U.S. ARMY COMBAT CAPABILITIES DEVELOPMENT COMMAND
CHEMICAL BIOLOGICAL CENTER
ABERDEEN PROVING GROUND, MD 21010-5424

DEVCOM CBC-TR-1801

Surface Decontamination of Polyurethane and Stainless Steel Following Carfentanil Contamination

Amanda M. Schenning
Michelle L. Sheahy
RESEARCH AND OPERATIONS DIRECTORATE

Jill L. Ruth
LEIDOS, INC.
Abingdon, MD 21009-1261

Michael J. Chesebrough
DCS CORPORATION
Abingdon, MD 21009-1283

April 2023

Distribution Statement A. Approved for public release: distribution unlimited.

Disclaimer

The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorizing documents.

REPORT DOCUMENTATION PAGE

1. REPORT DATE	2. REPORT TYPE	3. DATES COVERED	
		START DATE	END DATE
XX-04-2023	Final	Jun 2018	Dec 2018

4. TITLE AND SUBTITLE
Surface Decontamination of Polyurethane and Stainless Steel Following Carfentanil Contamination

5a. CONTRACT NUMBER	5b. GRANT NUMBER	5c. PROGRAM ELEMENT NUMBER
5d. PROJECT NUMBER	**5e. TASK NUMBER**	**5f. WORK UNIT NUMBER**

6. AUTHOR(S)
Schenning, Amanda M.; Sheahy, Michelle L. (DEVCOM CBC); Ruth, Jill L. (Leidos); Chesebrough, Michael J. (DCS)

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Director, DEVCOM CBC, ATTN: FCDD-CBD, APG, MD 21010-5424
Leidos, Inc.; 3465 Box Hill Corporate Center, Abingdon, MD 21009-1261
DCS Corp.; 100 Walter Ward Boulevard, Suite 100, Abingdon, MD 21009-1284

8. PERFORMING ORGANIZATION REPORT NUMBER
DEVCOM CBC-TR-1801

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)	10. SPONSOR/MONITOR'S ACRONYM(S)	11. SPONSOR/MONITOR'S REPORT NUMBER(S)
Product Director for Cross-Commodity Advanced Threats and Test Infrastructure; 8198 Blackhawk Road, Aberdeen Proving Ground, MD 21010-5424	PDCATTI	

12. DISTRIBUTION/AVAILABILITY STATEMENT
Distribution Statement A. Approved for public release: distribution unlimited.

13. SUPPLEMENTARY NOTES
U.S. Army Combat Capabilities Development Command Chemical Biological Center (DEVCOM CBC) was previously known as U.S. Army Edgewood Chemical Biological Center (ECBC).

14. ABSTRACT (LESS THAN 200 WORDS)
The effort presented here was a scoping study intended to elucidate knowledge gaps and inform decisions regarding the need for further research efforts into opioid decontamination. We used a limited panel study with carfentanil (citrate and free base forms), three decontaminant formulations (soapy water, Dahlgren Decon solution [First Line Technology; Chantilly, VA], and sprayable decontaminant slurry), and two material surfaces (stainless steel and polyurethane). The results showed that all three decontaminants have some potential for decontamination; however, contaminant form played a key role in efficacy and physical removal. The results highlight that the physical state of the contaminant (e.g., neat versus in solution) is a crucial factor when assessing the potential performance of a decontamination process, whether in the laboratory or in the field. Furthermore, this work revealed the need for enhancements in laboratory infrastructure and test methodology to support the study of nonliquid contaminant forms.

15. SUBJECT TERMS

Opioid	Carfentanil	Fentanyl	Decontamination
Free base	Pharmaceutical-based agent (PBA)	Dahlgren Decon solution (DD)	Slurry

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES
a. REPORT	b. ABSTRACT	C. THIS PAGE		
U	U	U	UU	28

19a. NAME OF RESPONSIBLE PERSON	19b. PHONE NUMBER *(Include area code)*
Renu B. Rastogi	(410) 436-7545

STANDARD FORM 298 (REV. 5/2020)
Prescribed by ANSI Std. Z39.18

Blank

PREFACE

The work described in this report was started in June 2018 and completed in December 2018. At the time this work was performed, the U.S. Army Combat Capabilities Development Command Chemical Biological Center (DEVCOM CBC; Aberdeen Proving Ground, MD) was known as the U.S. Army Edgewood Chemical Biological Center (ECBC).

The use of either trade or manufacturers' names in this report does not constitute an official endorsement of any commercial products. This report may not be cited for purposes of advertisement.

This report has been approved for public release.

Acknowledgments

The authors extend appreciation to the following individuals for their hard work and assistance with the execution of this technical program:

- Jeffrey Hofmann, Stephen Carrig, and Mary Beth Busch (Product Director for Cross-Commodity Advanced Threats and Test Infrastructure; Aberdeen Proving Ground, MD) for their financial support and encouragement of this project;
- Drs. Andrew Walz and Fulian Hsu (DEVCOM CBC) for synthesizing the contaminants used in this study;
- Stefanie Smallwood and Joe Myers (DEVCOM CBC) for helpful discussions and guidance regarding panel studies and sprayable decontaminant slurry;
- Kevin Morrissey (DEVCOM CBC) for helpful discussions and encouragement during this project; and
- Dr. Thomas Pearl (DEVCOM CBC) for insights, useful comments, and editing during the production of this report.

Blank

CONTENTS

FIGURES

TABLES

SURFACE DECONTAMINATION OF POLYURETHANE AND STAINLESS STEEL FOLLOWING CARFENTANIL CONTAMINATION

1. INTRODUCTION

Similar to traditional chemical warfare agents (CWAs), pharmaceutical-based agents (PBAs) can be used to cause great harm, incapacitation, and death to warfighters and the general public. The use of synthetic opioids in the 2002 Moscow theatre crisis made the world aware of this new threat.[1] Furthermore, the illicit drug trade has reinforced the threat by making many PBAs, such as opioids, readily available to those who either knowingly or unknowingly seek them. With lethality on the same level as the nerve agent *O*-ethyl-*S*-(2-diisopropylaminoethyl) methyl phosphonothiolate (VX), opioids present a significant potential hazard to personnel who come in contact with them. In light of the current opioid epidemic and the increasing potential for a military response involving a clandestine drug operation, the need to effectively decontaminate military assets following an operational scenario involving PBAs has recently come under the umbrella of credible threat scenarios.

The process and efficacy of decontamination efforts are complicated by the many available forms of opioid analogs, including the free base, hydrochloride, and citrate and oxalate salts. Few data exist regarding the effects of the PBA form (free base versus salt) on the decontamination burden for PBA-contaminated materials or the contact transfer potential for personnel interacting with contaminated surfaces. It is, therefore, difficult for researchers to make informed decisions to guide the efforts of warfighters and first responders.

In late spring of 2018, the Product Director for Cross-Commodity Advanced Threats and Test Infrastructure (PDCATTI; Aberdeen Proving Ground, MD) requested that the U.S. Army Combat Capabilities Development Command Chemical Biological Center (DEVCOM CBC, formerly U.S. Army Edgewood Chemical Biological Center; Aberdeen Proving Ground, MD) study surface decontamination following contamination events involving the opioid class of PBAs. The questions to be addressed included the following: Does rinsing with soapy water remove all of the contaminant from a surface? Is Dahlgren Decon solution (DD; First Line Technology; Chantilly, VA) an effective decontaminant against fentanyl and carfentanil? Does the form of the contaminant (such as the salt, a solution thereof, or the free base) affect decontaminant efficacy?

This effort was a scoping study to elucidate knowledge gaps and inform decisions regarding the need for further research into opioid contamination. To limit the scope of this study, some assumptions were made that are not necessarily representative of real-world scenarios. One of these assumptions was that a single opioid could be used to represent the behavior of an entire class of PBAs. The fentanyl class of opioids contains many analogs, each with its own set of physical and chemical properties, and each available in multiple salt forms. Carfentanil was chosen for study as a worst-case scenario due to its extreme toxicity; however, other fentanyl analogs are more often associated with illicit drug overdoses.[2] The scope of this study was limited, so conclusions were drawn using only three decontaminants and two surfaces. Perhaps even more problematic was the substitution of solvated contaminant for the neat

material, particularly when the neat contaminant existed in a solid or semisolid form such as carfentanil citrate salt and carfentanil free base. Several studies have demonstrated that the method of solids deposition has a significant impact on workers' ability to detect and remove contaminants from surfaces.[3,4] However, in light of these generalizations, this study did reveal some common themes and trends regarding contaminant form, decontaminant properties, and material type.

The experiments included the evaluation of three decontaminants in the removal of a single representative opioid, carfentanil, from two military-relevant surfaces. The decontaminant formulations were (1) a solution of soapy water, which is a nonreactive, currently fielded approach for physical removal; (2) DD, which is a commercially available product; and (3) sprayable decontaminant slurry (SDS), which is a novel formulation currently under development at DEVCOM CBC. The two materials, stainless steel (SS) and a polyurethane (PU) paint coating, were representative of impermeable (i.e., contaminant remains on material surface) and permeable (i.e., contaminant is capable of absorbing into the material) surfaces, respectively. The contaminant, carfentanil, is an ultrapotent opioid that may be encountered in a variety of forms, including the free base, various salt forms, and solutions thereof. This study was performed in accordance with the *Chemical Contaminant and Decontaminant Test Methodology Source Document, Second Edition* (SD2ED).[5] The remaining agent (RA)[5] mass was the metric by which decontaminant efficacy was evaluated. RA is defined as the mass of contaminant that remains on or in a material after a decontamination process has been performed.

Consistent with previous tests,[6,7] the primary challenge in conducting this scoping study was delivering a uniform amount of contaminant on the surface of the materials. In a typical panel test with liquid chemical agents, positive-displacement pipets are used to deposit accurate and precise amounts of contaminant on the surface of test coupons. The average mass of agent applied to the coupons is verified by dose-control samples (DCS); see SD2ED for further details.[5] In this study, however, the neat contaminant was not in liquid form. The citrate salt is a powder, whereas the free base is a viscous, sticky semisolid. The potential for aerosolization of the citrate salt and the sticky nature of the free base precluded the delivery of a consistent amount of contaminant across samples. To avoid making gravimetric measurements of milligram quantities of powder inside a glovebox (which would greatly reduce sample throughput), the carfentanil citrate was dissolved in methanol before it was applied to the panels. The free base, which was too viscous to be delivered volumetrically, was applied two ways: it was either dissolved in methanol like the citrate, or a bead of the neat contaminant was dabbed onto the coupon surface. As for the liquid samples, the starting challenge for the methanol-delivered samples was represented by the average DCS, whereas the starting challenge for the neat free base was determined gravimetrically.

The study results indicated that all three decontaminants have some potential for reducing the post-decontamination contact hazard associated with this opioid; however, contaminant form played a key role in decontaminant efficacy and physical removal. The delivery of the contaminant (as a free base versus a salt or neat versus in solution) must be considered when assessing the potential performance of a decontamination process. This work has also revealed the need for enhancements in laboratory infrastructure and test methodology to support the study of nonliquid contaminant forms.

2. PANEL TREATMENT PROCESSES AND METHODS

2.1 Panel Test Overview

The panel treatment process used in this study was adapted from the SD2ED.[5] The process (detailed below) included contamination, aging, decontamination, post-decontamination rinse, quench, and panel extraction. The concentration of carfentanil in the panel extract, which determines the RA, was determined using ultra-high-performance liquid chromatography/mass spectrometry (UHPLC/MS). The test parameters are listed in Table 1, and the panel treatment process is illustrated in Figure 1.

Table 1. Test Parameters for Panel Decontamination Scoping Study

Variable	Value
Contaminants	Carfentanil citrate solution (20 mg/mL), carfentanil free base solution (20 mg/mL), carfentanil free base (neat)
Contamination	Solutions: 100 µL droplet; neat free base: ~2 mg targeted mass
Materials	PU paint, SS
Age time	60 min
Decontaminants	1% soapy water, DD, SDS
Decontamination residence time	15 min
Quench chemistry by decontaminant	Soapy water: no quench; DD: sodium thiosulfate; SDS: glacial acetic acid followed by tetrahydrothiophene
Extraction process	20 mL methanol, 60 min
Replicates	Three (one test session using SDS was unintentionally designed with five replicates, so all data were included)
Measurement	RA

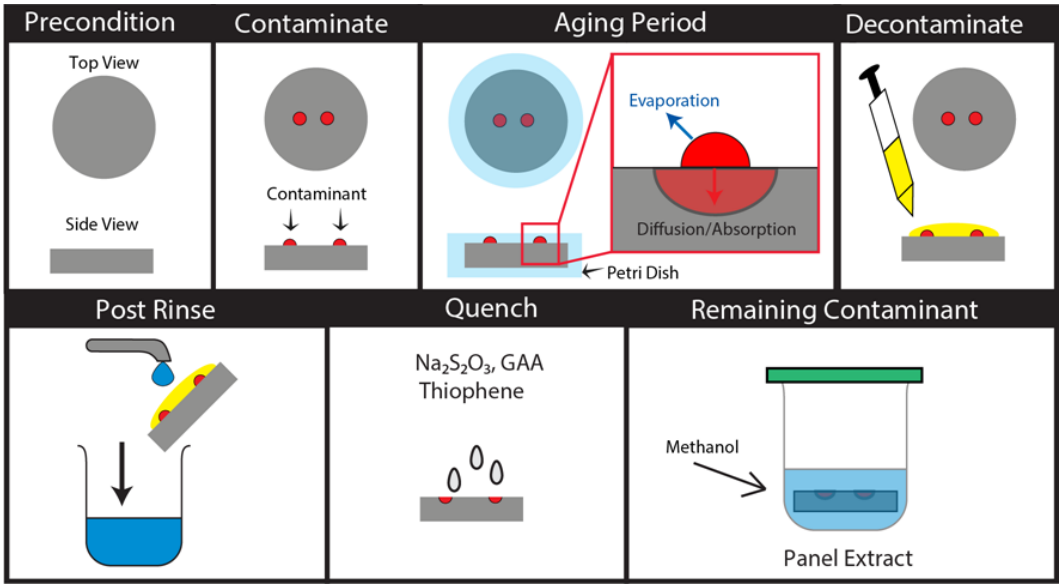

Figure 1. Panel treatment process for determination of RA following decontamination. GAA, glacial acetic acid.

2.2 Contaminants

The opioid contaminants used in this study, carfentanil citrate and carfentanil free base, were synthesized by the DEVCOM CBC Agent Chemistry Branch and assigned lot numbers of HF-13A-067 and HF-18063B, respectively. Purity information is on file with the Agent Chemistry Branch. The contaminants were used only in properly certified surety facilities capable of handling such chemicals safely. The personnel handling the chemical agents for this study were fully trained and certified for such operations.

Carfentanil citrate salt exists as a solid at room temperature and was handled in neat form only under the engineering controls of a fully validated glovebox. The potential for aerosolization of the solid salt and the sticky nature of the free base made it very difficult to dose an accurate known amount of contaminant. Because of the acute hazard present when working with the contaminants in powder form, the citrate was dissolved in methanol prior to dosing on panels. The free base was used neat as well as dissolved in methanol.

2.3 Contamination of Materials

Standard 2 in. diameter circular panels of SS and a PU paint coating were used. All panels began as 1/8 in. thick SS; some were left uncoated, and others received a PU paint coating (total PU paint thickness, ~100 µm). A contamination density of ~1 g/m^2 was targeted, corresponding to a 2 mg dose of contaminant to the panel. The panels were contaminated with solvated carfentanil by dosing 100 µL of a 20 mg/mL solution (after correction for the salt adduct). The methanol solution containing the 2 mg dose spread across most of the surfaces of both panel types without running off (Figure 2).

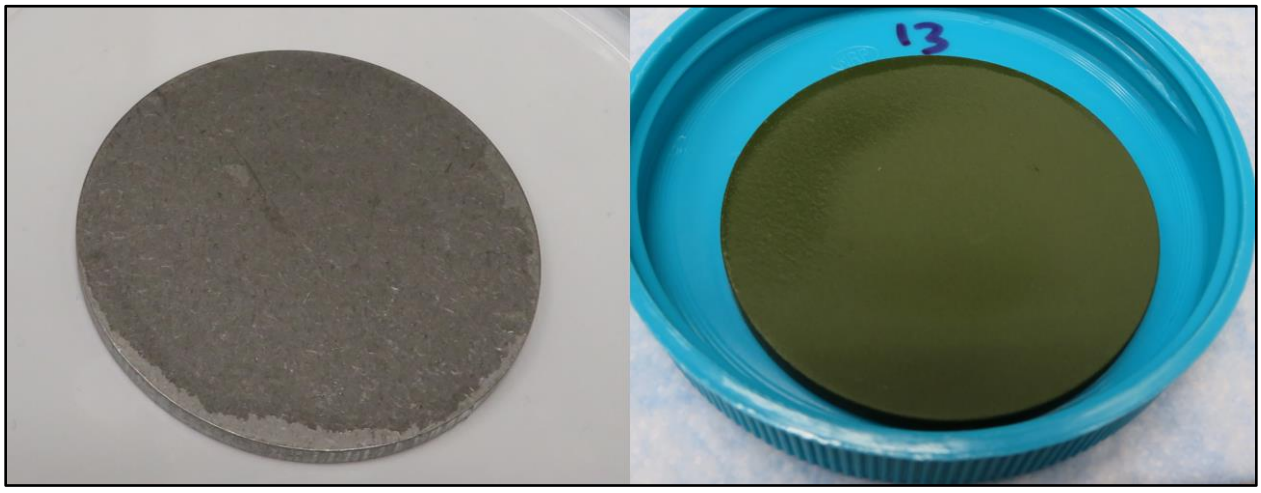

Figure 2. SS (left) and PU paint-coated (right) panels immediately following contamination with carfentanil free base in methanol solution.

As previously mentioned, it was not possible to apply a reproducible dose of contaminant to the panels because of the remarkably viscous nature of the free base. It had a glue-like consistency and was difficult to stir or pipet at ambient temperature; therefore, alternative methods of contaminant delivery were considered. To aid in handling, the neat agent was gently heated on an 80 °C warming stage with stirring. Although this treatment was sufficient to allow the free base to be drawn into a positive-displacement pipet, when it was dispensed, the free base formed a bead at the end of the plastic pipet tip and clung to the sides without dropping off.

Furthermore, the neat material appeared to thicken with repeated heating cycles, and it began to form string-like projections from the pipet tip as soon as it was removed from the warm vial and exposed to the cooler surrounding air. When the free base was originally prepared, the DEVCOM CBC synthesis chemists advised that it probably contained approximately 1% of residual chloroform from the synthesis process. We hypothesized that this residual solvent was being driven off during the heating process and that the free base was actually becoming purer with each subsequent use. Because the pipet tip had to be gently scraped across the surface of the panels to transfer the free base, the amount of contaminant transferred to each panel and the pattern of contamination were unique for each sample and DCS. As a result, the contamination mass for each sample was measured gravimetrically, and the starting challenge of the contaminated samples varied greatly. Although the targeted mass for contamination was 2 mg, the gravimetrically measured amounts of free base delivered to the panels ranged from 1.6 to 6.7 mg. The mass of free base delivered to each panel is listed in Table 2.

Table 2. Starting Mass of Neat Carfentanil Free Base Delivered to Panels

Decontaminant	**Starting Mass (mg)**					
	PU Paint			**SS**		
	Rep 1	**Rep 2**	**Rep 3**	**Rep 1**	**Rep 2**	**Rep 3**
Soapy water	2.8	4.4	5.0	1.6	3.1	3.0
SDS	4.0	4.2	4.0	3.9	3.2	3.0
DD	4.2	3.8	6.7	5.2	2.9	3.9

Rep, replicate.

Figure 3 shows the unique contamination patterns and the variability in starting mass due to the viscous nature of the free base.

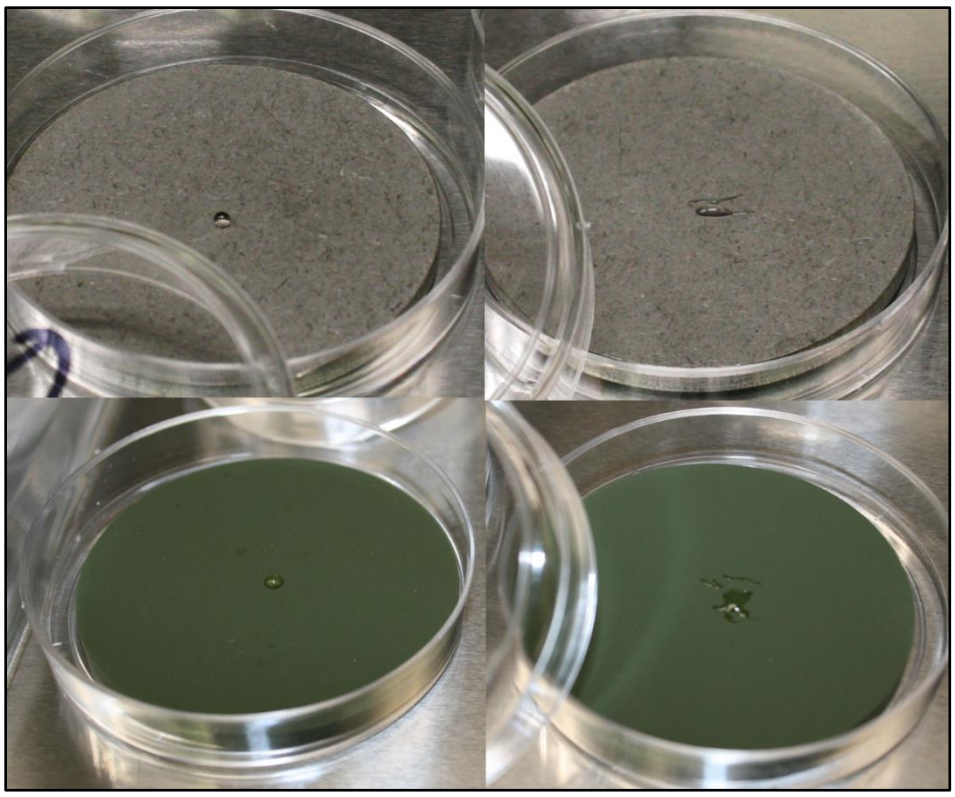

Figure 3. Examples of four different test coupons following contamination with carfentanil free base.

Following contamination, all panels were aged for 60 min at room temperature in the chemical fume hood. The extended age time was chosen to allow for methanol to evaporate from the coupons dosed with solvated carfentanil.

2.4 Decontamination of Materials

The decontaminants used in this study included a currently fielded formulation (soapy water), a commercial off-the-shelf product (DD), and a novel formulation under development (SDS); see Figure 4.

Soapy water is a nonreactive, aqueous-based surfactant decontaminant formulation. The soapy water used here was prepared from General Purpose Detergent: Liquid NonIonic, Water Soluble, Type 1; National Stock Number 7930-00-282-9699. This soap is specified in the decontamination field manual (FM) 3-11.5, and it aligns with the current treatment process for field conditions.[8,9] The soapy water was prepared by mixing 138 µL of the detergent into 250 mL of deionized water.

DD is a reactive decontaminant that contains an aqueous-based microemulsion carrier.[10] It is a three-part solution that generates peracetic acid, an oxidative compound that aids in chemical neutralization and disinfection. DD was purchased from First Line Technology in a ready-to-use 200 mL kit and was prepared just before testing in accordance with the manufacturer's directions.

SDS is a reactive, solvent-based decontaminant that is applied like a paint coating. It was under development at the time of this study. The SDS formulation contains various inert components, two active ingredients (zirconium hydroxide and 1,3-dibromo-5,5-dimethylhydantoin), and a solvent. The slurry was prepared fresh on each day of testing.

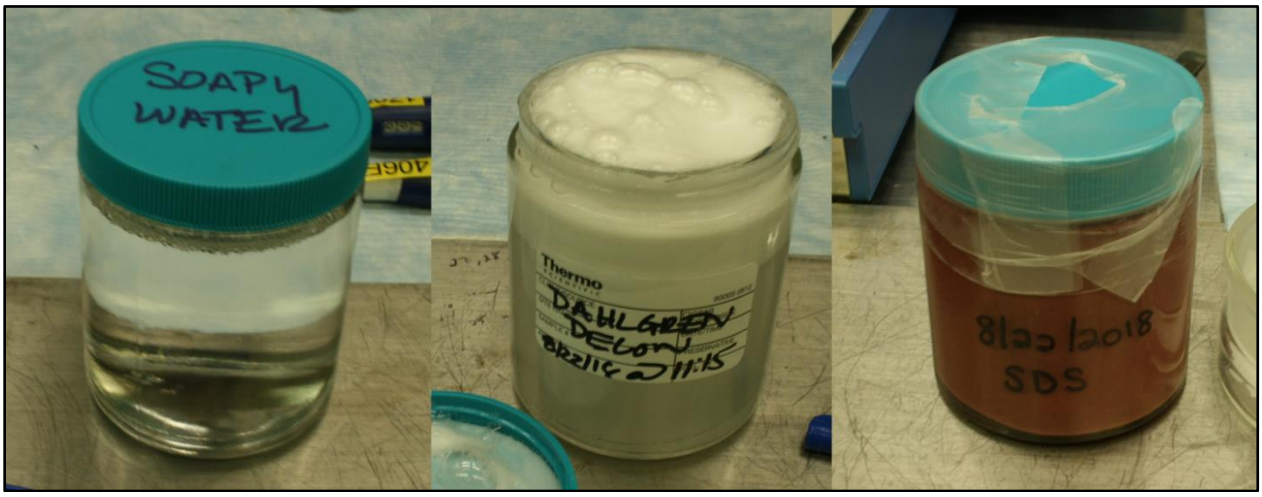

Figure 4. Fully prepared decontaminants for use in panel study.

Following the 60 min age time, the contaminated coupons were treated with 1 mL of decontaminant (Figure 5). The soapy water and DD were applied to the surface using a positive-displacement pipet. SDS has a thick consistency, so it was deposited onto the coupon and then spread across the surface with a spatula. The decontaminants were retained on the panels for 15 min.


Figure 5. DD on the surface of an SS coupon (left), and SDS decontaminant being applied to the surface of a PU paint-coated coupon (right).

2.5 Post-Decontamination Rinse, Quench, and Panel Extraction

Following the decontamination period, the panels were rinsed. Each panel was rinsed three times with 20 mL of deionized water to remove residual contaminant and decontaminant from the surface, as shown in Figures 6 and 7.

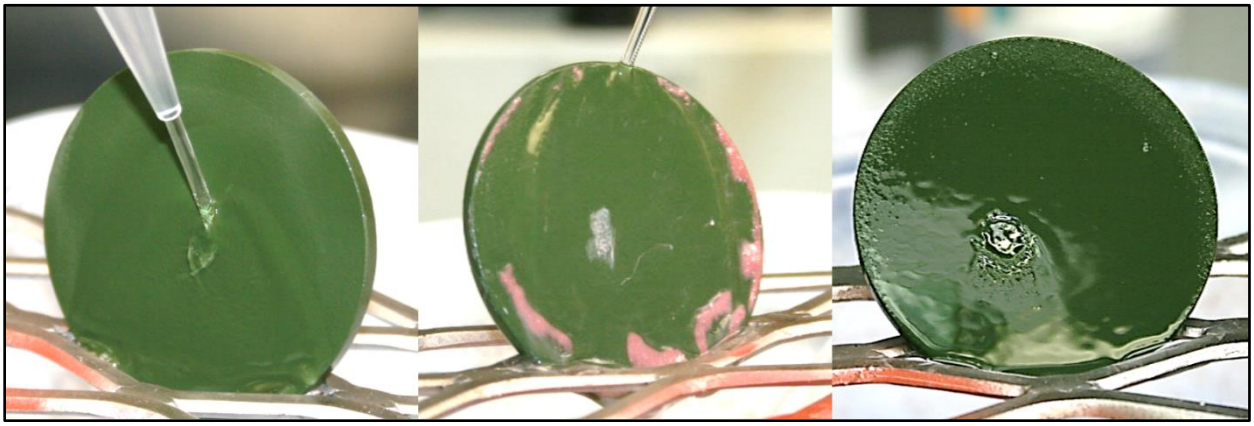

Figure 6. Post-decontaminant rinse of PU paint-coated coupons that were contaminated with neat carfentanil free base then decontaminated with soapy water (left), SDS (middle), and DD (right).

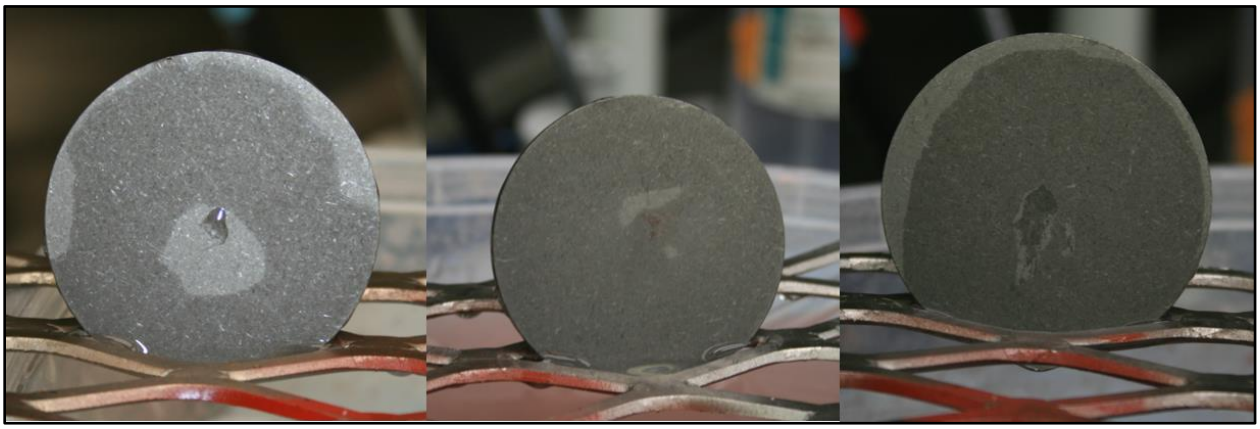

Figure 7. SS coupons after decontamination with soapy water (left), SDS (middle), and DD (right). Photographs show residual carfentanil free base remaining on the surface after three 20 mL post-decontamination rinses.

The panels treated with the reactive decontaminants, DD and SDS, were given an additional treatment to quench the activity of any residual decontaminant that may have remained on the panel surfaces. Preliminary studies demonstrated that sodium thiosulfate was an effective reducing agent to quench residual peracetic acid, and that glacial acetic acid and tetrahydrothiophene were able to quench the SDS. The quenching reagents were applied directly to the surface of the rinsed panel. The DD was quenched with 250 µL of a saturated solution of sodium thiosulfate pentahydrate, and the SDS was quenched with 200 µL of glacial acetic acid followed by 81 µL of tetrahydrothiophene.

Following the quenching step, the remaining contaminant was extracted by placing each panel in an 8 oz jar with 20 mL of methanol for 60 min. At the end of the extraction period, an aliquot of the extraction solution was removed and diluted for analysis. The RA for each sample was determined from the concentration of carfentanil in the extraction solution, which was quantified using an Applied Biosystems (Waltham, MA) API5500 QTrap triple-quadrupole MS, equipped with the TurboV ion source. Sample introduction and chromatography were performed with an Agilent Technologies (Santa Clara, CA) 1290 Infinity series UHPLC system, which included the Infinity binary pump, degasser, thermal column compartment, high-performance automatic liquid sampler (ALS), and the ALS thermostat. Sample effluent was directed from the liquid chromatograph directly to the TurboV ion source of the API5500 MS system. Instrument operation and data analysis were performed with the Applied Biosystems Analyst software package (v. 1.5.1). Details on the use of the chromatography platforms are published elsewhere.[11] Analysis conditions are summarized in Table 3.

Table 3. Analysis Parameters for Carfentanil

Liquid Chromatography Parameters		
Mobile phase A: 0.1% formic acid in deionized water		
Mobile phase B: 0.1% formic acid in acetonitrile		
Gradient:		
Time (min):	%A	%B
0.00	70.0	30.0
0.50	70.0	30.0
1.25	20.0	80.0
1.50	20.0	80.0
1.60	70.0	30.0
2.25	70.0	30.0
Flow rate: 0.5 mL/min		
Analytical column: Waters (Milford, MA) Acquity HSS T3, 1.8 µm, 2.1 × 50 mm		
Typical column pressure: 330 bar		
Analytical column temperature: 40 °C		
Injection volume: 1 µL		
Autosampler temperature: 5 °C		
Post-injection needle wash: 10 s of mobile phase B		
MS Parameters		
Scan type: multiple reaction monitoring (MRM)		
Polarity: positive mode		
Curtain gas: 30		
CAD gas: medium		
Source temperature: 500 °C		
GS1: 45		
GS2: 55		
Declustering potential: 100		
Exit potential: 10		
Collision energy: 25 (for all analytes)		
MRM for carfentanil: 395.2 > 335.2 MRM for norcarfentanil: 291.2 > 142.1 MRM for carfentanil-d_5 (internal standard): 400.2 > 340.2		

3. RESULTS AND DISCUSSIONS

3.1 RA and Log Difference (LD)

As previously indicated, RA was the metric by which decontaminant efficacy was evaluated, in accordance with the SD2ED. Better performing decontaminants result in lower RA values. The decontamination test methodology also indicates that starting challenge is measured as grams of contaminant per square meter of material.[5] It is worth noting, however, that when RA is compared across test conditions where the starting challenge varies, the RA measurement alone is not enough to determine relative decontaminant performance.

For this study, a log difference (LD) calculation was used to compare the RA results:

$$\mathrm{LD} = \log_{10}\frac{RA_{\mathrm{test}}}{RA_{\mathrm{reference}}} = \overline{\log_{10}(RA_{\mathrm{test}})} - \overline{\log_{10}(RA_{\mathrm{reference}})} \quad (1)$$

where RA_{test} is the remaining mass of contaminant for each coupon, and $RA_{\mathrm{reference}}$ is the mass of contaminant delivered to the coupon (starting challenge).

For this calculation, the mean of the log-transformed data is used, and the variance is used to calculate the 95% confidence interval on the difference, as defined in Procedure 5 of the SD2ED.[5] LD was used to account for potential non-normal distributions and the large variance often observed with panel test results. In this case, a positive LD indicates how much less mass was transferred after decontamination treatment as compared to the reference mass. If an LD error bar overlaps zero, then the difference is not statistically significant. For the samples contaminated with methanol solutions of carfentanil, a consistent mass was delivered across the panels and DCS; therefore, $RA_{\mathrm{reference}}$ is the average mass from the associated DCS measurement. For the neat free base, however, the mass delivered to the panels varied from sample to sample and among DCSs. Therefore, for the samples contaminated with the neat free base, the $RA_{\mathrm{reference}}$ for each sample was the individual, gravimetrically determined mass delivered to each panel (listed in Table 2) instead of the DCS.

Figure 8 shows the results of the RA measurements on a log scale for all samples and conditions tested. The results for the solvent-delivered contaminants are provided in Figure 8a, and the results for the neat free base contamination are displayed in Figure 8b. The average starting challenge for the solvent-delivered contaminants is indicated by the single dashed red line in Figure 8a, and the sample-specific contaminant dose masses for the free base are shown by the individual red lines in Figure 8b.

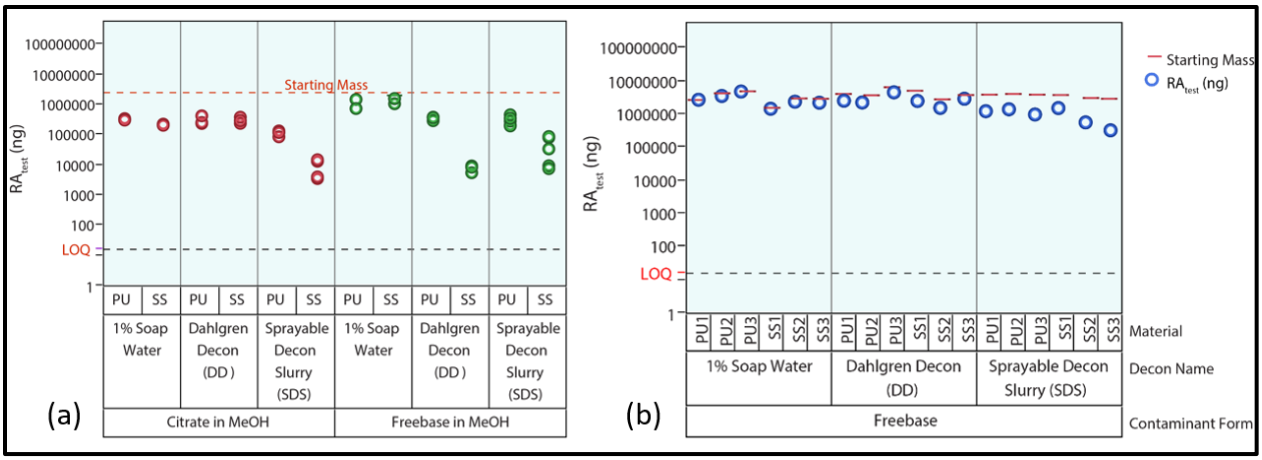

Figure 8. *RA*test for SS and PU paint contaminated by carfentanil in three different forms and after three different decontamination treatments. (a) Results for contamination with carfentanil citrate and free base, both dissolved in methanol. Dashed line is the average DCS across all conditions. (b) Results for contamination by neat carfentanil free base. Starting mass for each replicate is shown for each decontaminant–material pair.

Figure 9 shows the LD results for all conditions in the study, separated by material surface. Positive LD indicates how much less mass was remaining on the panel after decontamination treatment as compared to the reference mass; therefore, the higher the LD, the more effective the decontamination process.

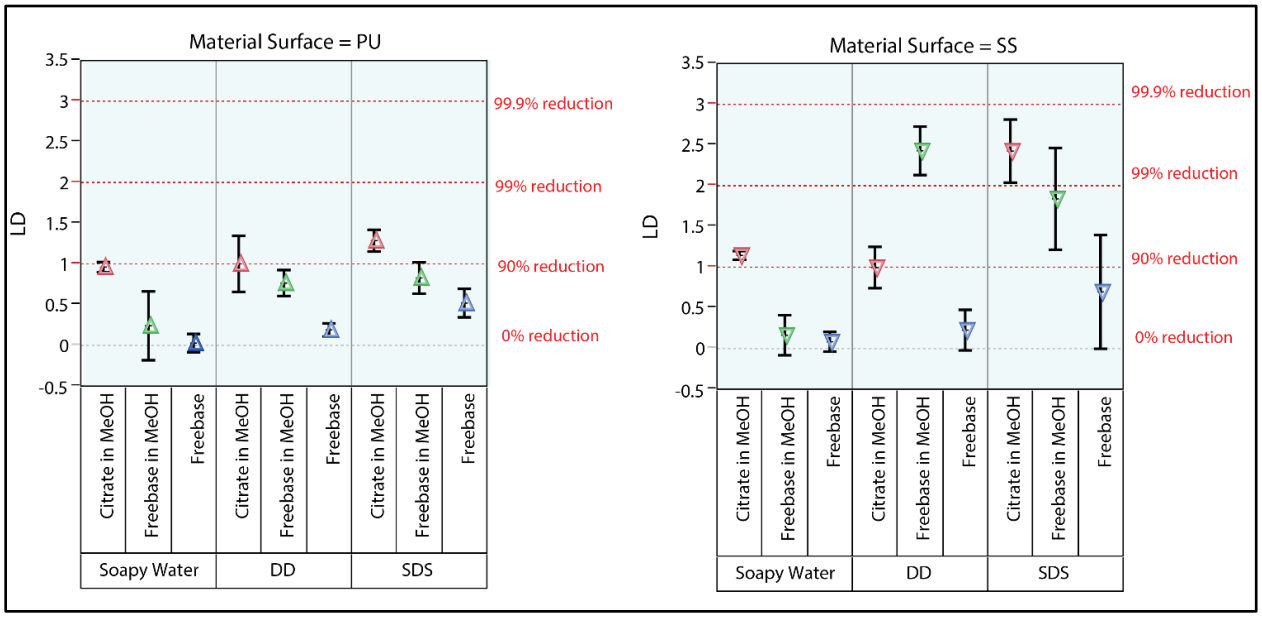

Figure 9. LD results (mean and 95% confidence interval) by material type for each condition evaluated.

3.2 Discussion

Based on the LD data presented in Figure 9, several conclusions can be made from the study results. Overall, three basic parameters (carfentanil contaminant form, decontaminant, and material surface type) were varied and combined in 18 different configurations for evaluation. The results in Figure 9 show that, in all but one condition (DD on SS for free base in methanol), the LD across the contaminant–decontaminant–material combinations was higher for the citrate than the free base forms of carfentanil. The higher LD suggests that solvated citrate is more easily removed from surfaces than the free base, regardless of whether the free base is solvated or neat. Consistent with previous results,[7] in the absence of a reactive component, the enhanced water solubility of the citrate over the free base allows for better physical removal by soapy water. Furthermore, the citrate in methanol achieved a minimum of 90% reduction in every condition, whereas the maximum reduction of the neat free base was ~77 %. This reduction was achieved with the reactive, solvent-based SDS on SS. Comparison of the two material types revealed that, in every case, decontaminant performance was equal or better on the impermeable SS than on the more permeable PU paint.

The performance of the reactive decontaminants was mixed. DD performed best against the methanol-delivered carfentanil free base on SS. This condition resulted in an ~2.5 LD, indicating more than 99% of the contaminant reacted or was removed from the surface. However, this same level of performance was not achieved on the PU surface or with either PU paint or SS contaminated with carfentanil citrate in methanol. Furthermore, when applied to the neat free base, DD performed only slightly better than soapy water. Because DD performance varied widely across contaminants and materials, this may be an area for further study with additional replicates and possibly more surface types.

Finally, SDS performed better than both soapy water and DD when applied to the methanol-delivered citrate and the neat free base, and it demonstrated similar performance to DD in the case of the methanol-delivered free base. In all cases studied, SDS achieved better performance on the impermeable SS than on the permeable PU paint; the most notable difference was observed when the contaminant was delivered in methanol.

4. CONCLUSIONS

This work was primarily a scoping study; the primary objective was to elucidate factors that may be influential in the study of decontaminants for materials contaminated with opioids. The results underscore that the outcome of any decontamination process is highly dependent upon the relationship between contaminant, material, and decontaminant. This is consistent with results of past decontamination evaluation studies for other contaminant classes.

The results of this study indicate that decontaminant efficacy varies significantly with contaminant form and the method of delivery to the test surface. With one exception, the LD data showed that carfentanil citrate, when delivered in methanol solution, is generally more easily removed from the surface of the PU paint and SS panels than the free base, regardless of whether the free base is delivered in methanol solution or as neat material.

These results also show that, when comparing decontaminant efficacy across a single contaminant form in each of the three forms studied, reactive decontaminants were more efficient than nonreactive soapy water at removing or neutralizing the contaminant.

Finally, this scoping study suggests that many unanswered questions remain regarding opioid decontamination, and these questions deserve research and attention. Because many variables are involved, including opioid analogs, contaminant forms, reactive chemistries and associated byproducts, and contact transfers and associated health effects, future efforts may best be directed by incorporating an experimental design and modeling effort.

LITERATURE CITED

1. Riches, J.R.; Read, R.W.; Black, R.M.; Cooper, N.J.; Timperley, C.M. Analysis of Clothing and Urine from Moscow Theatre Siege Casualties Reveals Carfentanil and Remifentanil Use. *J. Anal. Toxicol.* **2012,** *36* (9), 647–656. DOI: 10.1093/jat/bks078

2. O'Donnell, J.; Gladden, R.M.; Mattson, C.L.; Kariisa, M. Notes from the Field: Overdose Deaths with Carfentanil and Other Fentanyl Analogs Detected — 10 States, July 2016–June 2017. *MMWR Morb. Mortal Wkly. Rep.* **2018,** *67*, 767–768; DOI: 10.15585/mmwr.mm6727a4

3. Sweat, M.; Schenning, A.; Chesebrough, M.; Ruth, J.; Schenning, C.; Sheahy, M.; Smallwood, S. Evaluation of Contact Transfer of Pharmaceutical Based Agents from Contaminated Surfaces to Glove Materials*;* U.S. Army Combat Capabilities Development Command Chemical Biological Center: Aberdeen Proving Ground, MD; UNCLASSIFIED Report; in progress.

4. Miller, C.J.; Yoder, T.S. Explosive Contamination from Substrate Surfaces: Differences and Similarities in Contamination Techniques Using RDX and C-4. *Sens. Imaging* **2010,** *11*, 2, 77–87.

5 Lalain, T.; Mantooth, B.; Shue, M.; Pusey, S.; Wylie, D. *Chemical Contaminant and Decontaminant Test Methodology Source Document, Second Edition*; ECBC-TR-980; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2012; UNCLASSIFIED Report (ADA566601).

6. D'Onofrio, T.; Schenning, A.; Sheahy, M.; Ruth, J.; Chesebrough, M. *Permeation of Free Base Carfentanil through Protective Materials*; CCDC CBC-TR-1591; U.S. Army Combat Capabilities Development Command Chemical Biological Center: Aberdeen Proving Ground, MD, 2019; UNCLASSIFIED Report (AD1076692).

7. Schenning, A.; Sheahy, M.; Smallwood, S.; Morrissey, K.; Ruth, J.; Chesebrough, M. *Effect of Contaminant Form on the Contact Transfer of an Opioid to Latex and ACU Cloth*; CCDC CBC-TR-1665; U.S. Army Combat Capabilities Development Command Chemical Biological Center: Aberdeen Proving Ground, MD, 2021 (AD1123418).

8. Mantooth, B.; Stevenson, S.; Davies, J.; Escarsega, J.; Pearl, T.; Ruth, J. *Development of a Chemical Agent Resistance Method for Chemical Agent Resistant Coatings*; CCDC CBC-TR-1608; U.S. Army Combat Capabilities Development Command Chemical Biological Center: Aberdeen Proving Ground, MD, 2020 (AD1090020).

9. *CBRN Decontamination: Multiservice Tactics, Techniques, and Procedures for Chemical, Biological, Radiological, and Nuclear Decontamination*; FM 3-11.5; U.S. Department of Defense: Washington, DC, 2006; UNCLASSIFIED Field Manual.

10. Karney, T. Did You Know? There's More to Dahlgren Decon Than Just Peracetic Acid; *In News*, 8 October 2019. First Line Technology, https://www.firstlinetech.com/did-you-know-theres-more-to-dahlgren-decon-than-just-peracetic-acid/?v=7516fd43adaa (accessed 20 January 2023).

11. Shue, M.; Lalain, T.; Mantooth, B.; Humphreys, P.; Hall, M.; Smith, P.; Sheahy, M. *Low-Level Analytical Methodology Updates to Support Decontaminant Performance Evaluations*; ECBC-TR-883; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2011; UNCLASSIFIED Report (ADA546021).

ACRONYMS AND ABBREVIATIONS

ALS	automatic liquid sampler
CWA	chemical warfare agent
DCS	dose-control sample
DD	Dahlgren Decon solution
DEVCOM CBC	U.S. Army Combat Capabilities Development Command Chemical Biological Center
LD	log difference
MRM	multiple reaction monitoring
MS	mass spectrometry
PBA	pharmaceutical-based agent
PDCATTI	Product Director for Cross-Commodity Advanced Threats and Test Infrastructure
PU	polyurethane
RA	remaining agent
SD2ED	Chemical Contaminant and Decontaminant Test Methodology Source Document, Second Edition
SDS	sprayable decontaminant slurry
SS	stainless steel
UHPLC	ultra-high-performance liquid chromatography
VX	*O*-ethyl-*S*-(2-diisopropylaminoethyl) methyl phosphonothiolate

DISTRIBUTION LIST

The following individuals and organizations were provided with one electronic version of this report:

U.S. Army Combat Capabilities Development Command Chemical Biological Center (DEVCOM CBC)
FCDD-CBR-PD
ATTN: Schenning, A.
Morrissey, K.

DEVCOM CBC Technical Library
FCDD-CBR-L
ATTN: Foppiano, S.
Stein, J.

Defense Technical Information Center
ATTN: DTIC OA


U.S. ARMY COMBAT CAPABILITIES DEVELOPMENT COMMAND
CHEMICAL BIOLOGICAL CENTER