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DECONTAMINATION OF PERSONAL EFFECTS USING HUMIDIFIED CHEMICAL HOT AIR DECONTAMINATION (CHAD)

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Humidified chemical hot air decontamination (CHAD) was shown to be effective at removing absorbed chemical warfare agents (CWAs) from materials, such as coating systems, after an extended aging period. Humidified CHAD was evaluated as a process to remove sulfur mustard (HD) from the personal effects of fallen warfighters. Thorough decontamination of personal items is essential before an item can be returned to a chemical casualty's family members as a keepsake. Items evaluated in this study include coins, military patches, nylon webbing, ID cards, and pocket knives. Traditionally, surfaces that readily absorb CWAs, such as fabrics, present a significant decontamination challenge. Complex features, such as grooves and multi-material interfaces, also present significant challenges to decontamination. Before performing humidified CHAD treatment, all contaminated samples were subjected to a bleach pretreatment procedure. The samples were then placed into small item vapor chambers at CHAD conditions: 170 °F, ~20% relative humidity, and 2 air changes/h. The samples were removed at specified time points to develop an HD-decontamination profile on each material. Humidified CHAD treatment provided a significant reduction of remaining HD, as compared with bleach pretreatment as the only control.

15. SUBJECT TERMS

Chemical hot air decontamination (CHAD)	Chemical casualty	Personal effect (PE)
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PREFACE

The work described in this report was authorized under project number CB10411. The work was started in August 2017 and completed in December 2017.

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This report has been approved for public release.

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EXECUTIVE SUMMARY

With the increased threat of global use of chemical warfare agents (CWAs), handling of contaminated materials after an attack has the potential to result in further exposure. CWAs are acutely toxic substances, so that a small amount of exposure can result in fatality. Most warfighters carry personal items with them while they are deployed. In addition to returning the remains of the chemical casualty to his or her family for proper burial, the casualty's personal effects (PEs) would ideally be returned to loved ones as keepsakes. Therefore, a thorough decontamination process for PEs belonging to chemical casualties must be established to ensure that items can be safely returned without the risk of injury due to exposure.

The approved protocol for decontamination of PEs carried by casualties is to submerge the items in bleach for several minutes, with brushing to remove the majority of the contamination. However, if CWAs are absorbed into the material, a short exposure to bleach may not be sufficient to remove the contaminant. Stakeholders in this area of study support this view and wish to ensure that a chemical casualty's PEs should be returned to family members without the potential fear of CWA exposure and adverse health effects.

Humidified chemical hot air decontamination (CHAD) was shown to be effective at removing absorbed CWAs from materials, such as coating systems, after an extended aging period. Humidified CHAD was evaluated to decontaminate sulfur mustard (HD) from selected PEs typically carried by warfighters. The items evaluated in this study included coins, military patches, nylon webbing, identification (ID) cards, and pocket knives. Traditionally, surfaces that readily absorb CWAs, such as fabrics, present a significant decontamination challenge, as do complex features, such as grooves and multi-material interfaces. Before applying humidified CHAD treatment, all contaminated samples were subjected to a bleach pretreatment procedure. The contaminated samples were then placed into small vapor chambers at CHAD conditions: 170 °F, ~20% relative humidity, and 2 air changes/h. The samples were removed at specified time points to record the decontamination profile of HD on each material. Humidified CHAD treatment provided a significant reduction in remaining HD as compared with bleach pretreatment as the only control.

Humidified CHAD treatment provides a significant reduction of extracted HD from PEs made of fabric and metal, regardless of the presence of complex features. The amount of HD extracted from military patches, nylon webbing, and pocket knives was reduced to below the method limits of detection (MLOD) after 144 h of humidified CHAD treatment. In the case of coinage, the amount of HD extracted was reduced to below the MLOD after 24 h of humidified CHAD treatment. Humidified CHAD was not successful in decontaminating ID cards; the average amount of HD extracted after treatment was 86,700 ng.

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DECONTAMINATION OF PERSONAL EFFECTS USING HUMIDIFIED CHEMICAL HOT AIR DECONTAMINATION (CHAD)

1. INTRODUCTION

1.1 Background

With the increased threat of worldwide use of chemical warfare agents (CWAs), potential exposure due to handling of contaminated materials after a CWA attack is likely. CWAs are acutely toxic substances; exposure to a small amount of CWA may result in fatality. The term chemical casualty refers to a warfighter who has died as a result of CWA exposure. Handling and processing of chemical-casualty remains have become topics of interest to the Defense Threat Reduction Agency (Fort Belvoir, VA).

Most warfighters carry personal items with them on their deployments. In addition to returning the remains of chemical casualties to their respective families for proper burial, their personal effects (PEs) are returned to loved ones as keepsakes. Therefore, a thorough decontamination process for PEs belonging to chemical casualties must be established. This would ensure that the items do not cause injury due to exposure.

The approved protocol for decontamination of contaminated PEs is to submerge the items in bleach for several minutes and scrub them with a brush to remove the majority of the contamination.[1] However, if CWAs are absorbed into the material, a short exposure to bleach may not be sufficient to remove the absorbed contaminant. Handling of items with absorbed contaminants may cause contact or inhalation hazards, even after the items have been subjected to bleach treatment. Furthermore, contaminants such as bis-(2-chloroethyl)sulfide (HD; $C_4H_8Cl_2S$, Chemical Abstracts Service [CAS] no. 505-60-2) have limited solubility in aqueous-based decontaminants. Extended exposure to bleach could adversely affect the item’s integrity. Stakeholders in this area of study have determined that the bleach decontamination protocol may not be sufficient to clean the items and thereby avoid exposure and adverse health effects to family members when PEs are returned.

1.2 Study Objectives

The primary objective of this effort was to evaluate the ability of humidified chemical hot air decontamination (CHAD) to remove HD from contaminated PEs after pretreatment with bleach. A secondary objective of this effort was to determine whether extended treatment times could reduce the remaining HD to instrumental detection levels.

1.3 Literature Review

A literature search was performed to find previous studies that used a hot air approach to decontaminate toxic chemicals. This search encompassed four different electronic databases using the generic search term “hot air decontamination”. The findings were evaluated for possible relevance by reviewing their titles and abstracts. If the publication was thought to be relevant, a copy of the document was obtained. The following databases were used:

- The U.S. Army Edgewood Chemical Biological Center (ECBC; Aberdeen Proving Ground, MD) Technical Library electronic database.[2]
- The Defense Technical Information Center (Fort Belvoir, VA) Scientific and Technical Information Network.[3]
- The Science.gov website (a search engine), which is a gateway to over 1800 scientific websites.[4]
- The American Chemical Society (ACS; Washington, DC) publications (accessed through the ACS website).[5] The search encompassed 46 ACS publications dating back to the early 1900’s.

The majority of documents focused on using hot, humid air to decontaminate materials (including a C-130 aircraft) that were contaminated with *Bacillus* spores.[6–9] An additional area of reported research was the biological decontamination of foodstuffs, particularly eggs and pistachios.[10,11] In the foodstuff studies, hot air was typically used in conjunction with another treatment, such as cold air and infrared heat. Several studies discussed the use of hot air to decontaminate surfaces contaminated with chemicals, but these studies did not use CWAs.[12–14] In one study, Theys and Greer discussed the removal of a biological agent and methyl salicylate (a CWA simulant) from an F-35 Joint Strike Fighter using hot air.[14] In another study, Mundis et al. reported the use of hot air (at 150 °F and 3.5 ft/s) to investigate the decontamination of a C-141 aircraft.[15] Although the majority of this study was focused on developing equipment to achieve the desired temperature and flow within the aircraft, Mundis et al. also discussed some laboratory studies using CWAs. In 2017, Myers et al. published their work on the use of humidified CHAD to remove HD and *O*-ethyl-*S*-(2-diisopropylaminoethyl)methyl phosphonothiolate (VX, $C_{11}H_{26}NO_2PS$, CAS no. 50782-69-9) from multiple surfaces.[16] Myers et al. demonstrated that humidified CHAD could remove significant amounts of CWAs from materials without negatively impacting their structural integrity. The majority of materials evaluated in this study were not related to PEs, but one substrate (nylon webbing) that they investigated was directly related to our research.

2. EXPERIMENTAL PROCEDURES

Lalain et al. provide the chemical biological defense community with robust test methodologies for the evaluation of decontaminant performance on various materials of interest in *Chemical Contaminant and Decontaminant Test Methodology Source Document: Second Edition* (SD2ED).[17] We used the procedures described by Lalain et al. in our research, and these procedures are summarized in the following sections. Interested readers should consult the SD2ED for additional details.

2.1 Materials

Members of the Department of Defense Joint Mortuary Affairs Center provided an extensive list of items that could be found on the body of warfighters who have succumbed to a CWA attack.[*] Six materials that representated a wide variety of types were selected for the evaluation of humidified CHAD. The material types included absorptive and porous materials, bare metals, plastics, and materials with complex features. Coins are very common items that people carry in their pockets. A "lucky" coin is likely to be a desired item for the loved one of a fallen warfighter. Patches on a military uniform may indicate rank and denote special training that a warfighter may have completed. Porous fabric material, such as nylon webbing, is similar to clothing items a soldier may wear (e.g., a belt). Common access cards, driver's licenses, and other forms of identification (ID) may contain photographs of the warfighter, which are desirable keepsakes for family members. Pocket knives are sentimental items that can be passed down through generations of a family. Pocket knives also contain several complex features, such as grooves, hinges, etc., which may present a significant challenge for decontamination. The materials used in this study are listed in Table 1.

The coins and pocket knives were evaluated without any modification. The military patches, nylon webbing, and ID cards were cut into small pieces (approximately 1 × 1 in.) to enable simultaneous CHAD treatment of multiple replicates.

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[*] Dr. Catherine Keaty. Defense Threat Reduction Agency, Fort Belvoir, VA. Personal communication, 2017.

Table 1. Materials Used during This Study

Image	Substrate	Source
	Nickel	General circulation
	Penny	General circulation
	Military patch	Sportsman's guide, U.S. Military surplus patches grab bag, part no. WX2-663906
	Nylon webbing	www.strapworks.com, Mil Spec 4088 webbing (1 in.), desert tan
	ID card	ID edge, blank white CR80 30 mil polyvinyl chloride
	Pocket knife	www.Branders.com

Mil Spec: military specification.

2.2 Reagents and Standards

All the chemicals were used as received from the manufacturer. The chloroform (high-performance liquid chromatography [HPLC] grade) and *n*-hexane (Chromasolv Plus, HPLC grade, ≥95% purity) were purchased from Sigma–Aldrich (Milwaukee, WI). The bleach (Concentrated Chlorox germicidal bleach with 7.6% available chlorine) was purchased from Ability One Base Supply Center (Hanover, MD) and analysed at ECBC using a standard method to verify the amount of chlorine in the bleach.

ASTM Type I water was obtained from a Milli-Q Advantage A10 water purification system (Millipore Sigma; Burlington, MA). The A10 system was fed with reverse osmosis water produced by a GE Osmonics reverse osmosis water purification system (GE Osmonics, Inc.; Minnetonka, MN).

The neat HD used in this project was obtained through the Chemical Agent Standard Analytical Reference Material (CASARM) program (lot number HD-U-5038-CTF-N). The CASARM HD was clear and colorless and had a reported purity of 98.0 ± 0.4 mol%. It contained 0.47 mol% 1,4-dithiane ($C_4H_8S_2$, CAS no. 505-29-3) and 0.08 mol% 1,2-dichloroethane ($C_2H_4Cl_2$, CAS No. 107-06-2).[18] The isotopically labeled HD (^{13}C-HD), used as an internal standard (IS), was synthesized in-house. The ^{13}C-HD was 50% labeled at each carbon, and the chemical purity of the neat ^{13}C-HD was 99.5 wt%, which was determined using an established gas chromatography method[19]

2.3 Small Item Vapor (SIV) Emission Chamber

The SIV emission chamber (Figure 1) was constructed from 316 stainless steel, and the chamber dimensions were 12 × 12 × 10 in. (height × width × depth). The chamber walls were insulated to maintain temperature for long periods of time. Heat was transferred to the chamber using a liquid-air heat exchanger (A). Heated water flowed through the tubing, and a set of electronic fans (B) moved the air within the chamber through the heat exchanger. A 400 W resistive heater (C) that was mounted to the back wall of the chamber was used as a secondary source of heat in the chamber. The substrates rested on a stage constructed from expanded aluminum (D) to allow the heated air to circulate completely around the substrate for even heating. A humidified air stream was supplied to the chamber. Temperature and humidity sensors were integrated into the chamber walls to monitor the environmental conditions (E). Details on the development and validation of this small item testing approach were published in a report by Lalain.[20]

The air supplied to the SIV chamber was humidified using a Nafion membrane (PermaPure, product number FC200-780-7MP). Nafion is a tubular membrane, which humidifies an air stream by allowing water to migrate from inside to outside, where a heated air stream is flowing. Heating the water increases the rate of migration across the membrane, and heating the air outside the membrane increases the capacity of the air to hold moisture. The air lines between the humidifier and the SIV chamber were heat-traced to prevent condensation (i.e., loss of humidity).

The target environmental conditions for the humidified CHAD treatment were 76.7 °C (170 °F) and 20% relative humidity (RH). This target temperature was chosen because the upper temperature limit for sensitive aircraft electronics is 180 °F, and it was desired that the temperature remain below 180 °F to reduce the possibility of material breakdown.[20] The maximum RH achievable with the experimental apparatus at this temperature is 20%, which translates to 51 g/m^3 at the target temperature of 76.7 °C. The airflow through the SIV chamber was approximately 0.7 L/min, which is at a rate of approximately 2 air changes/h.

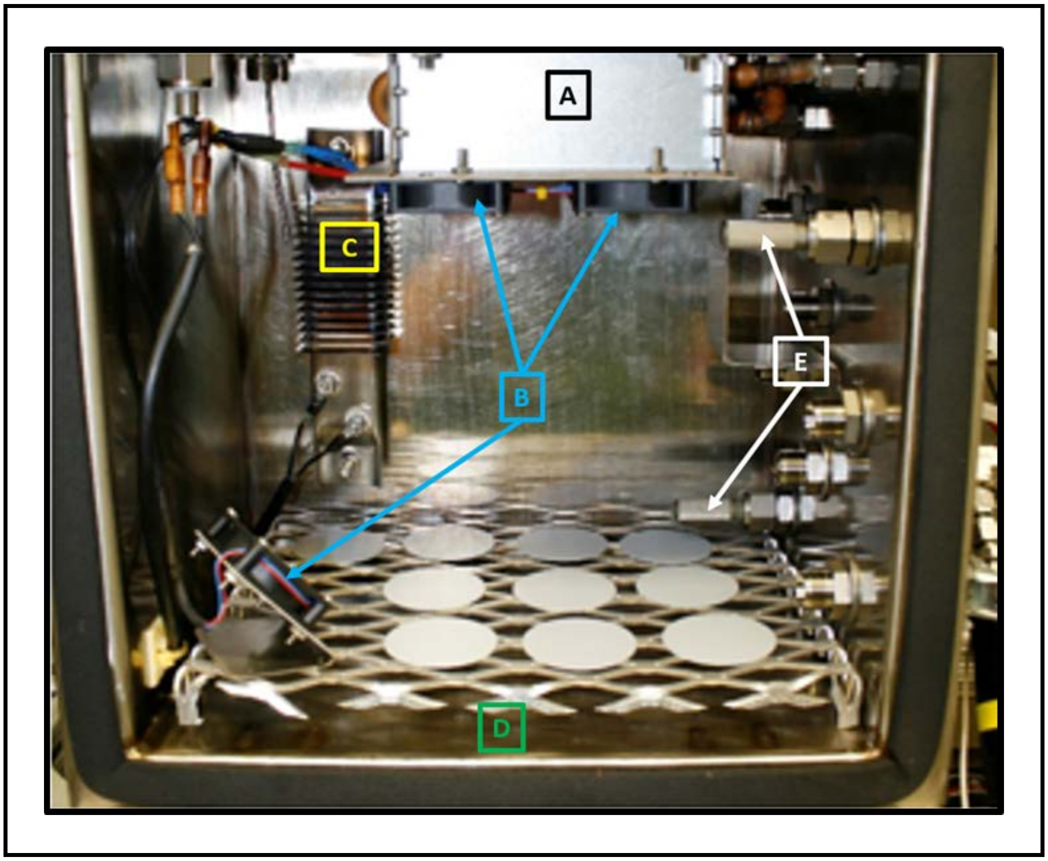


Figure 1. SIV emission chamber.

2.4 Test Procedures

2.4.1 Sampling Schedule and Controls

Each experiment required 7 days to complete. The experimental timing is listed in Table 2 and shown conceptually in Figure 2. Days 5 and 6 were nonwork days (weekends), so no samples could be collected during those times.

Two sets of treatment control samples were collected during each experiment. Aging controls were collected after the 24 h aging period for each substrate, but before any other treatment was performed. Evaporation is hypothesized to be the primary mechanism of loss during the aging time for HD, but there may have been other contributions to the observed losses, such as degradation and reactions with the substrate. Bleach controls were collected after the bleach was applied to characterize the efficacy of the pretreatment for contaminant removal and to provide a comparison point to evaluate the efficacy of humidified CHAD. The decision to collect the first sample after 24 h of humidified CHAD treatment was based on the Myers et al. study in which the authors suggested that at this time point, residual HD quantities would be above instrumental detection limits.[16]

Table 2. Experimental Timing for the Humidified CHAD Experiments

Day	Action
0	Contaminate substrates and start aging process
1	Conduct bleach pretreatment and begin CHAD treatment; extract only aging control samples
2	Remove 24 h CHAD samples from SIV chamber and extract for residual HD
3	Remove 48 h CHAD samples from SIV chamber and extract for residual HD
4	Remove 72 h CHAD samples from SIV chamber and extract for residual HD
7	Remove 144 h CHAD samples from SIV chamber and extract for residual HD

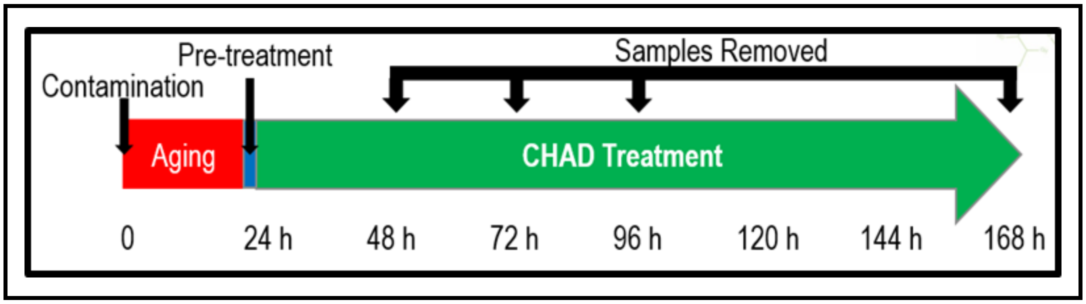
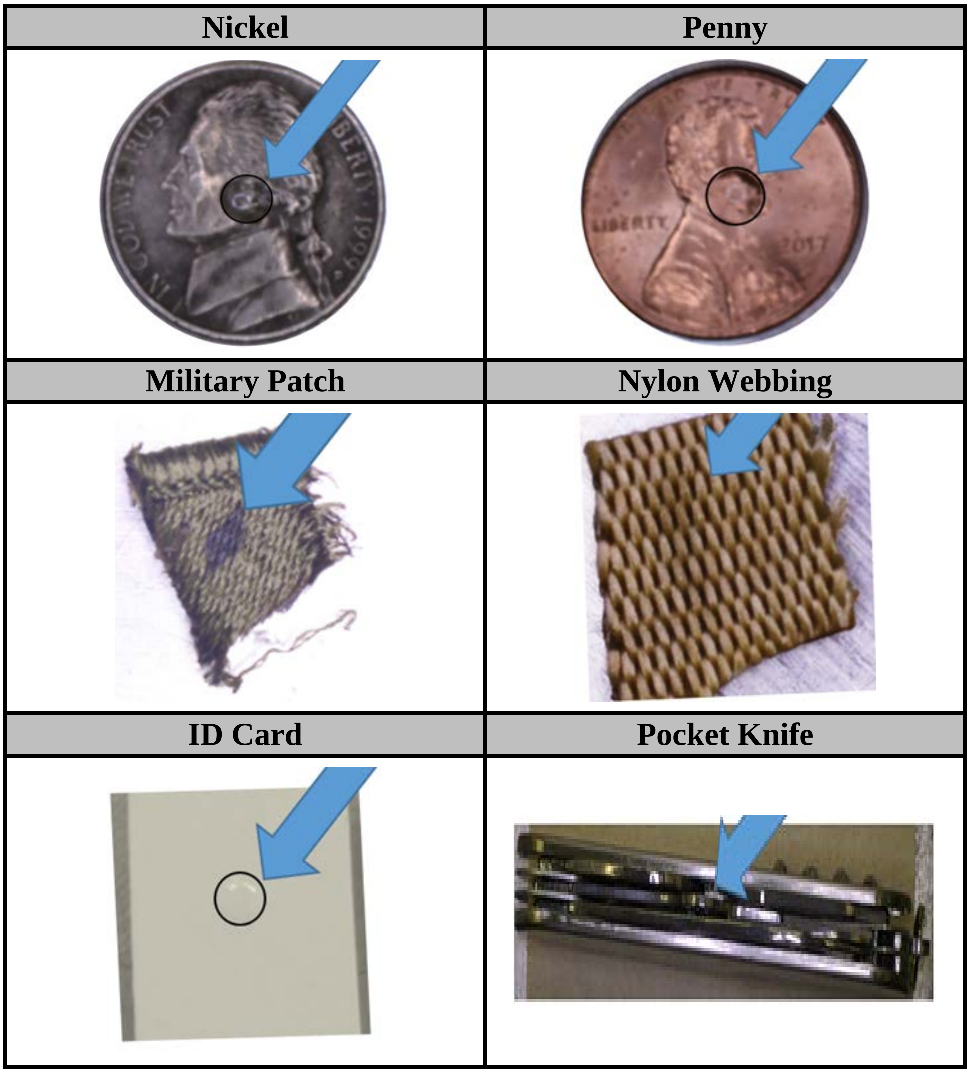


Figure 2. Experimental timing chart.

2.4.2 Substrate Contamination

Each substrate was contaminated with a single 2 µL droplet of CASARM HD using an Eppendorf Repeater M4 pipette (Eppendorf North America; Hauppauge, NY) with a 100 µL tip. The droplet was ejected from the pipette approximately 1 in. above the substrate and allowed to fall onto the substrate.

HD was dosed onto the center of the substrate at the beginning of the experiment. In the case of nickels and pennies, the HD droplet was always placed on the face of the coin in approximately the same location. In the case of the military patch, the HD droplet was always applied to the face of the patch and not the side containing the adhesive. Figure 3 shows the interaction of liquid HD with each of the material substrates approximately 5 min after contamination was performed. The blue arrows indicate the location of the HD for each substrate. For the pennies, nickels, and ID cards, the liquid HD remained as a sessile droplet. For fabrics, such as the nylon webbing and military patches, the HD absorbed into the material within 5 min and appeared to be dry after 24 h of aging. For the pocket knives, the complex features of the substrate made it impossible to see the HD interactions.


Nickel

Penny

Military Patch

Nylon Webbing

ID Card

Pocket Knife

Figure 3. Substrates immediately after contamination.

2.4.3 Substrate Aging

Each sample was aged in an environmental chamber for a period of 24 h before any other treatment was applied. The extended aging time was intended to maximize the interaction of the agent droplet with the substrate and allow for absorption into the bulk of the material, if absorption was possible. The aging time also mimicked the treatment of an agent-contaminated asset in the field, where treatment would likely not be applied immediately after contamination. The environmental chamber in which contaminated substrates were aged was maintained at 20 °C and between 30 and 50% RH during the aging period. In some cases, the HD was still visible on the substrate after 24 h of aging. This is illustrated in Figure 4.

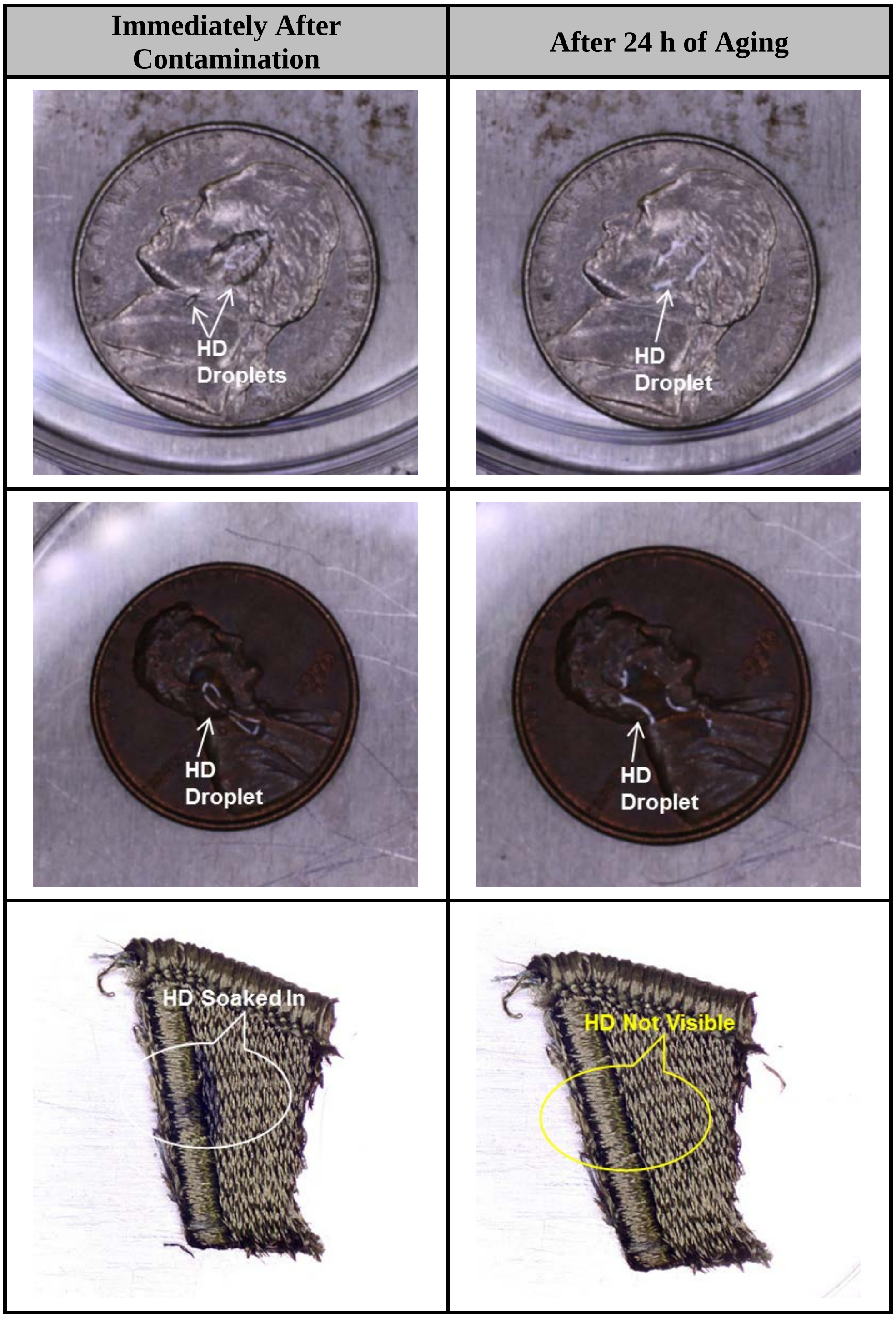


Figure 4. Substrate comparisons. Substrates immediately after contamination (left column) and after 24 h of aging (right column).

2.4.4 Bleach Pretreatment

The pretreatment procedure for contaminated PEs is to scrub them using a sponge or soft-bristle brush with an aqueous solution of sodium hypochlorite (bleach). The bleach is allowed to remain in contact with the PE for at least 5 min and then is rinsed off with clear water.[1] This procedure was mimicked in the laboratory. A 250 µL aliquot of household bleach was applied to the contaminated area of the substrate using a Gilson M250 positive displacement pipette (Gilson, Inc.; Middleton, WI). A small toothbrush was used to lightly scrub the bleach over the face of the contaminated items, moving in a circular (clockwise) pattern for 1 min. After brushing, each substrate was rinsed with deionized (DI) water to remove any remaining bleach. The protocol was to rinse the contaminated face of each substrate with 2 × 20 mL of DI water before the uncontaminated face of the substrate was rinsed with 1 × 20 mL of DI water. Once they were rinsed, the substrates were transferred to the SIV chamber (Section 2.3) for humidified CHAD treatment.

2.5 Determination of Residual HD

This section summarizes the analytical procedures that were used to determine residual HD in the test substrates. Shue et al. published details of this method, and interested readers should consult the referenced report.[21]

2.5.1 Extraction Procedure

Immediately after removal from the SIV chamber, the substrates were placed in an extraction vessel, chloroform was added, and the vessel was capped. The samples were swirled for approximately 30 s and then allowed to sit undisturbed for 60 min. The samples were then swirled again, and an aliquot of the extract was transferred to a clean glass vial. Because the substrates were of various sizes, different extraction vessels and solvent volumes were required. These conditions are summarized in Table 3. The pocket knives were completely submerged in solvent during the extraction process.

Table 3. Extraction Vessels and Solvent Volumes

Test Substrate	Extraction Vessel	Extraction Volume (mL)
Nickel	2 oz glass jar (Scientific Specialties Service, Inc.; Hanover, MD; part no. 370802)	10
Penny		
Military patch	20 mL scintillation vial (Scientific Specialties; part no. B33520)	
Nylon webbing		
ID card		
Pocket knife	50 mL Falcon centrifuge tube (Corning, Inc. Foundation; Corning, NY; part no. 352070)	40

2.5.2 Analysis Procedure

The instrument used in this study was an Agilent model 7000 MS triple quadrupole gas chromatography-mass spectrometry (GC-MS) system (Agilent Technologies; Santa Clara, CA). Sample introduction was performed through a multipurpose sampler (MPS; Gerstel, Inc.; Linthicum, MD). The Gerstel system included a cooled injection system (CIS) with Peltier cooling (TE Technology, Inc.; Traverse City, MI). A CIS-4 baffled deactivated liner, with no packing, was utilized in the Gerstel CIS. Helium was used as a carrier gas at an average linear velocity of 39 cm/s in solvent vent mode. All the experiments used a capillary column with a bonded phase of 5% phenylmethyl polysiloxane (30 m × 0.25 mm) that had a 0.25 µm film thickness. The instrumental and acquisition parameters are summarized in Table 4.

A primary stock of HD was prepared from CASARM HD on a volume/volume basis in chloroform. The concentration of the stock solution was ~10,000 mg/L. The stock solution was stored at ~4 °C in a glass vial with a Teflon-faced silicone cap liner. Working standards were prepared by serial dilution of the primary stock with hexane. The linear calibration range was established to be 2 to 2000 ng/mL. Calibration standards were analyzed with each group of samples along with three levels of continuing calibration verifications (CCVs). Isotopically labeled ^{13}C-HD was added as an IS to all standards and samples.

The average (n = 4 tests) instrumental limit of detection (ILOD) and instrumental limit of quantitation (ILOQ) were calculated using the calibration data from each test.[17] The ILOD value was 0.237 ng/mL, and the ILOQ value was 1.02 ng/mL. (Note: these values are for the extract presented to the instrument.) Sample-based detection and quantitation limits are discussed in Section 3.5.

Table 4. Instrumental Parameters Used in This Study

Injection Parameters	
Mode: solvent vent	Injection volume: 2.0 µL, splitless
Initial temperature: 15 °C	Post injection dwell: 0.00 min
Initial time: 0.40 min	Vent time: 0.30 min
Ramp rate: 12.00 °C/s	Vent flow: 20 mL/min
Final temperature: 260 °C	Vent pressure: 19.5 psi
Hold time: 6.50 min	Purge flow: 50 mL/min at 1.00 min
Inlet pressure: 19.53 psi	NA
Sample pumps: 7	Viscosity delay: 0 s
Solvent B washes: 3	Solvent A washes: 3
Solvent A: hexane	Solvent B: methanol
Oven Parameters	
Initial temperature: 70 °C	Initial time: 0.75 min
Ramp: 35 °C/min to 120 °C, then 10 °C/min to 150 °C, then 40 °C/min to 280 °C for 3.00 min	
Equilibration delay: 0.25 min	NA
Detector Parameters	
Solvent delay: 5.0 min	Gain: 10
Scan type: MRM	Dwell time: 20 ms
MS source: 230 °C	MS quad: 150 °C
MS transfer line: 280 °C	Collision energy: 15 V for quant MRM
He quench gas: 2.25 mL/min	N_2 collision gas: 1.5 mL/min
MRMs for HD: 109 > 63,[a] 158 > 109	MRMs for IS: 110 > 64,[a] 160 > 110

NA: not available.
MRM: multiple reaction monitoring.
MS: mass spectrometer.
[a] MRM used for quant.

3. RESULTS AND DISCUSSION

3.1 Summary

Humidified CHAD treatment provided significant reduction of extracted HD from PEs made of fabric and metal, regardless of the presence of any complex features. The amount of HD extracted from military patches, nylon webbing, and pocket knives was reduced to below the method limits of detection (MLODs) after 144 h of humidified CHAD treatment. In the case of coinage, the amount of HD extracted was reduced to below the MLOD after 24 h of humidified CHAD treatment. Humidified CHAD was not successful in decontaminating ID cards; the average amount of HD extracted from ID cards after treatment was 86,700 ng.

3.2 Prestudy Tests

A series of prestudy tests was conducted to verify whether the residual HD method (Section 2.5) was suitable for use on the substrates evaluated during this study. Results from these prestudy tests are summarized in Section 3.2.

3.2.1 Interference Tests

The SD2ED provided the chemical biological defense community with robust test methodologies for determining the amount of chemical contaminant residue after a treatment process.[17] The most common post-treatment evaluations available in the SD2ED are the total remaining contaminant, chemical agent detector paper response, contact transfer, and vapor emission tests. All of these post-treatment evaluations include the assessment of contaminant remaining in or on a test panel. This assessment is performed by placing the panel in solvent to extract the contaminant from the material. An aliquot of the extraction solvent is analyzed using the appropriate chromatographic technique.

Analyses of samples are performed on analytical instrumentation such as GC-MS systems. Confidence in a measured value depends on the ability of the analytical instrument to selectively detect and quantify the analyte of interest. Selective detection and quantification is required to ensure that the analyte is detected without bias caused by the presence of other compounds or components within a sample. The presence of these bias-inducing additional components in a sample is referred to as interference. An *interferent* is defined as anything that prohibits the confident measurement of the analyte of interest during analysis. Interference evaluation is a proactive approach to determine whether the standard experimental extraction solvents are compatible with the program materials and decontaminants prior to programmatic decontamination studies. Interference evaluations are performed before the analysis of samples generated from a decontamination project to ensure that an analyte is detected and quantified with high confidence.

An interference evaluation was performed only on the ID card substrate because previous evaluations examining metal substrates and nylon webbing indicated there would be no significant inferences. In addition, preliminary extractions of the ID card revealed that the ID card delaminated during the extraction process. Thus, there was a possibility that interferents had been introduced into the extract. An interference evaluation was performed in accordance with the standard protocol but with one modification:[17] only the lowest standard level (10 ng/mL HD) was evaluated during the interference evaluation.

In all of the cases, when the extract was diluted 20-fold, the responses were within 20% of the expected concentration, which indicated that no significant positive or negative bias was present when the ID card was extracted. Example chromatograms from the interference evaluation are illustrated in Figure 5. Because the analytical protocol included the addition of ^{13}C-HD as an IS to all of the samples, the comparison of blanks (top panel, Figure 5) shows a peak for HD in the solvent blank and ID card extract. Both material types were analyzed without the addition of the ^{13}C-HD (middle panel, Figure 5), and in both cases, there was no significant positive interference in either the solvent blank or ID card extract. While there were small peaks in the time window where HD eluted, the heights of the interfering peaks were over 600 times less than that of the lowest HD standard response (lower panel, Figure 5).

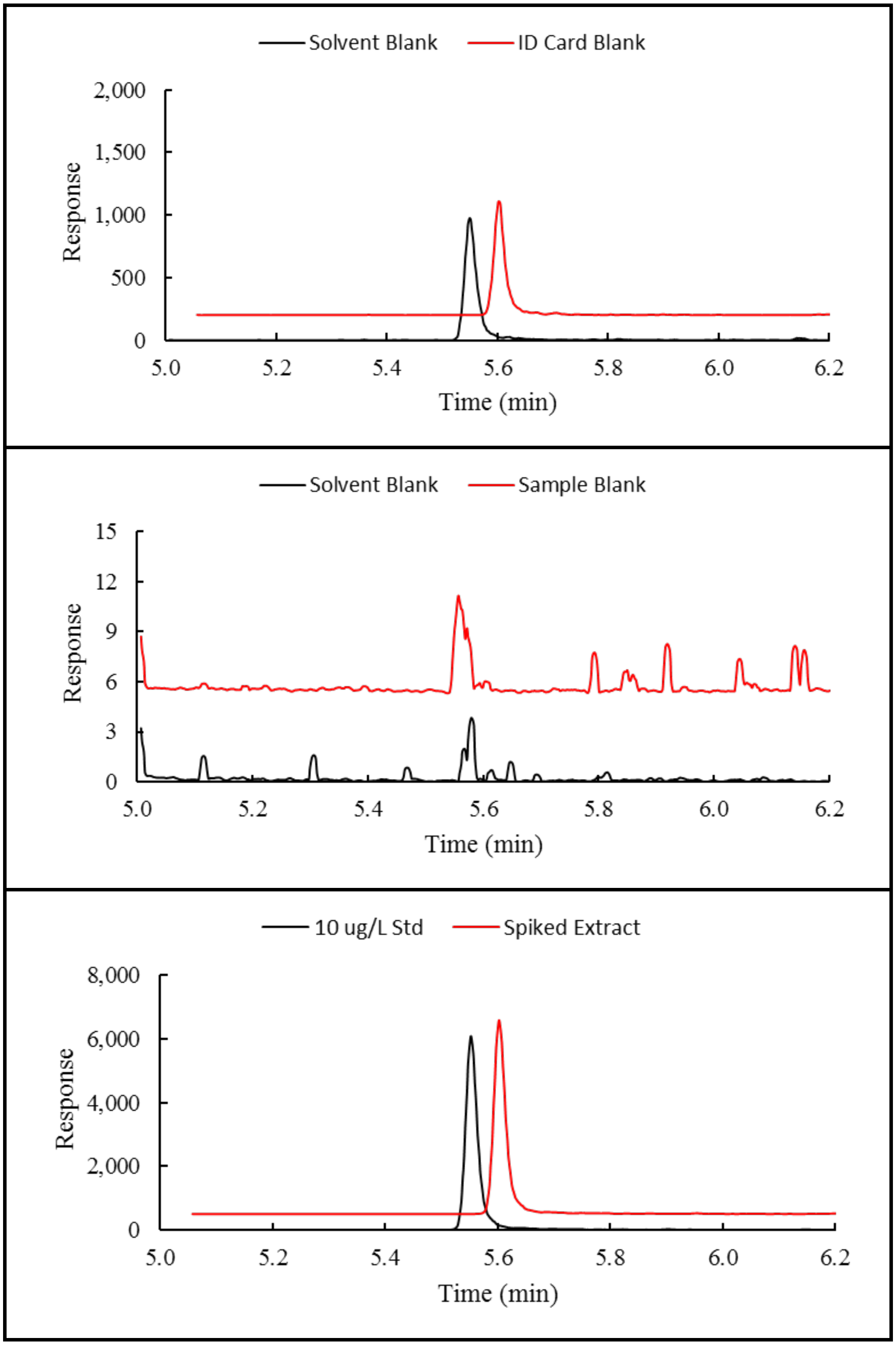


Figure 5. Example chromatograms from the interference evaluation. Solvent blank and ID card extract (upper panel), solvent blank and ID card extract without IS (middle panel), and 10 µg/L of HD standard and spiked ID card extract (bottom panel). For clarity, the chromatograms of the ID card extract are offset horizontally and vertically.

3.2.2 Solvent Recovery Tests

A solvent recovery test was conducted to evaluate the apparent increase in recovery of HD from the substrates. The substrates were spiked with a single 2 µL drop of neat HD and aged for 1, 60, or 1440 min, as described in Section 2.4.3. Once aged, the contaminated substrates were removed from the conditioning chamber and immediately extracted using the extraction protocol described in Section 2.5.1. Each treatment variable was conducted in triplicate. In addition, five dose confirmation samples (DCSs) were prepared (i.e., 2 µL of neat HD was introduced directly into the solvent). The 1440 min samples were the aging controls generated during the actual test.

The results are illustrated in Figure 6 and summarized in Table 5. The recoveries across all of the substrates were quantitative when the samples were aged for 1 or 60 min. When the contaminated substrates were aged for 1440 min before extraction, apparent recoveries were significantly lower and more variable. It is not known whether these lower apparent recoveries resulted from evaporation, degradation, reaction with the substrate, or a combination of these factors. Mundis et al. reported 11% HD recovery from nylon webbing after 60 min of aging (ambient conditions).[15] In their study, HD was applied as a single 1 µL drop, but they did not report any details on sample extraction and analysis, so it is difficult to compare their results with ours. In our study, average recovery of HD from nylon webbing was 96% when the samples were aged for 60 min before extraction.

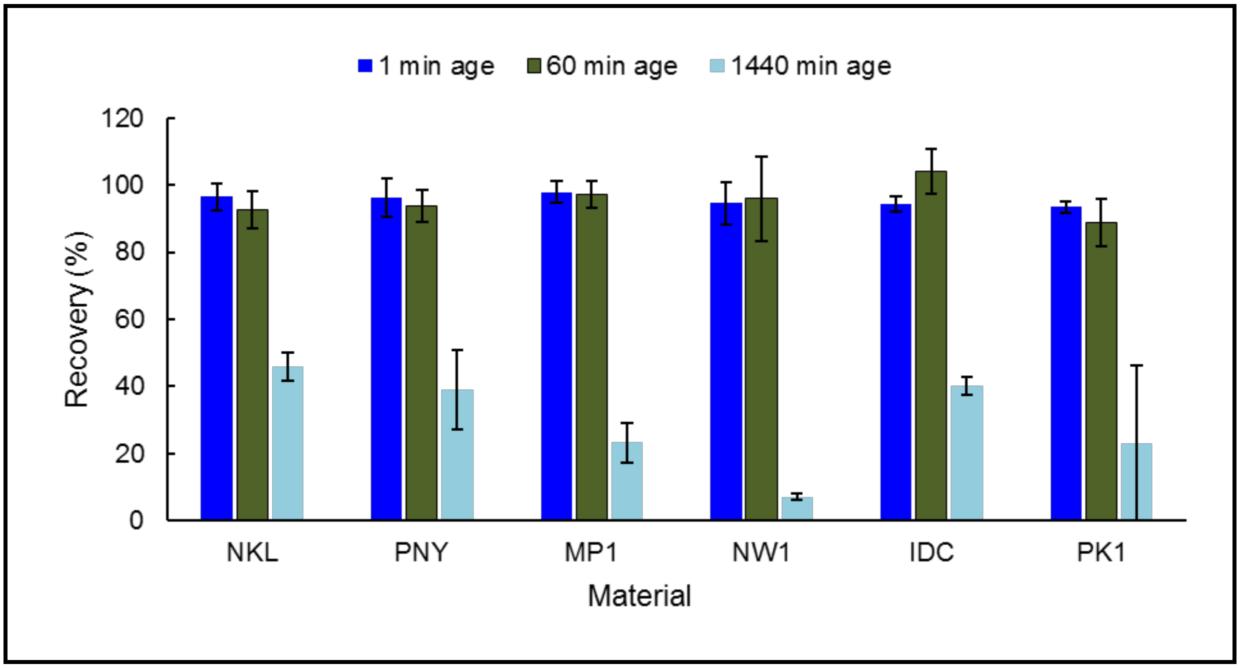


Figure 6. Apparent recovery as a function of aging time. Vertical black bars are ±1 standard deviation (SD).

Table 5. Average Apparent Recovery from Test Substrates

Material	**Age Time (min)**	**Average Recovery (%)**	**SD (%)**	**RSD (%)**
Nickel	1	96.5	4.00	4.14
	60	92.5	5.56	6.01
	1440[a]	45.9	4.03	8.77
Penny	1	96.3	5.66	5.88
	60	93.8	4.72	5.04
	1440	38.9	11.88	30.53
Military patch	1	97.9	3.27	3.34
	60	97.3	4.09	4.20
	1440	23.3	5.90	25.30
Nylon webbing	1	94.6	6.35	6.71
	60	96.0	12.61	13.1
	1440	7.04	0.935	13.3
ID card	1	94.3	2.21	2.35
	60	104	6.65	6.38
	1440	40.2	2.55	6.33
Pocket knife	1	93.4	1.55	1.66
	60	88.9	6.97	7.83
	1440	23.1	23.11	100

RSD: relative standard deviation.

[a] The 1440 min samples were the aging controls generated during the actual test.

3.3 Environmental Test Conditions

The environmental test conditions (i.e., temperature and RH) were controlled and monitored in the SIV chamber during the experiments. Readings were collected every 30 s during each test. Average (*n* = 8640 or 17,280) test conditions are summarized in Table 6, and the data are illustrated in Figures 7–10. Temporary perturbations in chamber temperature and RH can be observed at 24, 48, 72, and 144 h. These perturbations correspond to the times when the SIV chamber door was opened to remove samples for solvent extraction. The environmental conditions for all of the experiments were consistent with those of the humidified CHAD treatment.

Table 6. Average Environmental Conditions during Each Test

Test ID	Substrates in Test	Average Temperature (°C)	Average RH (%)
CHDP01	Nickels and pennies	78.8 ± 0.73	20.5 ± 1.57
CHDP02	Military patches and nylon webbing	79.2 ± 0.76	21.4 ± 2.61
CHDP03	ID cards	79.3 ± 0.57	19.1 ± 1.46
CHDP04	Pocket knives	79.7 ± 0.52	17.2 ± 1.43

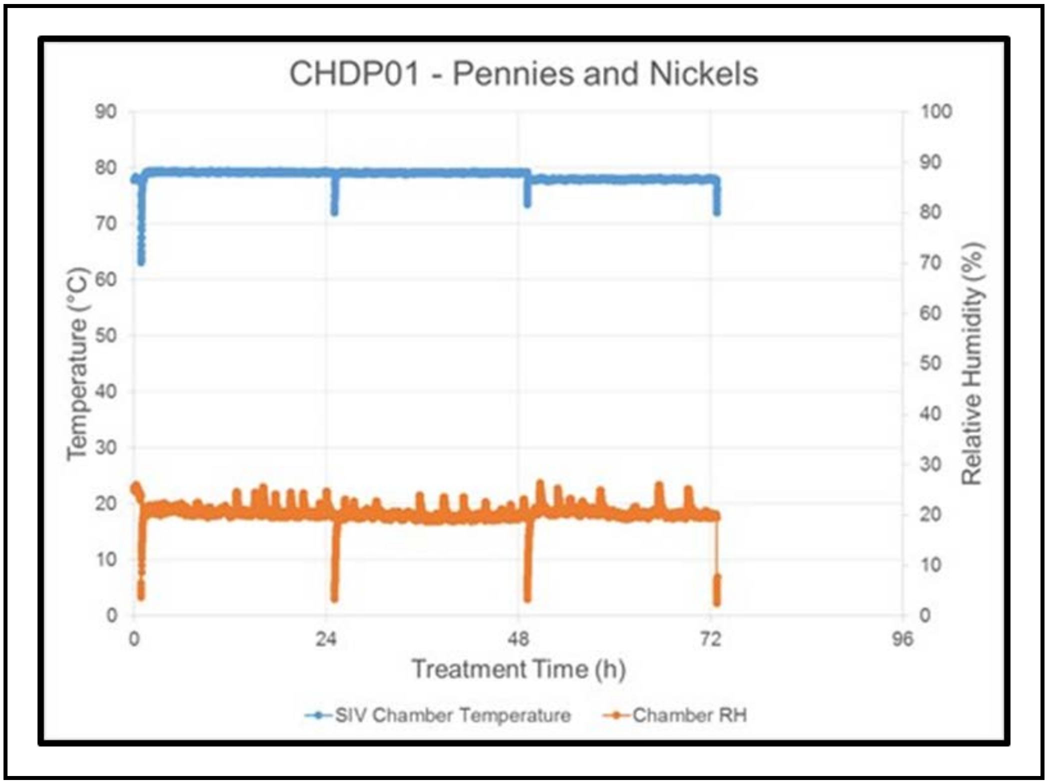


Figure 7. Environmental conditions during test CHDP01. This test was extended only to 72 h of humidified CHAD treatment.

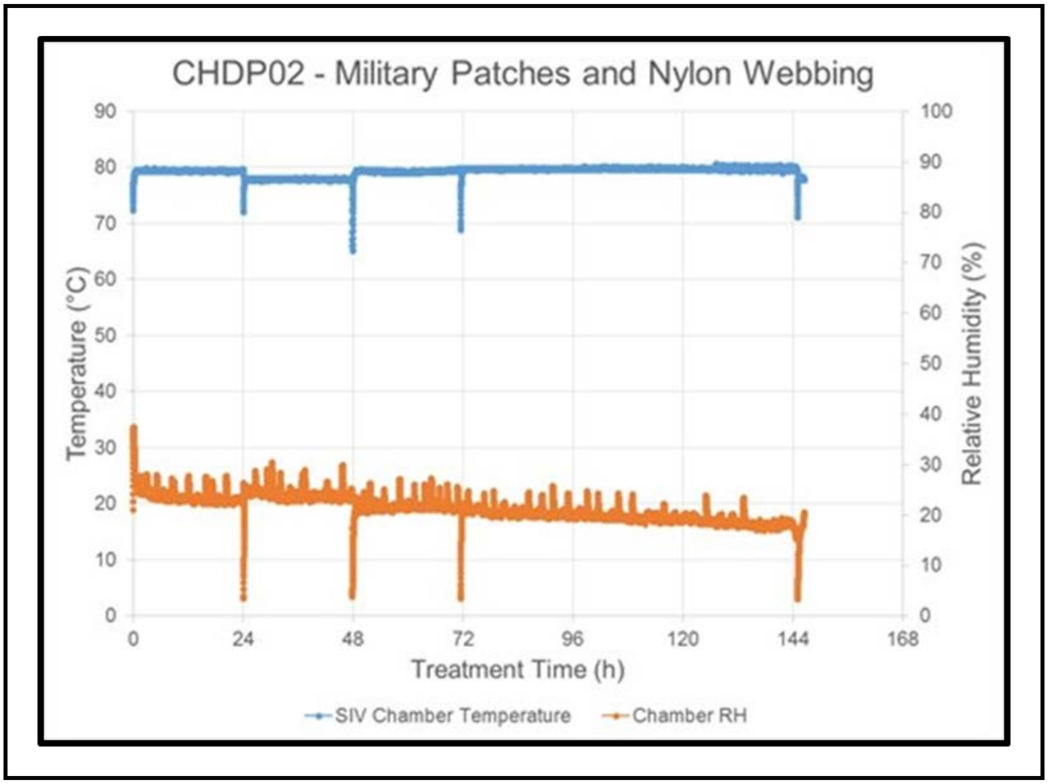


Figure 8. Environmental conditions during test CHDP02.

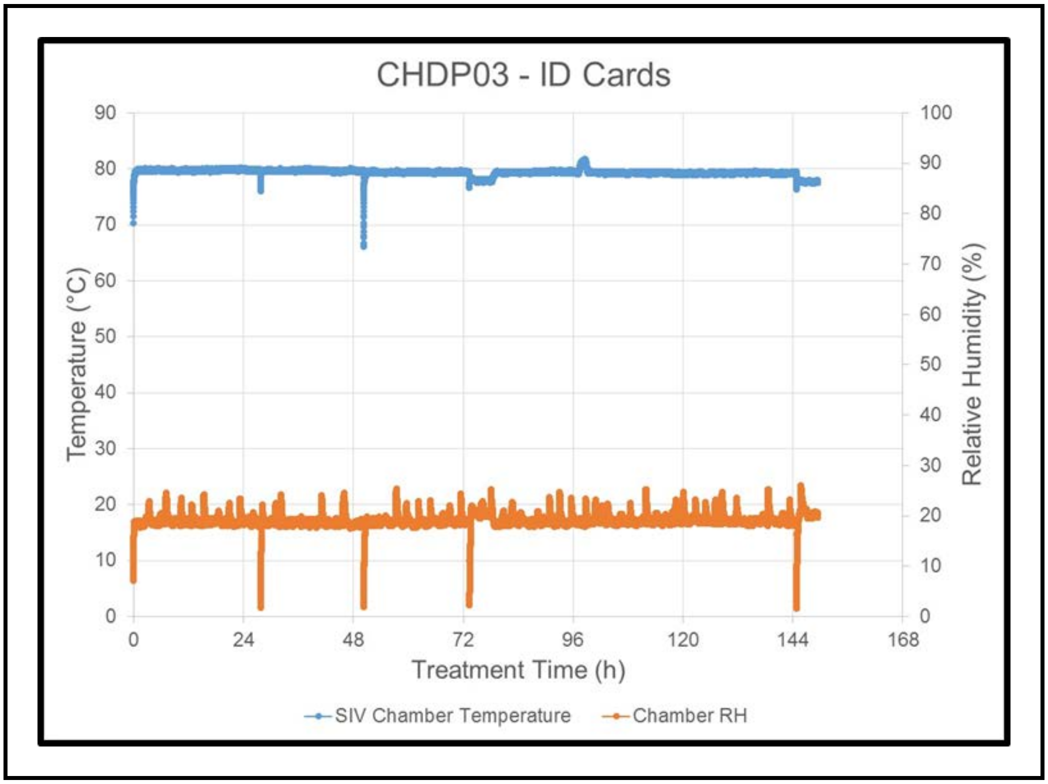


Figure 9. Environmental conditions during test CHDP03.

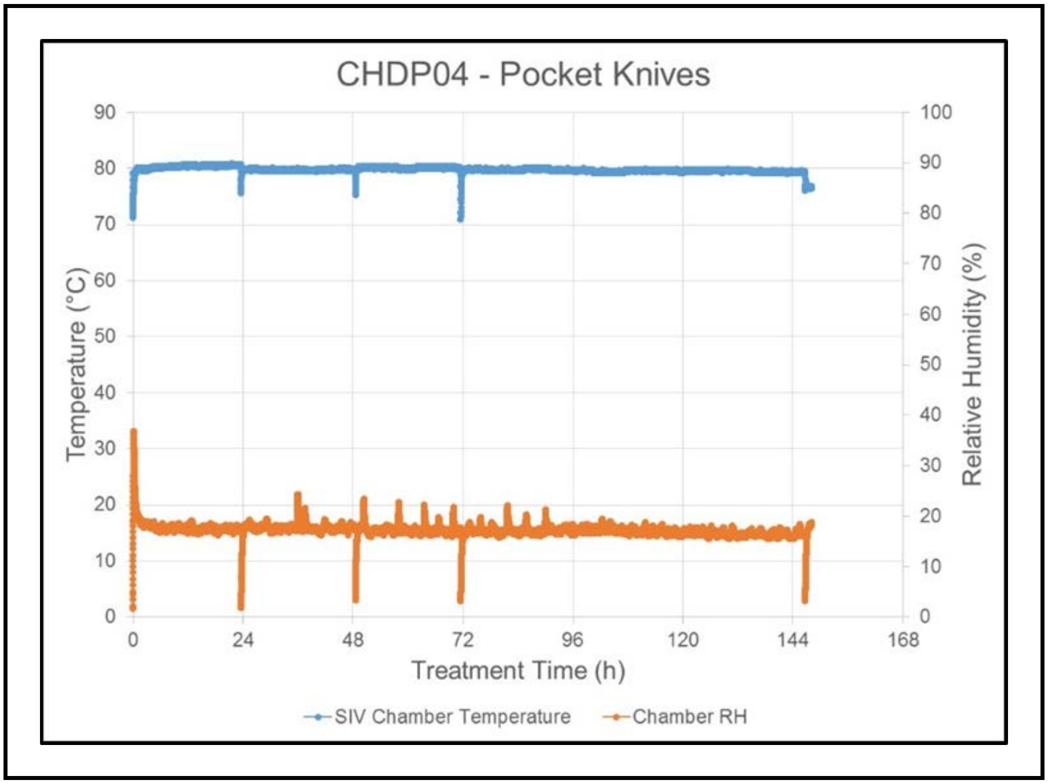


Figure 10. Environmental conditions during test CHDP04.

3.4 Quality Control Samples

During this study, five replicate DCSs were prepared and analyzed during each test. There were no significant differences in DCS response between tests, so an overall average (*n* = 20) value was used. The average DSC response was 2,410,000 ng of HD, with an RSD of 6.69%. This result demonstrates that the HD was dispensed consistently throughout the study.

In addition to the DCSs, additional quality control samples were analyzed concurrently with test samples. Thirty-eight solvent-only method blanks were analyzed with the test samples. Thirty-one method blanks were non-detects for HD, whereas seven method blanks yielded a trace response near the ILOD value. These results show that there was no significant carry-over during the analyses. Thirty-eight CCV samples at three concentration levels were analyzed concurrently with the test samples. Overall average CCV recoveries were 98.9, 100, and 99.9% for the 10, 150, and 750 ng/mL HD CCVs, respectively. These CCV results show that the analyses were under control, and calibrations were not changing.

3.5 Humidified CHAD Test Results

Overall MLOD and method limit of quantitation (MLOQ) values were calculated using the extraction volumes (Section 2.5.1, Table 3), instrumental limits (Section 2.5.2), and sample dilution factor. Coinage, military patch, and nylon webbing samples have an MLOD of 2.4 ng of HD and an MLOQ of 10.2 ng of HD. ID cards have an MLOD of 48 ng of HD and an MLOQ of 204 ng of HD. The values are higher for the ID cards because the lowest dilution analyzed was a 20-fold dilution of the original extract. The extract of the ID card had to be diluted a minimum of 20-fold to prevent interference during the analysis (Section 3.2.1). Pocket knife samples have an MLOD of 9.5 ng of HD and an MLOQ of 40.8 ng of HD.

It is not known whether the removal of HD results from evaporation, degradation, reaction with substrates, or a combination of these processes. The mechanism of removal is the subject of another study and will be reported at a later date.

3.5.1 Removal from Coinage

The results from the decontamination of HD from coinage are illustrated in Figures 11 and 12. The experiment conducted with the coinage only went through 72 h of humidified CHAD because of power issues in the building. There were no significant differences in decontamination efficiency between the nickels and pennies. On average, 58.4% of the applied HD was removed during the aging process, which is a 0.4 log reduction. On average, the bleach pretreatment removed an additional 38.5% of the applied HD, which is a combined 1.5 log reduction. Overall, an average of 96.9% of the applied HD was removed during the aging and bleach pretreatment steps. In both coinage materials, 24 h of humidified CHAD treatment resulted in residual HD values below the MLOD of 2.4 ng of HD.

In reported studies examining the evaporation of HD from nonporous, inert surfaces, reported evaporation rates ranged from 2.7 to 16 µg/min.[22–24] These rates were obtained at multiple temperatures (15, 25, and 30 °C) on multiple matrices (glass, aluminum, and stainless steel) with varying drop sizes (1, 3.5, and 6 µL). The reported studies concluded that environmental factors (such as temperature and air flow) influenced evaporation, as expected, with higher temperatures and airflows producing higher evaporation rates. In addition, the evaporation rates were affected by drop size; the larger drops yielded higher evaporation rates because of the increased surface area. In our study, 58% of the HD was removed from the coinage during the aging process, which is an overall apparent evaporation rate of ~1 µg/min, when evaporation is assumed to be the primary mechanism of removal. Taking into account the lower air flow and temperature used during the aging process, the apparent evaporation rate observed in our study is generally consistent with reported values.

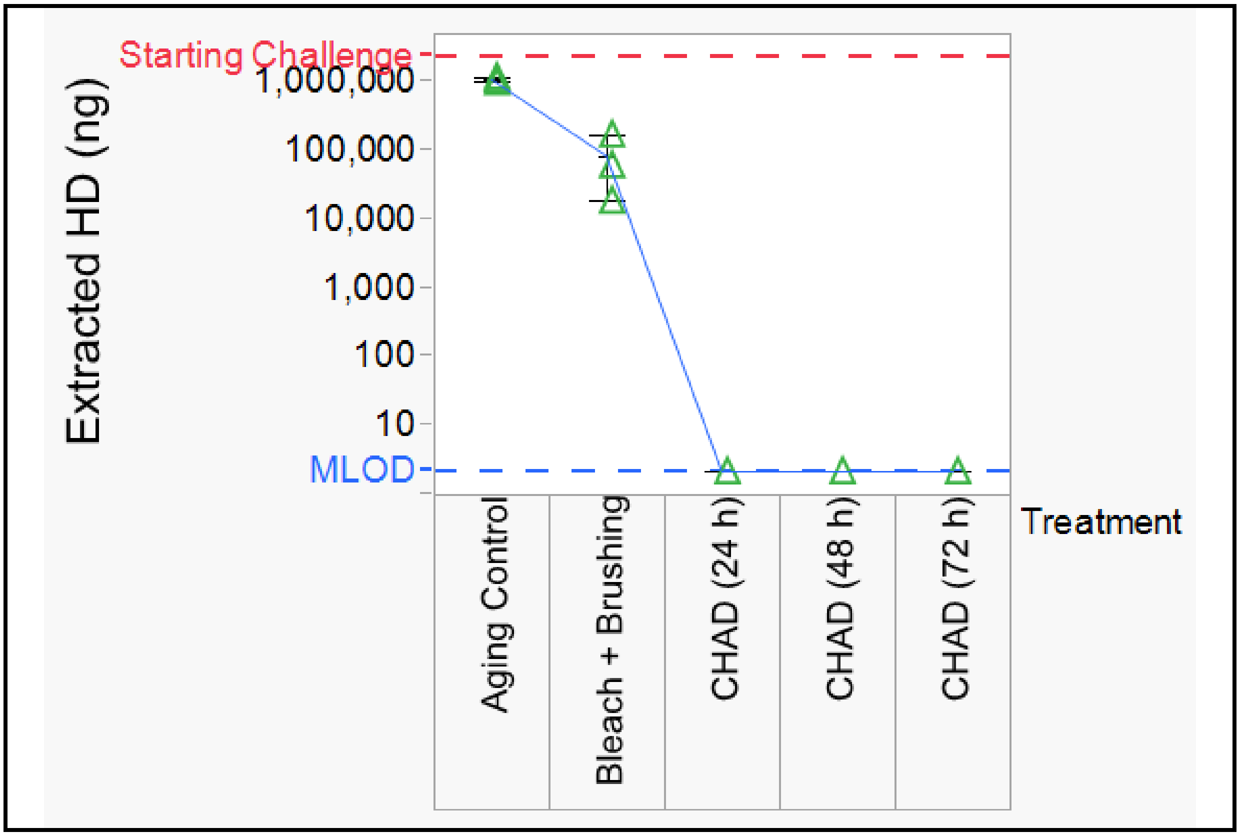


Figure 11. Decontamination of nickels.

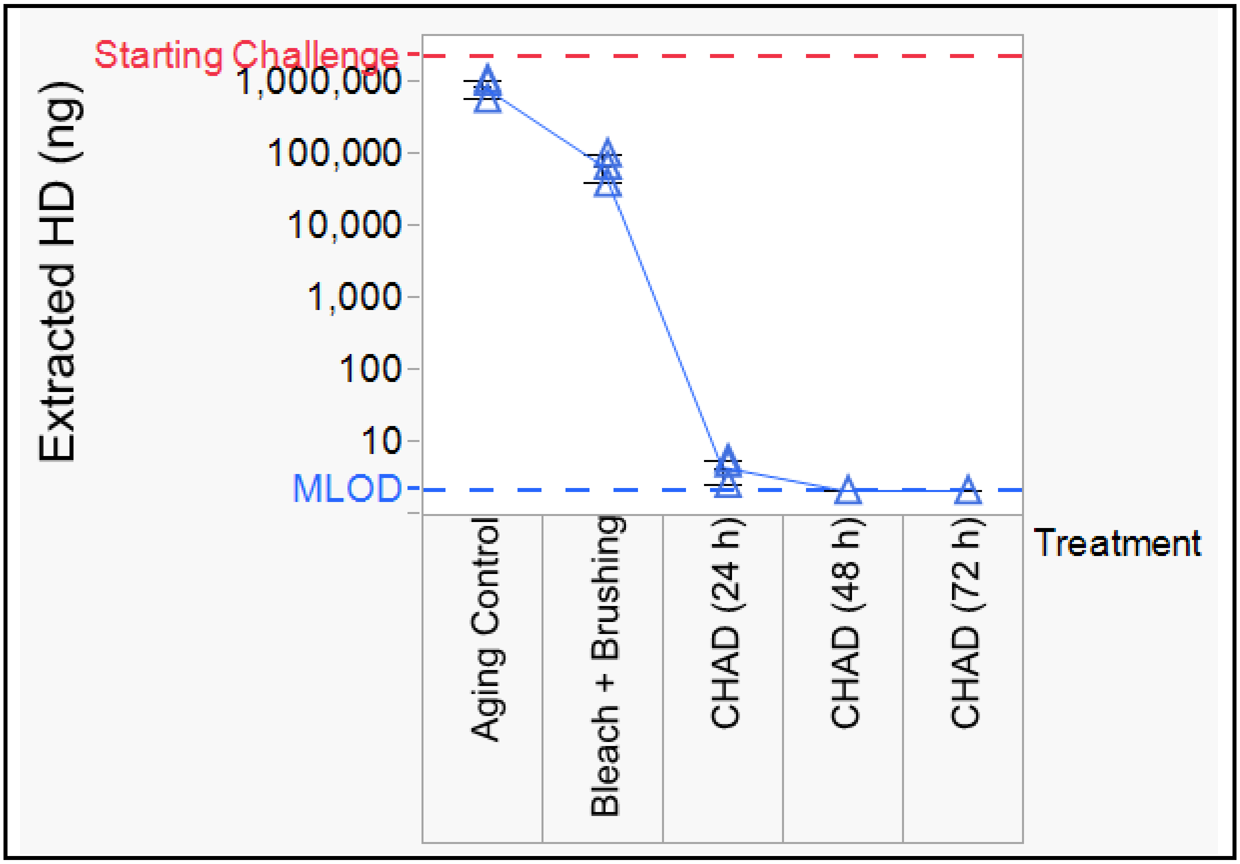


Figure 12. Decontamination of pennies.

3.5.2 Removal from Military Patch

The results of HD decontamination from military patch material are illustrated in Figure 13. On average, 74.3% of the applied HD was removed during the aging process, which is a 0.6 log reduction. The bleach pretreatment removed an additional 17.9% of the applied HD, which is a combined 1.0 log reduction. Overall, an average of 92.2% of the applied HD was removed during the aging and bleach pretreatment steps. In the first 24 h, humidified CHAD removed an additional 7.81% of the applied HD. The residual HD values after 48 and 72 h of humidified CHAD resulted in HD values below the MLOQ of 10.2 ng of HD, whereas 144 h of humidified CHAD treatment resulted in HD values below the MLOD of 2.4 ng of HD.

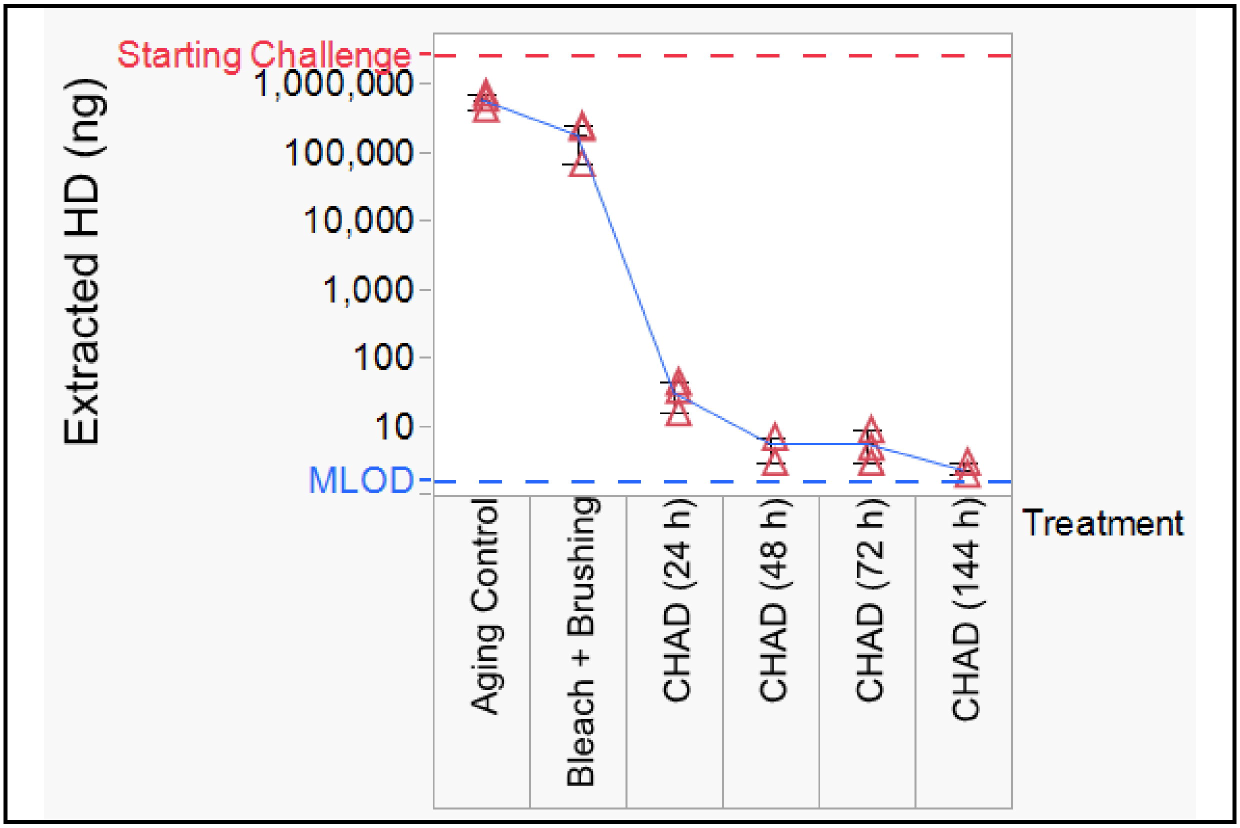


Figure 13. Decontamination of military patches.

3.5.3 Removal from Nylon Webbing

The results from the decontamination of HD from nylon webbing material are illustrated in Figure 14. On average, 92.2% of the applied HD was removed during the aging process, which is a 1.1 log reduction. The bleach pretreatment removed an additional 7.73% of the applied HD, which is a combined 3.5 log reduction. Overall, an average of 99.9% of the applied HD was removed during the aging and bleach pretreatment steps. In the first 24 h, humidified CHAD treatment removed an additional 0.025% of the applied HD. The residual HD values after 48 and 72 h of humidified CHAD resulted in variable results, with HD values ranging from below the MLOD of 2.4 ng of HD to just over the MLOQ of 10.2 ng of HD. The residual HD values after144 h of humidified CHAD treatment resulted in HD values below the MLOD of 2.4 ng of HD.

In their study, Myers et al. pretreated nylon webbing with soapy water before using humidified CHAD treatment.[16] The soapy water was less efficacious than the bleach step utilized in our study. On average, the combined aging and bleach pretreatment resulted in a 3.5 log reduction in residual HD, whereas the combined aging and soapy water pretreatment resulted in a 1.1 log reduction in residual HD. The soapy water pretreatment did not remove a significant amount of HD; the majority of the HD was removed during the aging process.

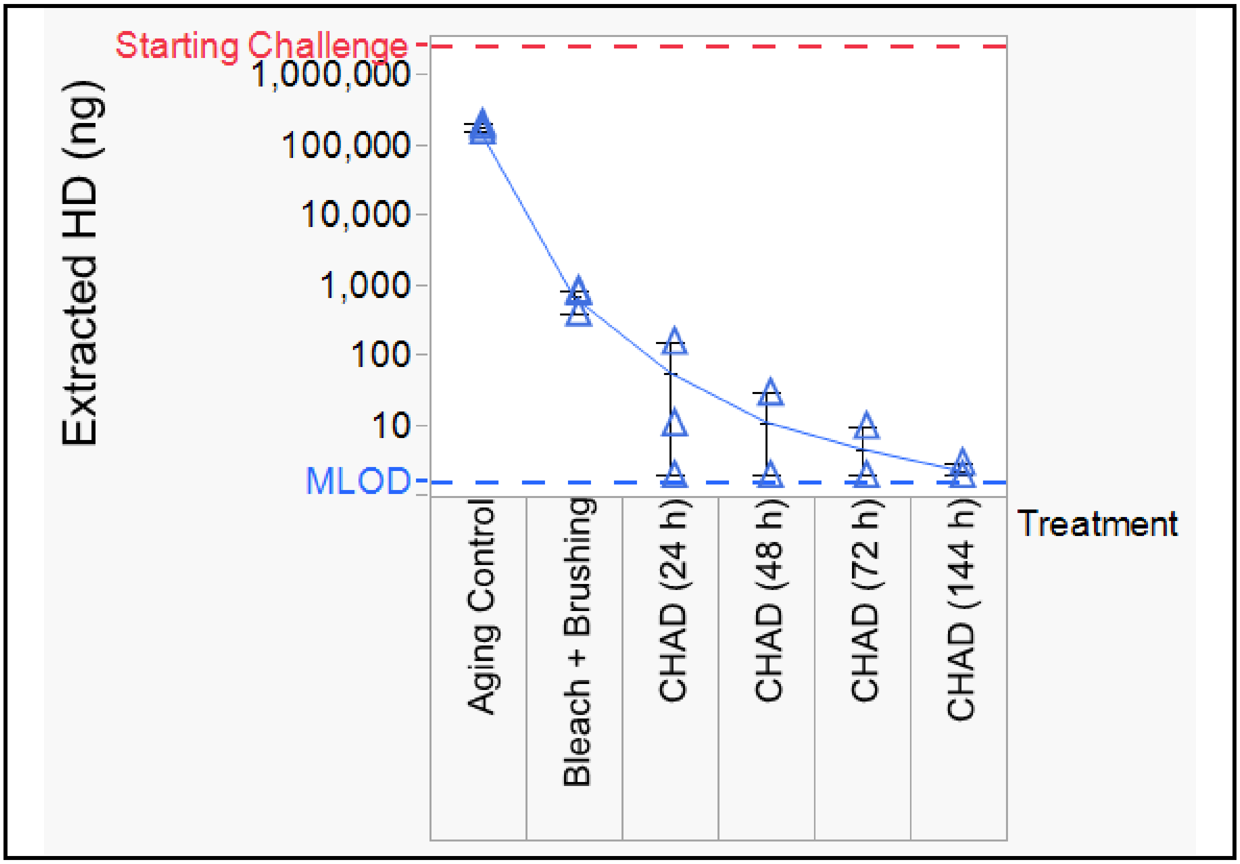


Figure 14. Decontamination of nylon webbing.

3.5.4 Removal from ID Card

The results from the decontamination of HD from ID card material are illustrated in Figure 15. On average, 62.3% of the applied HD was removed during the aging process, which is a 0.4 log reduction. The bleach pretreatment removed an additional 6.64% of the applied HD, which is a combined 0.5 log reduction. Overall, an average of 68.9% of the applied HD was removed during the aging and bleach pretreatment steps. In the first 24 h, humidified CHAD treatment removed an additional 25.0% of the applied HD, which is a combined 1.2 log reduction. Further treatment removed 1.39, 0.379, and 0.681% of applied HD for 48, 72, and 144 h of humidified CHAD treatment, respectively. The residual HD values never reached the MLOD or MLOQ levels. The average residual HD after 144 h of humidified CHAD treatment was 86,700 ng of HD.

During the extraction process, the ID card swelled and delaminated (Figure 16). This swelling and delamination is thought to be a contributing factor to the interference observed when analyzing extracts without any further dilution. In addition, once the layers delaminated, there was a noticeable area on the inner (white) layer that appeared to be melted. This area was associated with the location of the HD droplet. Sulfur mustard is known to be a plasticizer, and the observed interaction of HD with the inner layer is thought to be a contributing factor to the observed lack of efficacy of humidified CHAD against HD on this substrate.

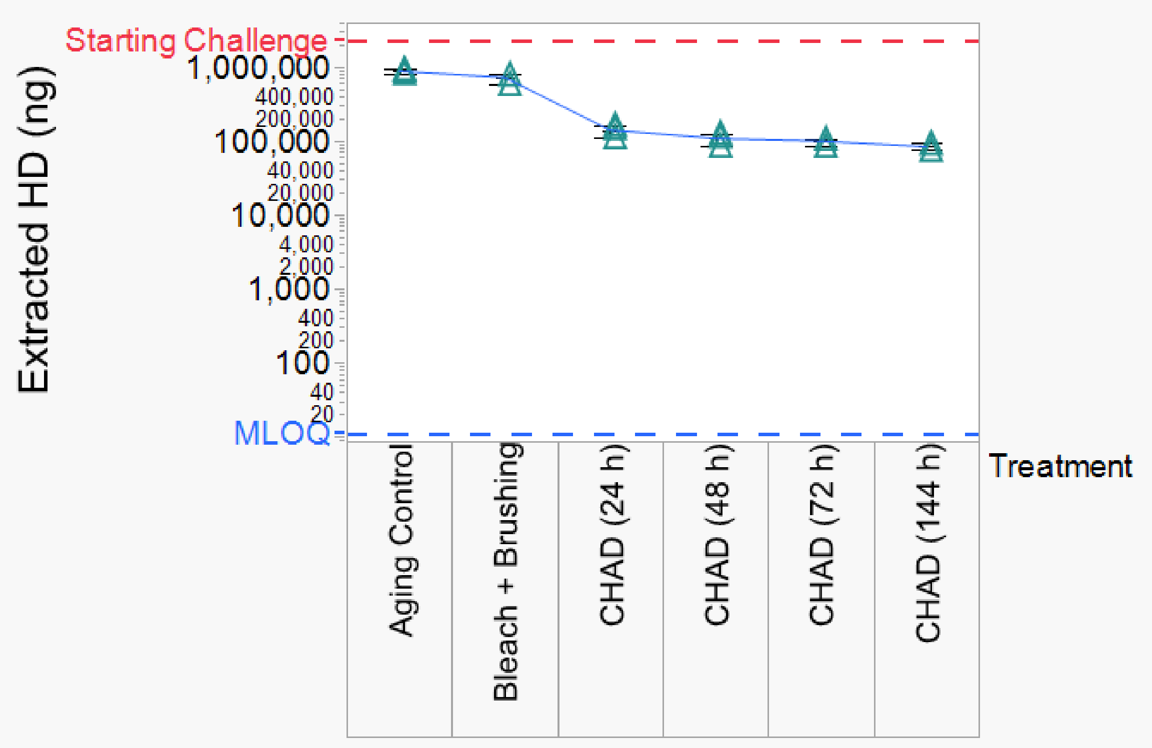
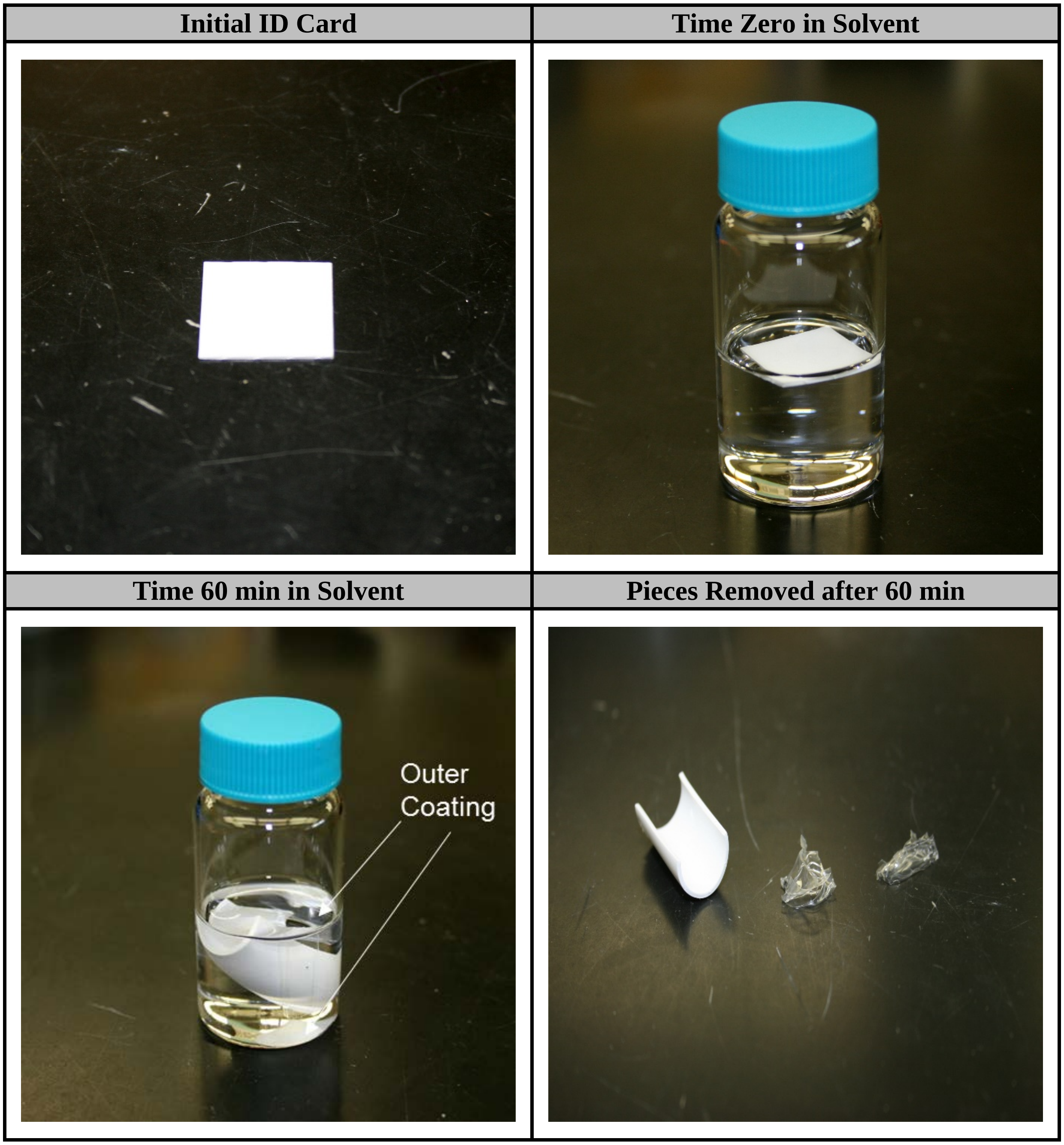


Figure 15. Decontamination of ID cards.



Initial ID Card

Time Zero in Solvent

Time 60 min in Solvent

Pieces Removed after 60 min

Figure 16. Delamination of ID card during the extraction process.

3.5.5 Removal from Pocket Knife

The results from the decontamination of HD from pocket knives are illustrated in Figure 17. On average, 77.4% of the applied HD was removed during the aging process, which is a 0.6 log reduction. The bleach pretreatment removed an additional 16.5% of the applied HD, which is a combined 1.2 log reduction. Overall, an average of 93.9% of the applied HD was removed during the aging and bleach pretreatment steps. The residual HD values after 24, 48, and 72 h of humidified CHAD treatment resulted in HD values at the MLOD (9.5 ng) and MLOQ (40.8 ng) levels, with variability within treatments. The residual HD values after 144 h of humidified CHAD treatment resulted in HD values at the MLOD (9.5 ng) level.

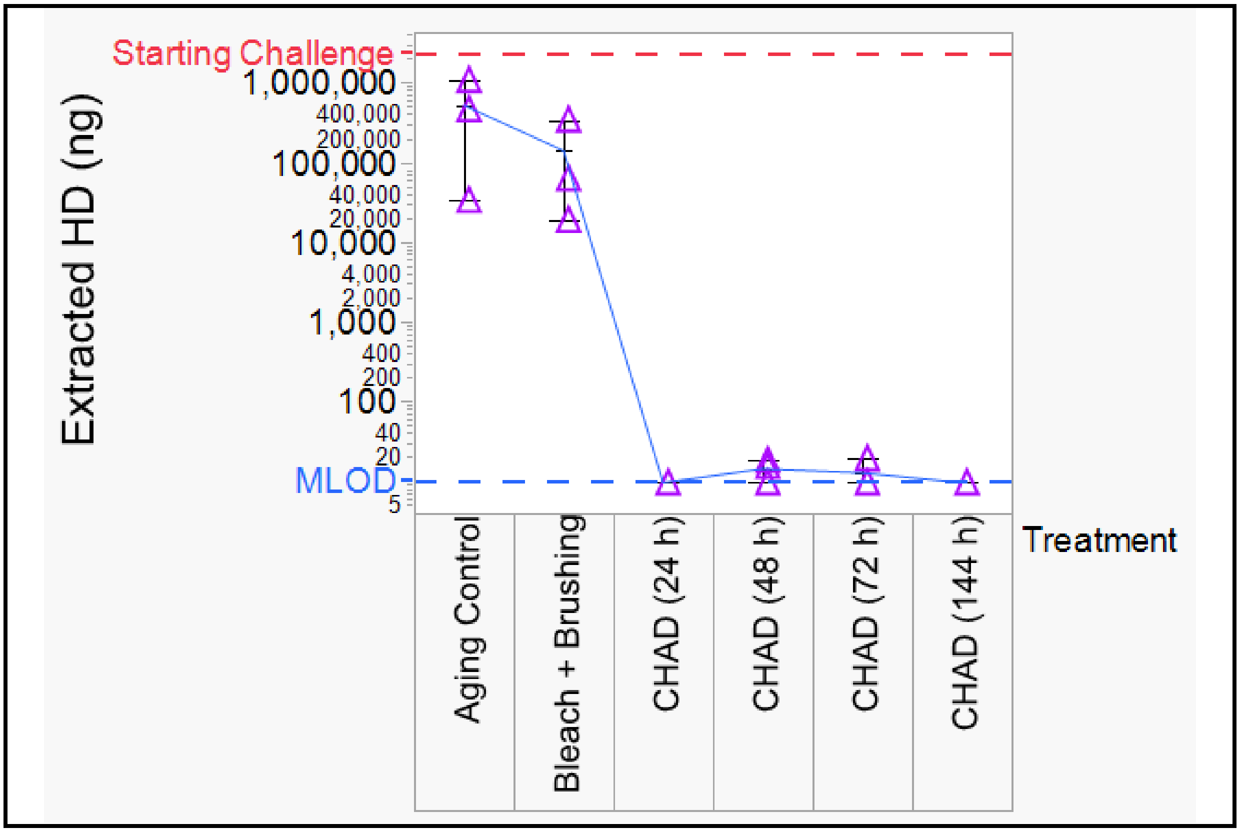


Figure 17. Decontamination of pocket knives.

3.5.6 Dynamic Chemical Extraction (DCE)

DCE, a novel extraction protocol, was applied to select substrates to further investigate whether residual HD was being retained in the substrates.[25] The DCE approach relied on extraction being conducted under infinite sink conditions and generation of cumulative release curves (CRCs). Data from the CRCs were evaluated using nonlinear sorption isotherm models developed at the U.S. Department of Agriculture (Washington, DC) to estimate maximum release (recovery) of residual HD.[26–28]

The select substrates (military patch, nylon webbing, ID card, and pocket knife) were contaminated with single 2 µL drops of neat HD and allowed to age for 24 h before extraction was started. First, chloroform or *n*-hexane was added to the sample jar containing the substrate. Then, a stir flea was added, and the solution was stirred. At various time points over the course of 48 h, 0.5 mL sample aliquots were removed and analyzed for residual HD. A fresh 0.5 mL aliquot of solvent was added after each sample was removed to maintain infinite sink conditions.

The CRCs are illustrated in Figures 18–21, and the maximum recovery estimates are summarized in Table 7. The DCE experiments conducted with chloroform (pocket knives excepted) exhibited decreasing HD concentration after the first several extractions. This suggests that the residual HD had been completely extracted, and the addition of fresh solvent had diluted the extraction solvent. The amount of HD extracted by the DCE protocol using chloroform for the ID cards and pocket knives was similar to that extracted when using the standard protocol. The DCE extractions using chloroform for the military patches and nylon webbing resulted in values less than the residual HD extracted using the standard extraction protocol. The use of *n*-hexane as an extraction solvent did not result in the extraction of more residual HD, when compared with the standard extraction protocol. Overall, the use of a more exhaustive extraction protocol did not result in more HD being extracted.

Table 7. DCE and Standard Extraction Comparison

Substrate	**Dynamic Chemical Extraction**		**Average Recovery Using Standard Extraction (% of Applied)[b]**
	Estimated Maximum Recovery (% Applied)[a]		
	Chloroform	***n*-Hexane**	
Military patches	7.7	7.8	23.3
Nylon webbing	4.2	6.3	7.04
ID cards	32	6.1	40.2
Pocket knives	31	2.2	23.1

[a] Estimated using a nonlinear Langmuir model.

[b] Substrates extracted with chloroform using procedure in Section 2.5.1.

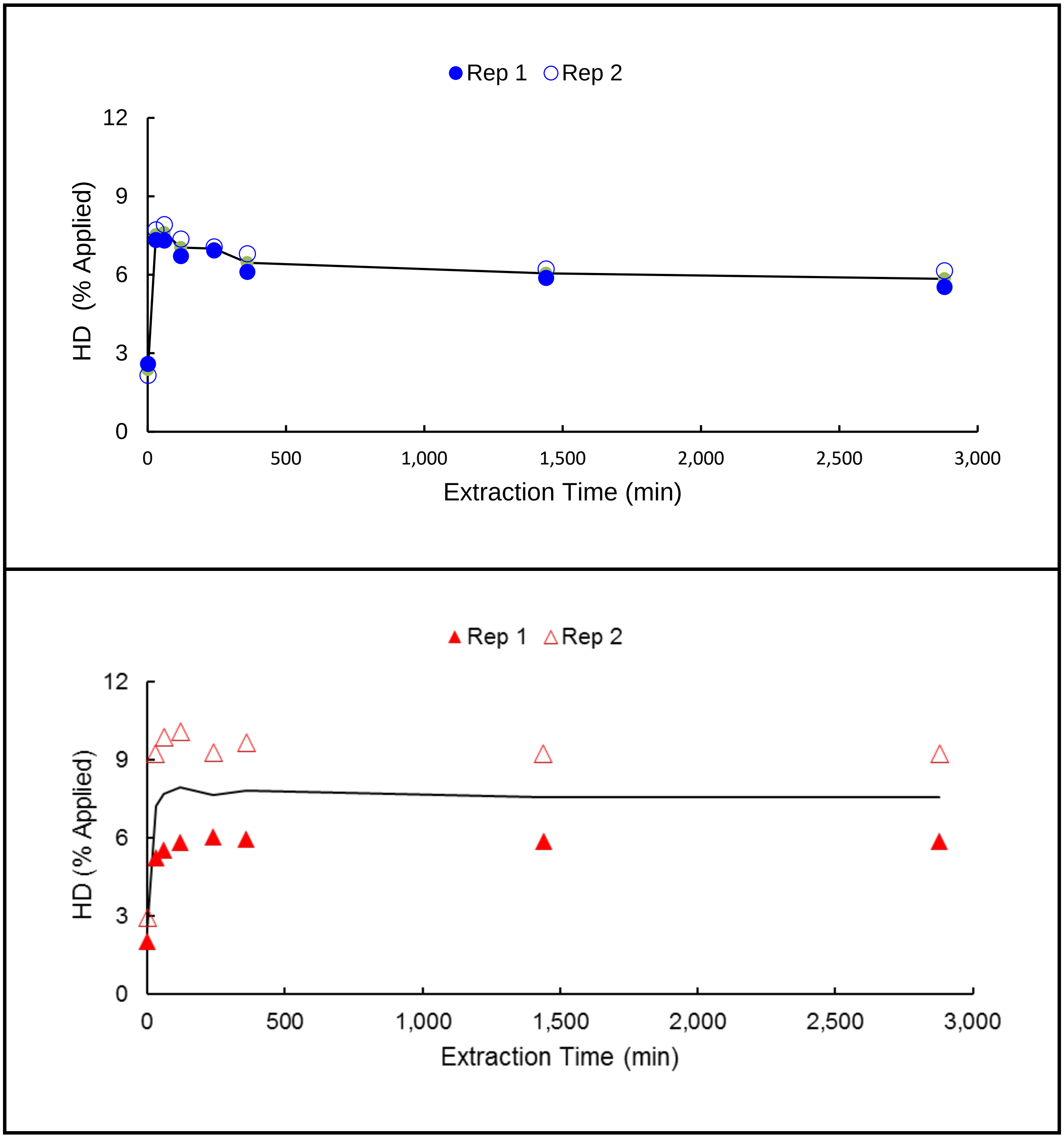


Figure 18. DCE of HD from military patches. Chloroform was the extraction solvent (top panel) and *n*-hexane was the extraction solvent (bottom panel).

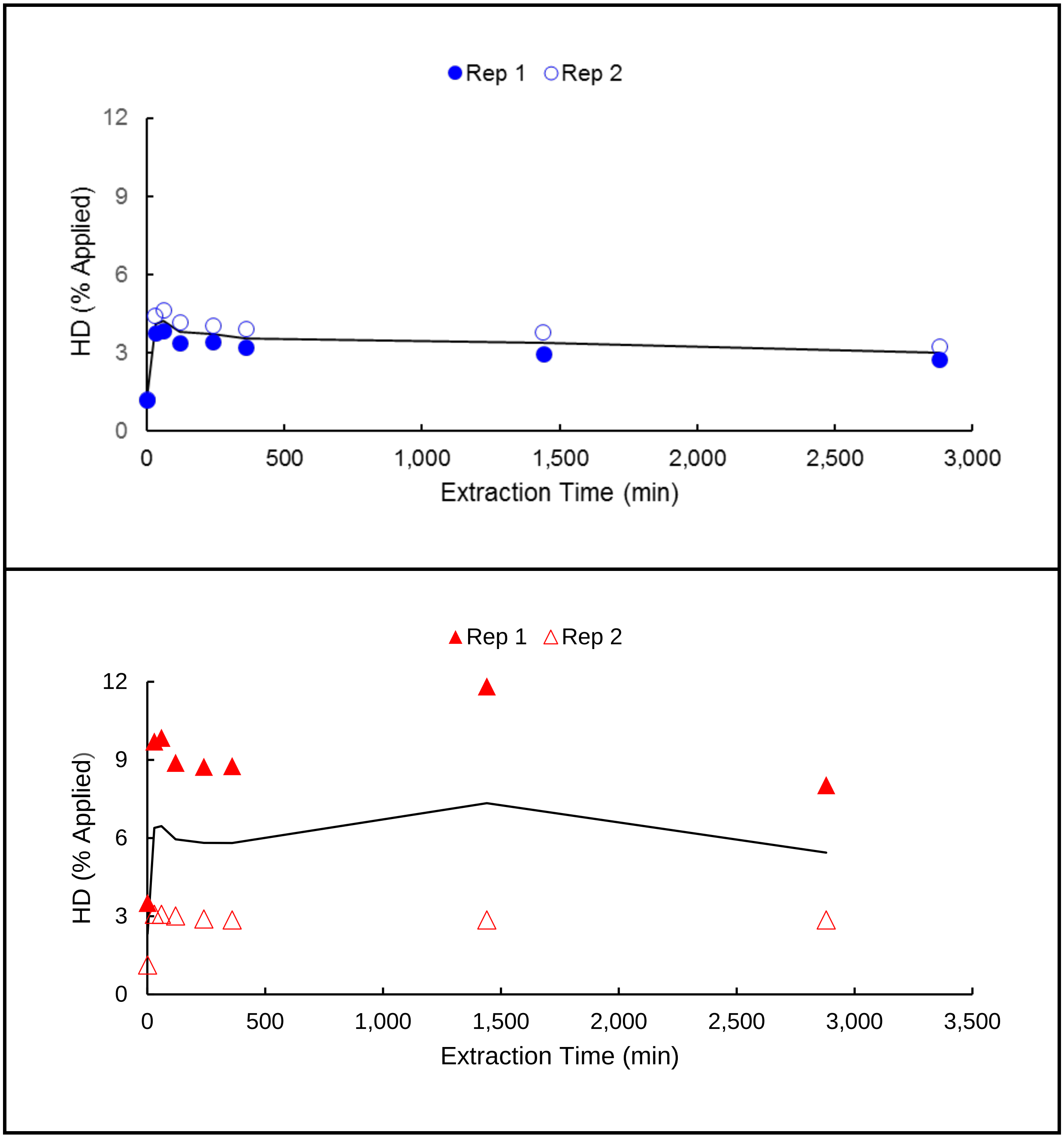


Figure 19. DCE of HD from nylon webbing. Chloroform was the extraction solvent (top panel) and *n*-hexane was the extraction solvent (bottom panel).

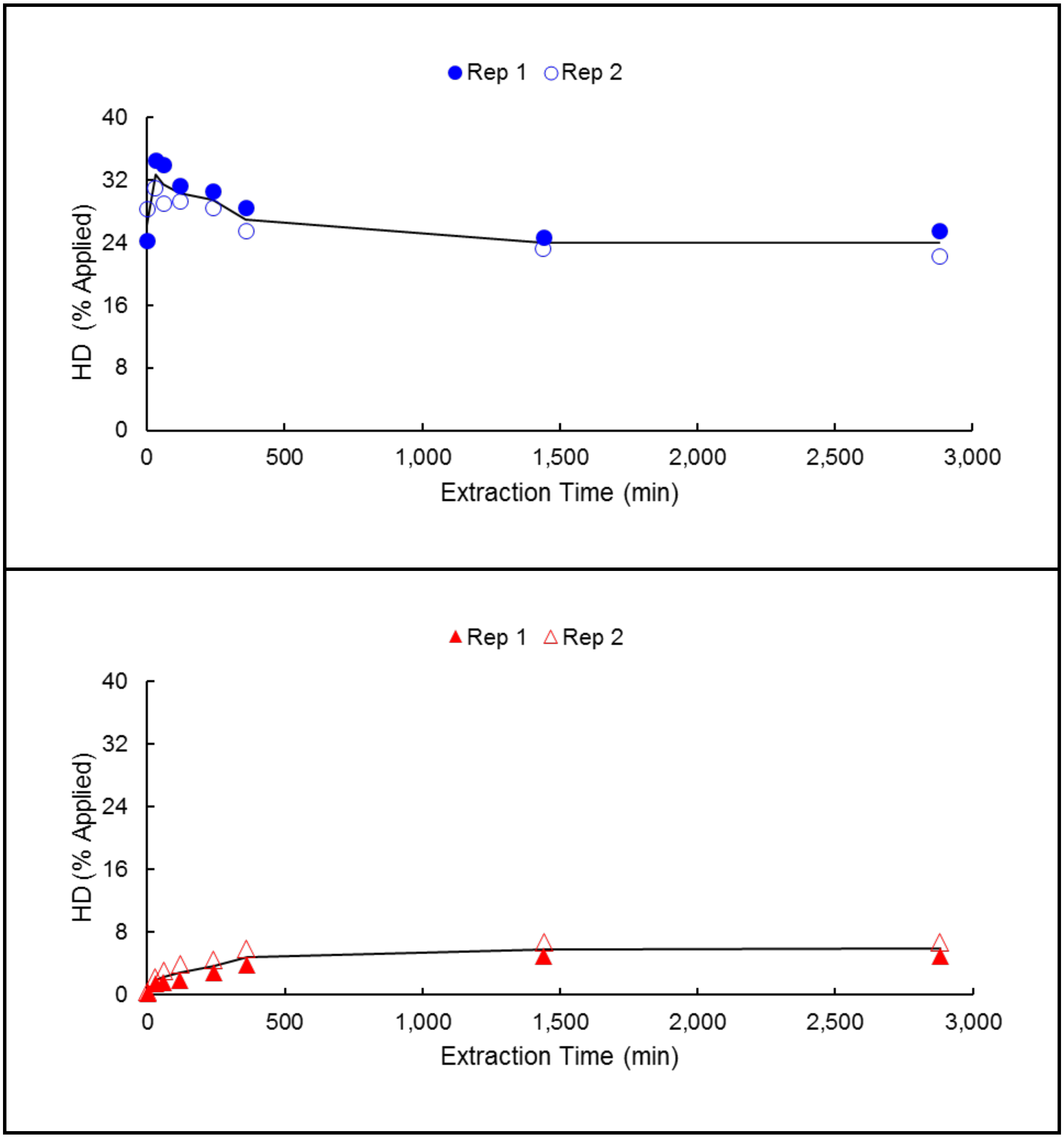


Figure 20. DCE of HD from ID cards. Chloroform was the extraction solvent (top panel) and *n*-hexane was the extraction solvent (bottom panel).

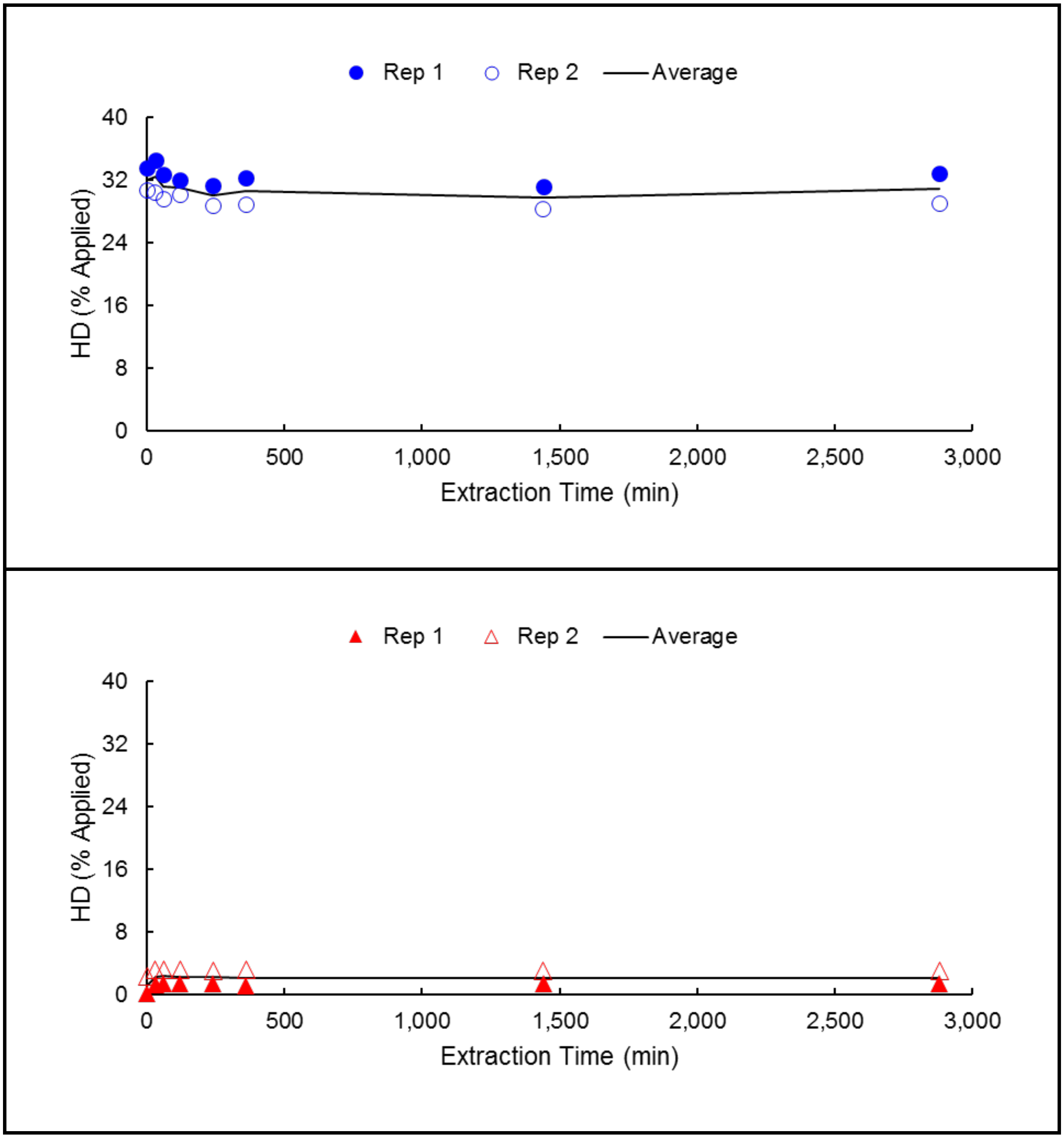


Figure 21. DCE of HD from pocket knives. Chloroform was the extraction solvent (top panel) and *n*-hexane was the extraction solvent (bottom panel).

4. CONCLUSIONS AND RECOMMENDATIONS

Decontamination of HD-contaminated PEs is conducted to ensure that loved ones receive non-CWA-contaminated personal items as remembrances and keepsakes of their fallen warfighters. For certain substrates, the bleach and brushing treatment is sufficient to remove approximately 90% of HD. However, the remaining 10% of the HD could pose a significant health risk for any unprotected persons who may come into contact with a contaminated item. For other substrates, such as pocket knives and ID cards, the bleach treatment was not effective in removing HD after an extended aging time because of the presence of complex features (such as grooves, etc. in a pocket knife) and strong interactions between the contaminant and the substrate (ID card).

Humidified CHAD treatment provides a significant reduction of extracted HD in PEs made of fabric and metal, regardless of the presence of any complex features. The amount of HD extracted from military patches, nylon webbing, and pocket knives was reduced to below the MLOD level after 144 h of humidified CHAD treatment. In the case of coinage, the amount of HD extracted was reduced to below the MLOD level after 24 h of humidified CHAD treatment. Humidified CHAD treatment was not successful in decontaminating ID cards; the average amount of HD extracted after treatment was 86,700 ng.

In most substrates, humidified CHAD treatment augments the bleach and brushing decontamination procedure for contaminated PEs and does not negatively affect the integrity of the material.

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ACRONYMS AND ABBREVIATIONS

ACS	American Chemical Society
CASARM	Chemical Agent Standard Analytical Reference Material
CCV	continuing calibration verification
CHAD	chemical hot air decontamination
CIS	cooled injection system
CRC	cumulative release curve
CWA	chemical warfare agent
DCE	dynamic chemical extraction
DCS	dose control sample
DI	deionized
ECBC	U.S. Army Edgewood Chemical Biological Center
GC–MS	gas chromatography–mass spectrometry
HD	*bis*-(2-chloroethyl)sulfide
HPLC	high-performance liquid chromatography
ID	identification
ILOD	instrumental limit of detection
ILOQ	instrumental limit of quantitation
IS	internal standard
LC–MS	liquid chromatography–mass spectrometry
MLOD	method limit of detection
MLOQ	method limit of quantitation
MRM	multiple reaction monitoring
PE	personal effect
RH	relative humidity
RSD	relative standard deviation
SD	standard deviation
SD2ED	*Chemical Contaminant and Decontaminant Test Methodologies Source Document–Second Edition*
SIV	small item vapor

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APPENDIX
TABULATED TEST DATA

Table A-1. Data from Test CHDP01, Nickels[a]

Sample Description	Treatment	Filename	Replicate Number	Recovered HD (ng)
Tool sample	None	021_CHDP01_1toolHD-01_Tool	1	2,170,000
		022_CHDP01_1toolHD-02_Tool	2	2,450,000
		023_CHDP01_1toolHD-03_Tool	3	2,390,000
		024_CHDP01_1toolHD-04_Tool	4	2,410,000
		025_CHDP01_1toolHD-05_Tool	5	2,430,000
Aging control	Aging only	029_CHDP01_004SS1_RES	1	1,090,000
		034_CHDP01_005SS1_RES	2	994,000
		035_CHDP01_006SS1_RES	3	1,180,000
Bleach control	Aging and bleach	049_CHDP01_010SS1_RES_RR	1	61,400
		050_CHDP01_011SS1_RES_RR	2	171,000
		051_CHDP01_012SS1_RES_RR	3	18,600
Sample	Aging, bleach, and 24 h CHAD	049_CHDP01_016SS1_RES	1	Non-detect[a]
		050_CHDP01_017SS1_RES	2	Non-detect[a]
		051_CHDP01_018SS1_RES	3	Non-detect[a]
Sample	Aging, bleach, and 48 h CHAD	024_CHDP01_022SS1_RES	1	Non-detect[a]
		025_CHDP01_023SS1_RES	2	Non-detect[a]
		026_CHDP01_024SS1_RES	3	Non-detect[a]
Sample	Aging, bleach, and 72 h CHAD	034_CHDP01_028SS1_RES	1	22.7
		035_CHDP01_029SS1_RES	2	Non-detect[b]
		036_CHDP01_030SS1_RES	3	Non-detect[b]

[a] Overall method limit of detection (MLOD) = 2.4 ng, and method limit of quantitation (MLOQ) = 10.2 ng.
[b] Value was >MLOD (2.4 ng).

CHAD: chemical hot air decontamination.

Table A-2. Data from Test CHDP01, Pennies

Sample Description	Treatment	Filename	Replicate Number	Recovered HD (ng)
Tool sample	None	021_CHDP01_1toolHD-01_Tool	1	2,170,000
		022_CHDP01_1toolHD-02_Tool	2	2,450,000
		023_CHDP01_1toolHD-03_Tool	3	2,390,000
		024_CHDP01_1toolHD-04_Tool	4	2,410,000
		025_CHDP01_1toolHD-05_Tool	5	2,430,000
Aging control	Aging only	045_CHDP01_001SS1_RES_RR	1	602,000
		027_CHDP01_002SS1_RES	2	1,130,000
		028_CHDP01_003SS1_RES	3	1,030,000
Bleach control	Aging and bleach	046_CHDP01_007SS1_RES_RR	1	40,600
		047_CHDP01_008SS1_RES_RR	2	70,300
		048_CHDP01_009SS1_RES_RR	3	105,000
Sample	Aging, bleach, and 24 h CHAD	042_CHDP01_013SS1_RES	1	Trace[a]
		047_CHDP01_014SS1_RES	2	Trace[a]
		048_CHDP01_015SS1_RES	3	Trace[a]
Sample	Aging, bleach, and 48 h CHAD	021_CHDP01_019SS1_RES	1	Non-detect[b]
		022_CHDP01_020SS1_RES	2	Non-detect[b]
		060_CHDP01_021SS1_RES_RR	3	Non-detect[b]
Sample	Aging, bleach, and 72 h CHAD	027_CHDP01_025SS1_RES	1	Non-detect[b]
		028_CHDP01_026SS1_RES	2	Non-detect[b]
		029_CHDP01_027SS1_RES	3	Non-detect[b]

[a] Value was >MLOD (2.4 ng) but <MLOQ (10.2 ng). [b] Value was <MLOD (2.4 ng).

Table A-3. Data from Test CHDP02, Military Patches

Sample Description	Treatment	Filename	Replicate Number	Recovered HD (ng)
Tool sample	None	021_CHDP02_1toolHD-01_Tool	1	2,700,000
		051_CHDP02_1toolHD-02_Tool_RR	2	2,620,000
		023_CHDP02_1toolHD-03_Tool	3	2,570,000
		024_CHDP02_1toolHD-04_Tool	4	2,670,000
		025_CHDP02_1toolHD-05_Tool	5	2,730,000
Aging control	Aging only	029_CHDP02_004MP1_RES	1	453,000
		052_CHDP02_005MP1_RES_RR	2	644,000
		053_CHDP02_006MP1_RES_RR	3	764,000
Bleach control	Aging and bleach	057_CHDP02_010MP1_RES_RR	1	238,000
		058_CHDP02_011MP1_RES_RR	2	71,900
		059_CHDP02_012MP1_RES_RR	3	255,000
Sample	Aging, bleach, and 24 h CHAD	049_CHDP02_016MP1_RES	1	16.1
		065_CHDP02_017MP1_RES_RR	2	45.0
		051_CHDP02_018MP1_RES	3	33.9
Sample	Aging, bleach, and 48 h CHAD	024_CHDP02_022MP1_RES	1	Trace[a]
		025_CHDP02_023MP1_RES_RR	2	Trace[a]
		026_CHDP02_024MP1_RES_RR	3	Trace[a]

(Continued)

Table A-3 Continued

Sample	Aging, bleach, and 72 h CHAD	034_CHDP02_028MP1_RES	1	Trace[a]
		076_CHDP02_029MP1_RES_RR	2	Trace[a]
		036_CHDP02_030MP1_RES_RR	3	Trace[a]
Sample	Aging, bleach, and 144 h CHAD	040_CHDP02_034MP1_RES	1	Non-detect[b]
		041_CHDP02_035MP1_RES	2	Non-detect[b]
		087_CHDP02_036MP1_RES_RR	3	Trace[a]
[a] Value was >MLOD (2.4 ng) but <MLOQ (10.2 ng). [b] Value was <MLOD (2.4 ng).				

Table A-4. Data from Test CHDP02, Nylon Webbing

Sample Description	Treatment	Filename	Replicate Number	Recovered HD (ng)
Tool sample	None	021_CHDP02_1toolHD-01_Tool	1	2,700,000
		051_CHDP02_1toolHD-02_Tool_RR	2	2,620,000
		023_CHDP02_1toolHD-03_Tool	3	2,570,000
		024_CHDP02_1toolHD-04_Tool	4	2,670,000
		025_CHDP02_1toolHD-05_Tool	5	2,730,000
Aging control	Aging only	026_CHDP02_001NW1_RES	1	210,000
		027_CHDP02_002NW1_RES	2	190,000
		028_CHDP02_003NW1_RES	3	161,000
Bleach control	Aging and bleach	054_CHDP02_007NW1_RES_RR	1	866
		055_CHDP02_008NW1_RES_RR	2	411
		056_CHDP02_009NW1_RES_RR	3	809
Sample	Aging, bleach, and 24 h CHAD	064_CHDP02_013NW1_RES_RR	1	160
		047_CHDP02_014NW1_RES	2	11.0
		048_CHDP02_015NW1_RES	3	Non-detect[a]
Sample	Aging, bleach, and 48 h CHAD	074_CHDP02_019NW1_RES_RR	1	Non-detect[a]
		086_CHDP02_020NW1_RES_RR	2	30.0
		023_CHDP02_021NW1_RES	3	Non-detect[a]
Sample	Aging, bleach, and 72 h CHAD	027_CHDP02_025NW1_RES	1	Non-detect[a]
		028_CHDP02_026NW1_RES	2	10.5
		029_CHDP02_027NW1_RES	3	Non-detect[a]
Sample	Aging, bleach, and 144 h CHAD	037_CHDP02_031NW1_RES	1	Trace[b]
		038_CHDP02_032NW1_RES	2	Non-detect[a]
		039_CHDP02_033NW1_RES	3	Non-detect[a]

[a] Value was <MLOD (2.4 ng). [b] Value was >MLOD (2.4 ng) but <MLOQ (10.2 g).

Table A-5. Data from Test CHDP03, ID Cards

Sample Description	Treatment	Filename	Replicate Number	Recovered HD (ng)
Tool sample	None	021_CHDP03_1toolHD-01_Tool	1	2,250,000
		022_CHDP03_1toolHD-02_Tool	2	2,260,000
		023_CHDP03_1toolHD-03_Tool	3	2,250,000
		024_CHDP03_1toolHD-04_Tool	4	2,330,000
		025_CHDP03_1toolHD-05_Tool	5	2,210,000
Aging control	Aging only	026_CHDP03_001SI1_RES	1	920,000
		027_CHDP03_002SI1_RES	2	960,000
		028_CHDP03_003SI1_RES	3	847,000
Bleach control	Aging and bleach	029_CHDP03_004SI1_RES	1	811,000
		034_CHDP03_005SI1_RES	2	832,000
		035_CHDP03_006SI1_RES	3	604,000
Sample	Aging, bleach, and 24 h CHAD	036_CHDP03_007SI1_RES	1	152,000
		037_CHDP03_008SI1_RES	2	115,000
		038_CHDP03_009SI1_RES	3	170,000
Sample	Aging, bleach, and 48 h CHAD	039_CHDP03_010SI1_RES	1	130,000
		040_CHDP03_011SI1_RES	2	118,000
		041_CHDP03_012SI1_RES	3	88,600
Sample	Aging, bleach, and 72 h CHAD	042_CHDP03_013SI1_RES	1	112,000
		047_CHDP03_014SI1_RES	2	88,200
		048_CHDP03_015SI1_RES	3	109,000
Sample	Aging, bleach, and 144 h CHAD	049_CHDP03_016SI1_RES	1	77,700
		050_CHDP03_017SI1_RES	2	95,600
		NA	3	NA

NA: not available. Sample spilled during extraction procedure.

Table A-6. Data from Test CHDP04, Pocket Knives

Sample Description	**Treatment**	**Filename**	**Replicate Number**	**Recovered HD (ng)**
Tool sample	None	021_CHDP04_1toolHD-01_Tool	1	2,380,000
		022_CHDP04_1toolHD-02_Tool	2	2,330,000
		023_CHDP04_1toolHD-03_Tool	3	2,420,000
		024_CHDP04_1toolHD-04_Tool	4	2,320,000
		025_CHDP04_1toolHD-05_Tool	5	2,380,000
Aging control	Aging only	060_CHDP04_001SS1_RES_RR	1	34,800
		061_CHDP04_002SS1_RES_RR	2	1,120,000
		062_CHDP04_003SS1_RES_RR	3	481,000
Bleach control	Aging and bleach	063_CHDP04_004SS1_RES_RR	1	65,200
		064_CHDP04_005SS1_RES_RR	2	357,000
		065_CHDP04_006SS1_RES_RR	3	19,800
Sample	Aging, bleach, and 24 h CHAD	066_CHDP04_007SS1_RES_RR	1	Non-detect[a]
		067_CHDP04_008SS1_RES_RR	2	Non-detect[a]
		068_CHDP04_009SS1_RES_RR	3	Non-detect[a]
Sample	Aging, bleach, and 48 h CHAD	039_CHDP04_010SS1_RES	1	Non-detect[a]
		040_CHDP04_011SS1_RES	2	Trace[b]
		041_CHDP04_012SS1_RES	3	Trace[b]
Sample	Aging, bleach, and 72 h CHAD	073_CHDP04_013SS1_RES_RR	1	Non-detect[a]
		047_CHDP04_014SS1_RES	2	Trace[b]
		048_CHDP04_015SS1_RES	3	Non-detect[a]
Sample	Aging, bleach, and 144 h CHAD	049_CHDP04_016SS1_RES	1	Non-detect[a]
		050_CHDP04_017SS1_RES	2	Non-detect[a]
		051_CHDP04_018SS1_RES	3	Non-detect[a]

[a] Value was <MLOD (9.5 ng). [b] Value was >MLOD (9.5 ng) but <MLOQ (40.8 ng).

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