovery using hexane.
Agent on Laboratory Blank, μ g/mL	Extraction, GC	Laboratory blanks should have less than 1 % of the amount of derL-1 compared to that found on positive controls	All laboratory blanks were blank (no agent detected)
Agent on Procedural Blank, μ g/mL	Extraction, GC	Procedural blanks should have less than 10 % of the amount of derL-1 compared to that found on positive controls	All were within the acceptable range
Arsenic on Positive Control, μ g/mL	Extraction, GFAA	The mean recovery of arsenic must be 40 %-120 % and have a coefficient of variation of <30 % between replicates	All were within the acceptable range for mean recovery using Certifier 6 and EPA 200.9. Observed range 87.9 % - 99.7 %. The coefficient of variance was within the range of acceptance.

Parameter	Measurement Method	QC Requirement	Results
			Observed range <1 % - 11 %.
Arsenic on Laboratory Blank, µg/mL	Extraction, GFAA	Laboratory blanks should have less than 1 % of the amount of arsenic compared to that found on positive controls	In method development, metal and sealed concrete were shown to have background levels of arsenic greater than 1 % of the arsenic recovered from positive controls. Only glass and wood, which had no arsenic above the detection limit (<2.5 ng/mL), were used in arsenic removal testing.
Arsenic on Procedural Blank, µg/mL	Extraction, GFAA	Procedural blanks should have less than 10 % of the amount of arsenic compared to that found on positive controls	All were within the acceptable range

*NIST is the National Institute of Standards and Technology.

4.2 Equipment Calibrations

The instrumentation used to determine L-1, derL-1, CVAOA, and arsenic is identified in Section 2.5.1. The required analytical equipment was maintained and operated according to the quality requirements and documentation of the BBRC. All equipment was calibrated at the time of use and at the frequency specified in Table 24. The LC/MS equipment was not explicitly calibrated as the CVAOA analysis was semi-qualitatively only.

Table 24. Equipment Calibration Schedule

Equipment	Frequency
Calibrated Pipette	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
GFAA	A calibration curve was analyzed and saved prior to testing. Calibration verification standards were run at the beginning of each sample batch, every six samples, and at the end of a batch of samples

For both GC/MS and GFAA spectrometry, independently prepared continuing calibration verification (CCV) standards were analyzed prior to sample analysis, following at least every six samples and at the end of each batch of samples. Two or more CCV concentrations were used, one of which was equal to the low calibration standard and the other(s) within the calibration range. CCV response within 25 % (for the low standard) or 15 % of nominal concentration was acceptable. Samples analyzed prior to or following CCVs that were outside of acceptance limits were re-analyzed, except that the low CCVs for direct measurement of L-1 and L-2 sometimes failed and were not repeated. In those cases, the lowest acceptable calibration provided the lowest value for the calibration curve. (See Section 4.7 for a discussion of this deviation from the test/QA plan.)

At least a five-point calibration was used for each batch of samples for analysis for L-1 with a lower level of approximately 2.5 µg/mL and an upper range of approximately 150 µg/mL. The GC/MS calibration curves met the following performance requirements:

- r^2 greater than 0.99
- % bias for the lowest standard less than 25 %
- % bias for the remaining standards less than 15 %.

Standards do not exist for L-2 and L-3 so only a qualitative analysis of these species of Lewisite was performed. They were reported as a ratio to L-1.

At least a five-point calibration was used for each batch of samples for analysis for derL-1 with a lower level of ~2.5 µg/mL and an upper range of ~60 µg/mL. The GC/MS calibration curves met the following performance requirements:

- r^2 greater than 0.99
- % bias for the lowest standard less than 25 %
- % bias for the remaining standards less than 15 %.

Standards do not exist for derL-2 so only a qualitative analysis of this species of Lewisite was performed. They were reported as a ratio to derL-1.

At least a five-point calibration curve for arsenic was used with a lower calibration level of ~2.5 nanograms (ng)/mL and an upper range of ~50 ng/mL.

The GFAA calibration curves met the following performance requirements:

- r^2 greater than 0.99
- % bias for the lowest standard less than 25 %
- % bias for the remaining standards less than 15 %.

The calibration curve was verified each day of use with the analysis of two calibration standards, one of which was equal to the low calibration level and the other within the calibration range. Independently prepared CCV standards were analyzed prior to sample analysis, following at least every six samples and at the end of each batch of samples. Two or more CCV

concentrations were used, one of which was equal to the low calibration standard and the other(s) within the calibration range. CCV response within 25 % (for the low standard) or 15 % of nominal concentration was acceptable. Samples analyzed prior to or following CCVs that were outside of acceptance limits were re-analyzed.

4.3 Technical Systems Audit

The Quality Assurance (QA) Manager performed a Technical Systems Audit (TSA) during the performance of the decontamination testing. The purpose of the TSA was to ensure that testing was performed in accordance with the test/QA plan. In the audit, the QA Manager reviewed the sampling and analysis methods used, compared actual test procedures to those specified in the test/QA plan and Amendment 1, and reviewed handling procedures. The QA Manager prepared a report, the findings of which were addressed either by modifications to the test procedures or by documentation in the test records. TSA results are summarized in Table 25.

Table 25. TSA Results

Reference	Finding	Corrective Action
<i>Amendment 1, Table A1</i>	While Table A1 in the amendment does specify that GC/MS operating conditions may be modified by the analyst as needed to optimize performance, it does not really cover the changes needed for analysis of derivatized samples.	A formal deviation was prepared to reflect the changes needed for GC/MS of derivatized Lewisite.

4.4 Performance Evaluation Audits

A performance evaluation (PE) audit was conducted for temperature (± 1 °C), relative humidity (± 10 %), and time (± 1 sec/min). Results are shown in Table 26.

Table 26. PE Results

Parameter	Audit Procedure	Expected Tolerance	Results
Temperature	Compare to independent thermometer value	± 1 °C	All were within the acceptable range (± 0.9 °C)
Relative Humidity	Compare to independent hygrometer value	± 10 %	All were within the acceptable range (± 5 %)
Time	Compare time to independent clock	± 1 sec/min	All were within the acceptable range (± 0 sec/min)

4.5 Data Quality Audit

The QA Manager audited at least 10 % of the investigation data and traced the data from initial acquisition, through reduction and statistical comparisons, to final reporting. All data analysis calculations were checked.

4.6 Amendments

One amendment was requested to evaluate alternative analyses for Lewisite degradation products: derivatization and analysis by GC/MS and use of LC/MS. Decisions on decontamination methods, weathering time (time between contamination and decontamination), changes in coupons used, and minor edits were also captured in this amendment.

4.7 Deviations

CCV Deviation. Some low calibration standards failed to satisfy the 15 % criterion and low CCVs failed to satisfy the 25 % criterion for the cool on-column GC/MS analyses. The cool on-column inlet allows for an aqueous sample to be directly deposited onto the capillary column, minimizing heat-induced degradation of Lewisite and enabling L-1 and L-2 to be measured. However, direct sample injection degrades the column phase more quickly resulting in poor chromatography. Study samples were numerous, resulting in frequent reduction in analyte sensitivity and costly instrument maintenance. The arsenic component of these samples also contributes to loss of instrument performance. The ion source becomes dirty over time as sample components accumulate, resulting in deteriorating performance accelerated by the cool on-column injections.

The impact of the CCV deviation on study data was minimal. Changes in calibration range were noted on all affected analyses. Multiple CCVs were included in every analysis and only the lower end of the calibration range required adjustment. Detectable results below the verified calibration range were noted as “less than” the lowest calibration concentration. All study samples were also derivatized for additional GC/MS analysis that was not impacted by calibration challenges.

Analytical Changes to Address MDL of DerL-1 Rather than L-1. Method detection limits were determined for derivatized L-1 rather than L-1. Because the decontamination methods were all aqueous, hydrolysis of L-1 would be expected in all cases. Therefore, the use of derivatization, which detects and measures both L-1 and its similarly harmful hydrolysis product CVAA, was considered to be the more useful analytical approach. Therefore the MDL was determined for the derivatized products using GC/MS rather than L-1 using cool on-column GC/MS.

The impact of the deviation was to improve the usefulness of results compared to measuring only changes in L-1 and L-2 with decontamination.

Internal Standard Deviation. An internal standard was not included in analysis standards or sample extractions; an alternative method for calculating Lewisite concentration that did not include measurement of internal standards was used. In the cool on-column method development the instrument response for the internal standard varied widely for analysis of both samples and

calibration standards. A suitable internal standard for the derivatization of Lewisite was also unknown. Additional methods development would be required to determine a suitable internal standard for use with both Lewisite analysis methods that would substantially add to the project scope. Therefore an internal standard was not used.

An alternative method was used to calculate Lewisite concentration that did not include measurement of an internal standard concentration. Lewisite concentration in a coupon extract or positive control solution sample was determined by Equation 7.

$$A_s = a \cdot C_s^2 + b \cdot C_s + c \quad (7)$$

where:

A_s = Area of the target analyte peak in the sample

C_s = Concentration of the target analyte in the sample

a, b, c = Constants

Procedures in the QAPP provided other control measures that minimized the impact of not having an effective internal standard. Dilutions of Lewisite were prepared on every test day to verify acceptable purity of the neat Lewisite. These dose confirmation samples were prepared using the same positive displacement pipette and a volumetric flask to further aid in identifying any potential equipment failures. All results were acceptable ($\pm 25\%$ of expected).

Each test also included multiple positive control samples for each building material. Positive controls undergo the same manipulation as test samples providing a reference on which decontamination efficacy was established.

5.0 Summary

The objective of this evaluation was to develop/demonstrate methods and apply the methods to determine the neutralization efficacies of various readily-available, liquid-based methods for Lewisite decontamination.

Because decontamination of Lewisite generates arsenical compounds, physical removal of arsenic may be required to adequately remediate a contaminated site or facility. Here, the amount of residual arsenic remaining on building materials after decontamination and spraying with water or LeadOff followed by wiping with wetted gauze was evaluated.

Method development was used to determine extraction efficiencies for L-1 extracted from four materials (sealed concrete, wood flooring, galvanized metal, and glass). Three solvents were evaluated: acetone, hexane, and toluene. Efficiencies varied by material. The best L-1 recovery efficiencies were from glass: 81 % for acetone, 65 % for hexane, and 100 % for toluene. Recoveries from sealed concrete were very low, ranging from 0 % for acetone to 4 % for toluene.

Because L-1 hydrolyzes in the presence of water, method demonstration was included to show that derivatization could be used during analysis to determine the mass of derL-1 (the product of derivatization of both L-1 and CVAA). Extraction efficiencies for derL-1 from glass and wood were 77 % and 43 % respectively. The method detection limit analyzing derL-1 using GC/MS was determined to be 1.1 µg/mL from glass and 1.7 µg/mL from wood. (Glass and wood were selected for subsequent decontamination testing; derL-1 extraction efficiencies were not determined from sealed concrete and metal.)

Extraction efficiencies for arsenic were also determined in method development. Arsenic recoveries ranged from 85 % from galvanized metal to 110 % from sealed concrete using the EPA 200.9 extraction method and 89 % from glass to 100 % from galvanized metal using the Certifier 6 extraction method. Background levels of arsenic were detected in the negative controls for sealed concrete using both extraction methods; from wood flooring using EPA 200.9; and from galvanized metal using Certifier 6⁵. The MDL using the Certifier 6⁵ method was 3 ng/mL or less from solution, glass, wood, and sealed concrete.

Because of the chemical properties of these various vesicant compounds, multiple methods of analysis were required. L-1, L-2, and L-3 have low solubility in water and are more volatile than CVAA and CVAOA which are water soluble, thus complicating extraction and analysis of Lewisite and degradation products. Method development identified hexane as a single extraction for subsequent analysis of L-1, L-2, and L-3 (analyzed using a cool on-column inlet with GC/MS as well as by derivatization and GC/MS) and CVAA and BCVAA (using derivatization and GC/MS). Acetone was used to extract CVAOA that was analyzed using LC/MS.

The amount of L-1 recovered from glass positive controls after a combined 60 min interaction time of Lewisite on glass was less than 30 % while for wood, no L-1 was extracted (below detection limit). This decrease can be attributed to a combination of evaporation, degradation, and absorption of L-1.

Decontamination efficacy was evaluated for four decontaminants: water, hydrogen peroxide (3 %), bleach (8.7 % hypochlorite), and DF200. The results of mixing the decontaminant with Lewisite (no coupons) was summarized in Table 27. L-1 recovered from coupons was at or below 2 % after 15 min contact with water or hydrogen peroxide. However, 22 % of derL-1 was recovered, suggesting that a significant amount of L-1 had been converted to CVAA in the presence of water. An important finding is that evaluating the decontamination efficacy against L-1 alone may lead to an inaccurate conclusion that vesicant properties have been eliminated. Decontamination products with vesicant properties, such as CVAA, may still be present. Decontamination with hydrogen peroxide, bleach, and DF200 was >98 % efficacious (measured as of derL-1).

Table 27. Efficacy (% reduction) of Decontaminants Analyzed as L-1 or derL-1

Reaction time, min	Decontamination Method	Mean Efficacy, % (L-1)	Mean Efficacy, % (derL-1)
No Exposure	Positive Control	[76 % of applied mass recovered]	[91 % of applied mass recovered]
15	Water Test	97	72
15	Hydrogen Peroxide Test	>98	>98
15	Bleach Test	--	>98
15	DF200 Test	--	>98

The results of decontamination by various methods for 30 min or 60 min reaction times are shown in Table 28. While all methods showed efficacy, water exhibited the lowest efficacy at 30 min and no additional efficacy was observed with the longer (60 min) reaction time. No derL-1 was detected after a 30 or 60 min reaction time with hydrogen peroxide applied to either glass or wood. A small amount of derL-1 was detected after a 30 min reaction time for the derL-1 on wood (but not glass) with both bleach and DF200; after a 60 min reaction time with bleach or DF200 no derL-1 was detected.

Table 28. Mean Efficacy of Decontamination Methods Analyzed as DerL-1

Reaction Time, min	Material	Decontamination Method	Mean Efficacy, % (p value)
30	Glass	Water	31 (<0.05)
30	Wood	Water	81 (<0.05)
60	Glass	Water	53 (<0.05)
60	Wood	Water	86 (<0.05)
30	Glass	Hydrogen Peroxide	>96 (<0.05)
30	Wood	Hydrogen Peroxide	>95 (<0.05)
60	Glass	Hydrogen Peroxide	>95 (<0.05)
60	Wood	Hydrogen Peroxide	>94 (<0.05)
30	Glass	Bleach	>96 (<0.05)
30	Wood	Bleach	94 (<0.05)
60	Wood	Bleach	>97 (<0.05)
30	Glass	DF200	>96 (<0.05)
30	Wood	DF200	87 (<0.05)
60	Wood	DF200	>97 (<0.05)

The amount of L-2 present in samples was defined as the percentage of L-2 to L-1 chromatographic peak area. The percent of L-2 relative to L-1 was 53 % for glass positive control coupons in the 30 min water decontamination test and substantially higher for corresponding test coupons. DerL-2 was not detected for 30 min decontamination testing. No derL-2 was found in positive controls coupons, but 4.5 % (relative to derL-1) was detected in 60 min water decontamination glass test coupons and 6.4 % in wood test coupons. No L-2 or derL-2 was detected after decontamination with hydrogen peroxide, bleach, or DF200.

Tables 29 and 30 provide a summary of the results for L-1 and L-2 as determined using both the cool on-column approach and the derivatization during analysis of the Lewisite. All four methods demonstrated efficacy. However, water was not effective at reducing the Lewisite below the limits of detection. Hydrogen peroxide, bleach, and DF200 decontamination were able to reduce both derL-1 and derL-2 below the limits of detection.

Table 29. Summary of Efficacy Results for L-1 and DerL-1

Form of Agent Analyzed and Decontaminant	Efficacy on Building Materials	
	30 min Reaction Time	
L-1 conversion by water	Glass	
L-1 conversion by hydrogen peroxide (3 %)	Glass	
	30 min Reaction Time	60 min Reaction Time
DerL-1 conversion by water	Glass	Glass
	Wood	Wood
DerL-1 conversion by hydrogen peroxide (3 %)	Glass	Glass
	Wood	Wood
DerL-1 conversion by bleach (8.7 % hypochlorite)	Glass	Not tested
	Wood	Wood
DerL-1 conversion by DF200	Glass	Not tested
	Wood	Wood

*: Insufficient amount of L-1 recovered from positive controls after 30 min.

Key:

-  Efficacy less than 87 % for agent in specified form, e.g., L-1 or derL-1.
-  Agent detected on some of the test coupons in specified form with efficacy greater than 87 %.
-  No agent detected in specified form and efficacy greater than 94 %.

Table 30. Summary of Efficacy Results for L-2 and DerL-2

Form of Agent Analyzed and Decontaminant	Detection on Building Materials	
	30 min	60 min
L-2 conversion by water	Glass	Not Tested
	Wood	Not Tested
L-2 conversion by hydrogen peroxide (3 %)	Glass	Not Tested
	Wood	Not Tested
DerL-2 conversion by water	Glass	Glass
	Wood	Wood (Detected on Two of Five Coupons)
DerL-2 conversion by hydrogen peroxide (3 %)	Glass	Glass
	Wood	Wood
DerL-2 conversion by bleach (8.7 % hypochlorite)	Glass	Not Tested
	Wood	Wood
DerL-2 conversion by DF200	Glass	Not Tested
	Wood	Wood

Key:

-  Detected in specified form, e.g., L-2 or derL-2
-  No agent detected in specified form

Results for analysis of coupon extracts seeking to detect CVAOA are summarized in Table 31. Analysis for CVAOA using LC/MS showed that a brief (60 min) contact with wood appears to convert some L-1 into relatively large quantities of CVAOA (>250 ng). After a one hour reaction time with water, CVAOA was not detected on glass or wood. The dynamics of CVAOA formation and degradation were not obvious from these results. In the presence of hydrogen peroxide for 30 or 60 minutes, amounts of CVAOA > 250 ng may be formed.

Table 31. Results of Qualitative Analysis for CVAOA

Reaction time, min	Material	Decontamination Method	CVAOA Positive Controls, n = 3	CVAOA Test Coupons, n=5
30	Glass	Water	Non-detect: 1 Detect, < 25 µg: 2 Detect, ≥ 25 µg: 0	Non-detect: 0 Detect, < 25 µg: 5 Detect, ≥ 25 µg: 0
30	Wood	Water	Non-detect: 0 Detect, < 25 µg: 0 Detect, ≥25 µg: 3	Non-detect: 0 Detect, < 25 µg: 1 Detect, ≥ 25 µg: 4
60	Glass	Water	Non-detect: 3 Detect, < 250 µg: 0 Detect, ≥ 250 µg: 0	Non-detect: 5 Detect, < 250 µg: 0 Detect, ≥ 250 µg: 0
60	Wood	Water	Non-detect: 2 Detect, < 250 µg: 1 Detect, ≥ 250 µg: 0	Non-detect: 5 Detect, < 250 µg: 0 Detect, ≥ 250 µg: 0
30	Glass	Hydrogen Peroxide	Non-detect: 0 Detect, < 25 µg: 3 Detect, ≥ 25 µg: 0	Non-detect: 0 Detect, < 25 µg: 0 Detect, ≥ 25 µg: 5
30	Wood	Hydrogen Peroxide	Non-detect: 0 Detect, < 25 µg: 0 Detect, ≥ 25 µg: 3	Non-detect: 0 Detect, < 25 µg: 0 Detect, ≥25 µg: 5
60	Glass	Hydrogen Peroxide	Non-detect: 3 Detect, < 250 µg: 0 Detect, ≥250 µg: 0	Non-detect: 0 Detect, < 250 µg: 1 Detect, ≥250 µg: 4
60	Wood	Hydrogen Peroxide	Non-detect: 3 Detect, < 250 µg: 0 Detect, ≥250 µg: 0	Non-detect: 0 Detect, < 250 µg: 5 Detect, ≥250 µg: 0
30	Glass	Bleach	Non-detect: 0 Detect, < 250 µg: 3 Detect, ≥ 250 µg: 0	Non-detect: 1 Detect, < 250 µg: 4 Detect, ≥250 µg: 0
30	Wood	Bleach	Non-detect: 0 Detect, < 250 µg: 3 Detect, ≥250 µg: 0	Non-detect: 0 Detect, < 250 µg: 5 Detect, ≥250 µg: 0
60	Wood	Bleach	Non-detect: 3 Detect, < 250 µg: 0 Detect, ≥ 250 µg: 0	Non-detect: 4 Detect, < 250 µg: 1 Detect, ≥250 µg: 0
30	Glass	DF200	Non-detect: 0 Detect, < 250 µg: 3 Detect, ≥250 µg: 0	Non-detect: 0 Detect, < 250 µg: 4 Detect, ≥ 250 µg: 1

Reaction time, min	Material	Decontamination Method	CVAOA Positive Controls, n = 3	CVAOA Test Coupons, n=5
30	Wood	DF200	Non-detect: 0 Detect, < 250 µg: 3 Detect, ≥ 250 µg: 0	Non-detect: 0 Detect, < 250 µg: 5 Detect, ≥ 250 µg: 0
60	Wood	DF200	Non-detect: 3 Detect, < 250 µg: 0 Detect, ≥ 250 µg: 0	Non-detect: 1 Detect, < 250 µg: 4 Detect, ≥ 250 µg: 0

Removal of residual arsenic after decontamination with water and hydrogen peroxide was evaluated by wiping with a gauze pad wetted with either water or LeadOff. As shown in Table 32, spraying the coupon and wiping with gauze wetted with water and LeadOff were efficacious removing arsenic from glass. LeadOff removal efficiencies from glass were, in each case, slightly better than using water. Removal of arsenic from wood was ineffective with either water spray or wiping or LeadOff spray and wiping. It was assumed that the arsenic soaks into the wood where it was not readily removed by wiping.

Table 32. Summary of Arsenic Removal Efficiency Using Gauze Wetted with Water or LeadOff

Decontaminant, Material, and Coupon Type	Water Efficacy, %	LeadOff Efficacy, %
Water, Glass Positive Control	88	94
Water, Glass Test Coupon	84	97
Water, Wood Positive Control	0*	0*
Water, Wood Test Coupon	0*	0*
Hydrogen Peroxide, Glass Positive Control	89	92
Hydrogen Peroxide, Glass Test Coupon	92	98
Hydrogen Peroxide, Wood Positive Control	0*	0*
Hydrogen Peroxide, Wood Test Coupon	0*	0*

*Recovery after wiping was greater than recovery before wiping.

In summary, hydrogen peroxide, bleach, and DF200 all showed significant efficacy against Lewisite and associated vesicant decontamination byproducts. Wiping glass (and possibly other smooth surfaces) with gauze wetted with water or LeadOff appears efficacious. Wiping wood (and possibly other rough and/or porous surfaces) may not be efficacious for arsenic removal.

6.0 References

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