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CHEMICAL BIOLOGICAL CENTER

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# Characterization of the Multi-Point Pin Technology (MPPT) Device: FY19 IDEAS Program Report

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The 2019 IDEAS (Innovative Development of Employee Advanced Solutions) program provided the opportunity to fully invest in the development of a modification to the established low-volatility agent permeation (LVAP) method. The current LVAP method requires the material under test to be perfectly flat to create sufficient contact to collect permeated agent for analysis. The goal of this program was to implement new technology to enable the LVAP method to be used with test surfaces that are not totally flat. A test device was developed that creates uniform contact across uneven or featured surfaces. The contact transfer hazard test method was modified to evaluate the effectiveness of the new Multi-Point Pin Technology (MPPT) device. The new MPPT weight was compared to the original LVAP weight on flat and uneven surfaces. Results showed that on flat surfaces, the MPPT and LVAP weights both provided adequate contact with no statistical difference in uptake. However, on featured surfaces, use of the MPPT weight resulted in 98% recovery, whereas the LVAP weight recovery was ≤1%. The addition of an electromagnetic plate is being explored to increase the initial contact pressure of the MPPT weight to 1 psi and provide the ability to adjust contact pressure instantaneously.

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# PREFACE

The work described in this report was authorized under the FY19 IDEAS (Innovative Development of Employee Advanced Solutions) program. The work was started in January 2019 and completed in March 2020. At the time this work was performed, the U.S. Army Combat Capabilities Development Command Chemical Biological Center (CCDC CBC; Aberdeen Proving Ground, MD) was known as the U.S. Army Edgewood Chemical Biological Center (ECBC).

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## Acknowledgments

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- Mr. Charles Steinert (Advanced Design and Manufacturing, Engineering Directorate, CCDC CBC) for design and manufacturing of test materials.

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# EXECUTIVE SUMMARY

Existing methods of personal protective equipment (PPE) testing cannot contend with uneven or featured material surfaces. As a result, improvements are needed to support accurate characterization of these features at the laboratory scale. The development of the low-volatility agent permeation (LVAP) test method brought forth a significant advancement in test capability for evaluating the permeation of low-volatility agents by creating uniform contact between the test and sorbent materials. To ensure contact, the method requires a 1 lb weight with a 1 in. contact area to generate a 1 psi contact pressure across the testing area. However, in previous studies using the LVAP method, testing of featured surfaces was problematic because the weight could not apply uniform pressure across an uneven surface. The Individual Protection community has identified improvements to swatch testing of features through the Test and Evaluation Capabilities and Methodologies Integrated Process Team (TECMIPT), sponsored by the office of the Deputy Under Secretary of the Army for Test and Evaluation. Current programs are limited to evaluating seams and closures with simulants as part of a full system test. A component-level test is necessary to evaluate these materials as a risk reduction measure during the development phase, prior to full system testing.

To address this, a novel test device was developed to create uniform contact across featured surfaces. The multi-point pin technology (MPPT) device is composed of an array of identical pins captured in an outer assembly. The pins move freely and independently on the vertical axis to create uniform contact across featured surfaces.

The MPPT device was evaluated using three methods: (1) contact pressure mapping with an I-Scan test pressure measurement system from Tekscan, Inc. (Boston, MA); (2) agent contact transfer hazard testing with *O*-ethyl-*S*-(2-diisopropylaminoethyl) methyl phosphonothiolate (VX) from flat and featured stainless steel 2 in. panels; and (3) LVAP comparison, measuring VX permeation through flat and featured 2 in. swatches of a standard chemical-resistant, air-permeable suit material.

Contact pressure mapping was completed using the I-Scan test pressure measurement system. Contact using the MPPT device was compared to that obtained using the traditional flat LVAP weight on flat and featured stainless steel panels. Each featured panel consisted of a flat stainless steel disk with a second half-circle disk welded on top that created a 1/8 in. rise. The LVAP weight showed adequate contact on the flat surface, but it only showed three total points of contact on the featured surface. The MPPT device showed uniform contact on both the flat and the featured surfaces.

The agent contact transfer hazard test was used to compare the MPPT device with the traditional LVAP weight. The test measures the uptake of VX from the surface of both flat and featured stainless steel panels. A divinyl benzene pad was used as the contact sampler. The MPPT device and the LVAP weight both showed adequate contact on the flat surface. This was proven by measuring >95% VX recovery. On the featured surface, the MPPT device created adequate contact that resulted in 98% recovery from polished stainless steel panels.

In the LVAP comparison study, the MPPT device and the traditional LVAP weight were tested in accordance with TECMIPT Test Operating Procedure (TTOP) 8-2-503, *Low Volatility Agent Permeation (LVAP) Swatch Testing* to identify the permeation of VX through a chemical-resistant, air permeable suit material. Both the LVAP weight and the MPPT device provided adequate contact across the flat surface; the relative standard deviations (RSDs) for flat swatches were 2.7 and 11.8, respectively. The variability increased greatly when the featured test swatches were evaluated. The RSDs for the LVAP weight and the MPPT device from the featured surface were 40 and 52, respectively. Even with the increased variability, greater permeation was measured using the MPPT device, demonstrating that the MPPT device created more contact between the swatch and the sampling media.

The current MPPT device was shown to create uniform contact across featured surfaces and can be utilized in its present configuration with agent contact transfer hazard testing across complex geometries.

The current version of the MPPT device is significantly lighter than the traditional LVAP weight. Although the MPPT device created uniform contact across the featured test swatch, for permeation testing, it is suspected that the total weight needs to increase, to ensure that adequate contact exists between the contact sampler and the underside of the test swatch. This limitation is being evaluated. Different construction materials that would increase the contact pressure of the overall MPPT device are being evaluated.

An electromagnetic plate is being developed that will be integrated directly into the current LVAP infrastructure. With the use of the MPPT device, the plate will allow for sustained and uniform contact across featured surfaces. This novel capability will also allow for instantaneous adjustment of contact pressures using the LVAP methodology, in accordance with customer requests. The electromagnetic plate will allow for the testing of pressures that directly correspond to those of a soldier sitting, kneeling, carrying a rucksack, leaning against a wall, and potentially, carrying a fellow soldier. Having the ability to efficiently test PPE material effectiveness under these conditions will aid in making informed decisions regarding warfighter safety.

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# CHARACTERIZATION OF THE MULTI-POINT PIN TECHNOLOGY (MPPT) DEVICE: FY19 IDEAS PROGRAM REPORT

## 1. OBJECTIVE

The objective of this program was to show proof of concept of a novel testing device designed to provide uniform contact on uneven and complex surfaces. The program was designed to show improvement against the traditional low-volatility agent permeation (LVAP) testing weight using the contact transfer hazard methodology.

This report documents the characterization results of the Multi-Point Pin Technology (MPPT) device, which was demonstrated to provide uniform contact across featured surfaces.

## 2. BACKGROUND

The objective of this effort was to develop a laboratory-scale test device to address a gap in permeation testing of personal protective equipment (PPE) against chemical warfare agents (CWAs), and specifically, to address evaluation of PPE features such as seams, folds, and closures. Current PPE testing methods cannot contend with uneven or featured surfaces, and no capability exists to accurately characterize these features at the laboratory scale. The development of the LVAP test method brought forth significant advancement in test capability to evaluate the permeation of low-volatility agents by creating uniform contact between the testing material and the sorbent material beneath. To ensure contact, the method requires a 1 lb weight with a 1 in. contact area to generate a 1 psi contact pressure across the testing area. However, in previous studies using LVAP testing, the evaluation of featured surfaces was shown to be problematic because the flat weight could not apply uniform pressure across an uneven surface. The MPPT device was designed and developed to address this need.

## 3. SCOPE

This report describes and documents the initial characterization of the MPPT device. The goal of this effort was to demonstrate proof of concept of an innovative MPPT test system that will address the challenges associated with permeation evaluation of featured PPE surfaces.

## 4. MPPT DEVICE DESCRIPTION

The MPPT device is composed of an array of identical pins captured in an outer assembly. The pins move freely and independently on the vertical axis to create uniform contact across featured surfaces. In the current MPPT device, aluminum pins are held together in a polycarbonate housing. The MPPT device is designed to have a 1 in. contact area, which is the same as that for the current LVAP weight. Figure 1 shows a computer-aided design (CAD) drawing of the MPPT device. Figure 2 shows the device creating a 1 in. contact area on both flat and featured surfaces.

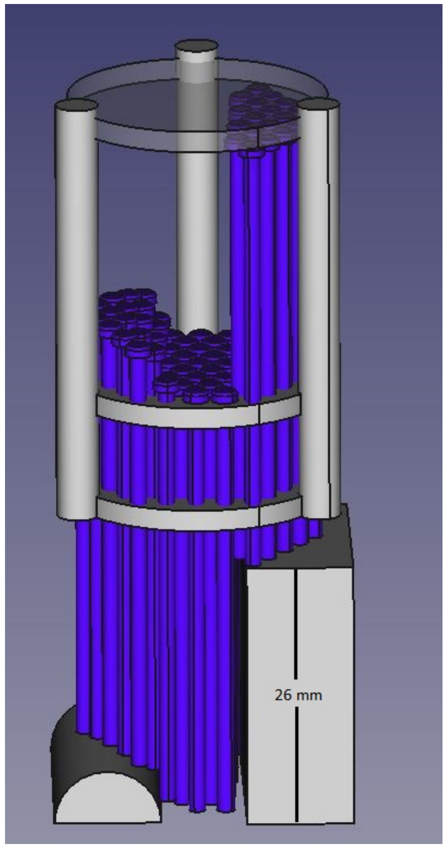


Figure 1. CAD drawing of MPPT device.

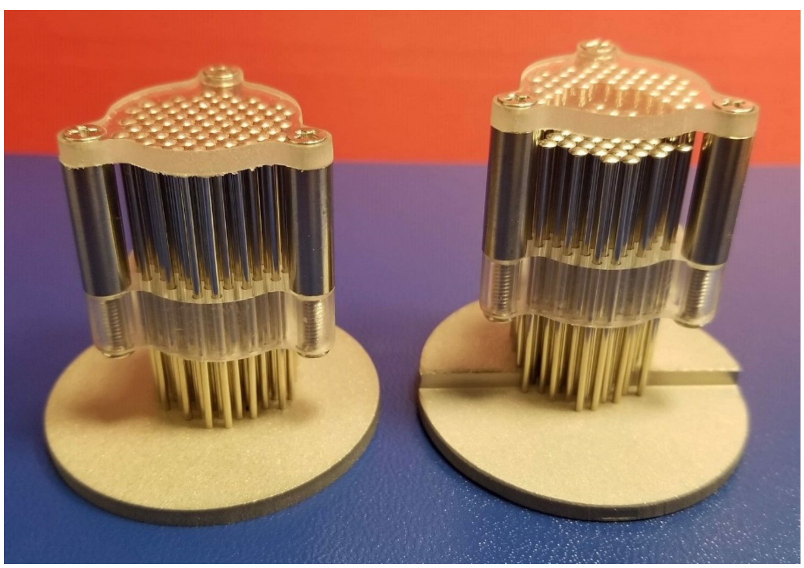

Figure 2. Photograph of MPPT device on flat (left) and featured (right) surfaces.

# 5. EXPERIMENTAL METHOD

## 5.1 Contact Pressure Evaluation

The purpose of this experiment was to determine whether the MPPT device could generate uniform contact across featured surfaces. The MPPT device was compared to the LVAP weight as applied to both flat and featured surfaces (Figure 3).

For each evaluation, the test panel was a 2 in. stainless steel disk. The featured-surface disks were created by adding a half-circle stainless steel piece to the top of the flat disc. The half-circle sat 1/8 in. above the 2 in. disc surface.

The pressure for each configuration was measured using an I-Scan test pressure measurement system from Tekscan, Inc. (Boston, MA). The I-Scan measurement system was equipped with a thin pressure sensor (Figure 4) that provided an array of measurement over an approximately 2 in. surface. The thin, flexible probe was placed between the stainless disk and either the LVAP weight or the MPPT device.

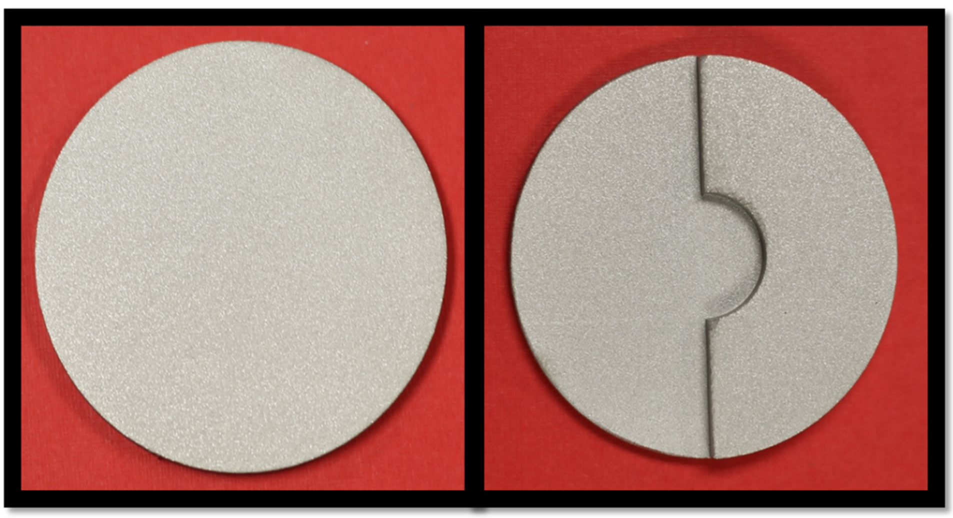

Figure 3. Stainless steel panels for pressure measurements: flat (left) and featured (right).

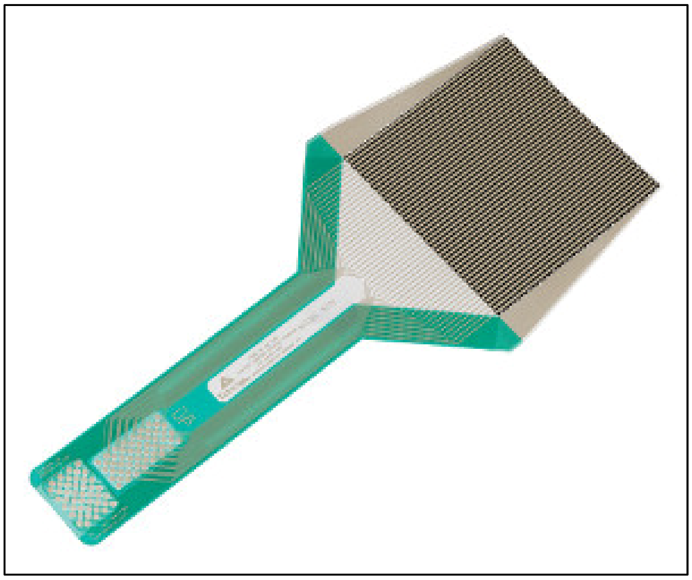

Figure 4. Thin pressure sensor with spatial array.

## 5.2 Agent Contact Transfer Hazard Test

To determine the efficacy of the MPPT device to create contact across featured surfaces, an agent contact transfer hazard test was designed to compare the flat LVAP weight with the MPPT across both flat and featured surfaces. This approach eliminates the inherent variability in permeable materials and instead focuses on evaluating the contact made by the testing device. The flat and featured surfaces were the same materials that were evaluated during the contact pressure test.

Stainless steel was chosen as a substrate because it is resistant to absorbing CWAs and it represents a contact transfer hazard rather than a permeation hazard. *O*-ethyl-*S*-(2-diisopropylaminoethyl) methyl phosphonothiolate (VX) was selected as the CWA for evaluation because it has low volatility, and it was used in the verification and validation of the LVAP methodology.[1] Divinyl benzene pads (DVBs) were used as the contact sampler collection media. These pads were previously shown to be effective for collection of VX from a surface.[1] In the first part of the experiment, VX uptake was evaluated from smooth stainless steel surfaces with use of both the LVAP weight and the MPPT device. In the second part of the experiment, VX uptake was evaluated from featured stainless steel surfaces with use of both the LVAP weight and the MPPT device.

For this experiment, a single 1 µL droplet of neat VX was placed onto each panel. The polished surface of the panels allowed the VX to remain as single sessile droplets. For flat panel testing, the VX droplet was placed directly in the middle of the stainless steel panel. For featured surface testing, the VX droplet was placed inside the edges of the circle while remaining on the horizontal surface. A 50 µL Hamilton gas-tight syringe equipped with a repeating dispenser was used to precisely deliver the individual VX droplets. Figure 5 shows a single VX droplet on a featured surface.

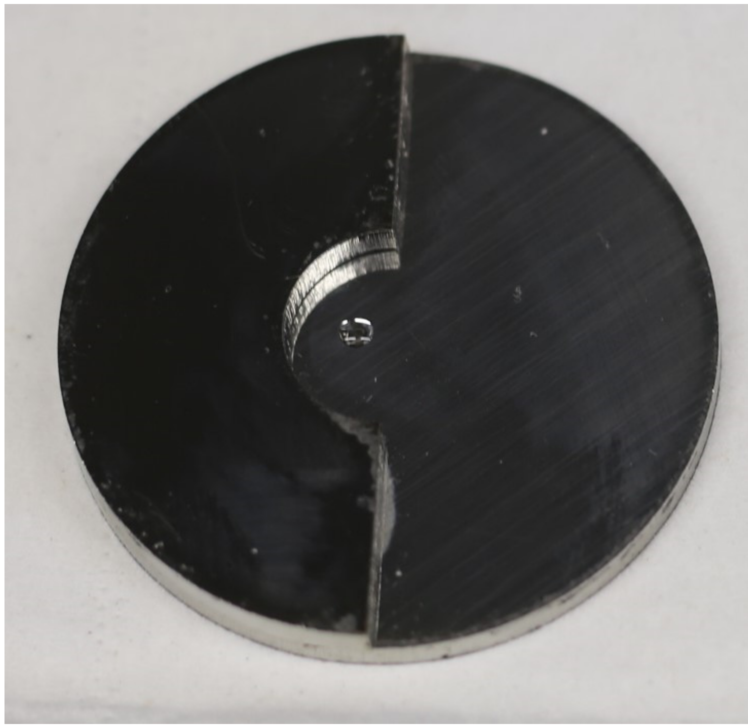

Figure 5. Featured stainless steel panel with a single VX droplet.

After one individual panel was contaminated, the DVB pad was placed onto the surface. This was immediately followed by placement of either the LVAP weight or the MPPT device, depending on the test scenario.

Each sample had a dwell time of 15 min in accordance with (IAW) Test and Evaluation Capabilities and Methodologies Integrated Process Team (TECMIPT) Test Operating Procedure (TTOP) 8-2-061A: *Chemical Decontaminant Testing*.[2] After the dwell time had elapsed, each DVB was removed and placed into an assigned extraction jar with 20 mL of acetone. The stainless steel panels were solvent-rinsed with 20 mL of acetone, and the rinsate was collected. The panels were solvent-rinsed instead of being extracted for 60 min to reduce the amount of potential contaminant from the panels that could interfere with the analytical instrumentation.

Each extraction was allowed to sit for at least 60 min before aliquots were pulled and sent for analysis. The extracts were analyzed for total VX uptake via liquid chromatography–triple quadrupole mass spectrometry (LC–MS/MS). Figure 6 shows the test in progress with all samples sitting during the 15 min dwell time.

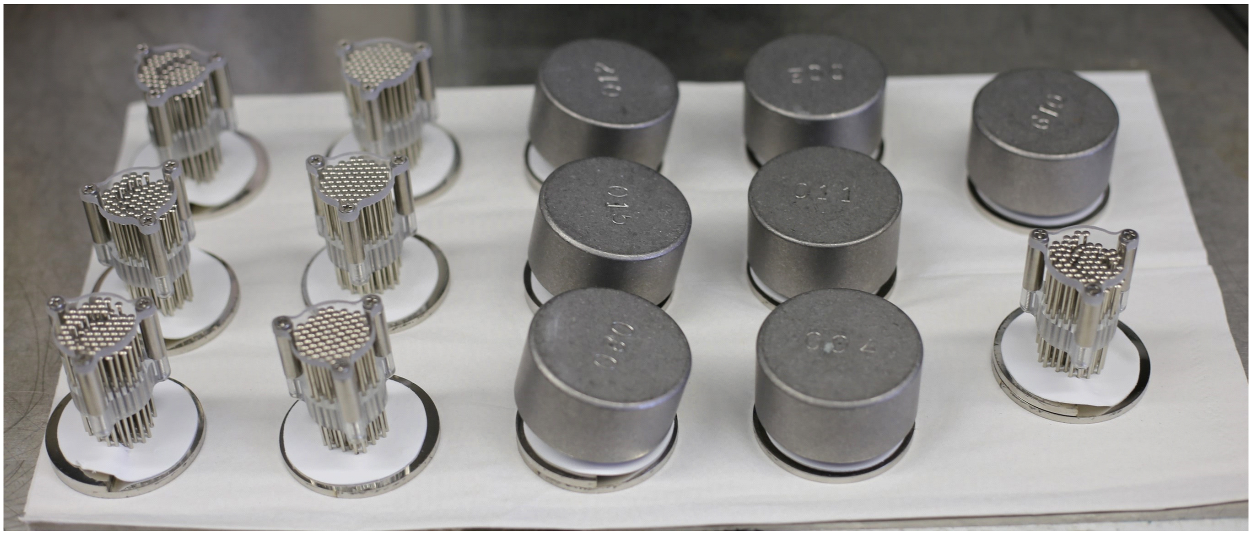

Figure 6. Agent contact transfer hazard test design.

### 5.3 LVAP Test Comparison

The LVAP comparison test was designed to compare the effectiveness of the MPPT device with that of the traditional LVAP weight using the traditional LVAP methodology. The testing was conducted IAW TTOP 8-2-503, *Low Volatility Agent Permeation (LVAP) Swatch Testing*.[3]

The experiment was intended to evaluate the effectiveness of the MPPT and the LVAP weight on flat and featured or seamed test swatches. A standard chemical-resistant, air-permeable suit material was chosen for testing. This material was tested extensively during the development of the LVAP test method, and it was readily available. Figure 7 shows flat and featured swatches of this material as prepared for the experiment.

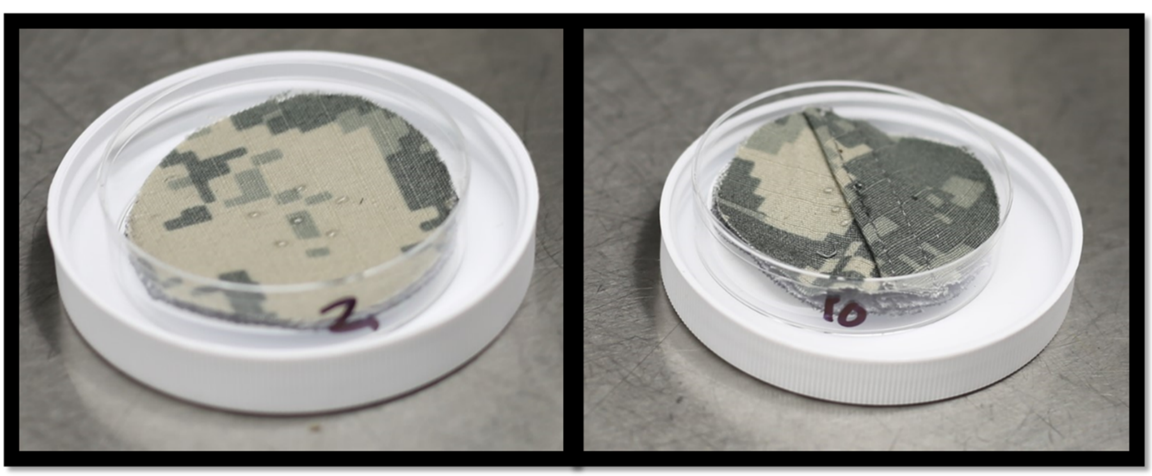

Figure 7. Flat (left) and featured and seamed (right) test swatches contaminated with VX.

VX was the CWA for evaluation because of its low volatility and its known interaction with the chosen test material. DVBs were used as a contact sampler. These pads were previously shown to be effective at collecting VX with use of the LVAP test method. All samples were tested at a 10 $g/m^2$ contamination density. The VX was delivered in six individual 1 mL droplets. A 50 mL Hamilton gas-tight syringe equipped with a repeating dispenser was used to accurately deliver the individual droplets. The droplets were placed into a pattern IAW TTOP 8-2-503. A schematic of the LVAP test fixture (Figure 8) shows the layout for the LVAP test method.

After each sample was spiked, it was placed into the LVAP temperature-controlled chamber (set at 32 °C) for a 24 h dwell time. Each test sample was then removed from the temperature-controlled chamber. The weight was removed and rinsed with acetone. The test cell was broken down, and the DVB was placed into an extraction jar filled with 20 mL of acetone. Each DVB was allowed to extract for at least 60 min before aliquots were pulled and sent for analysis. All samples were analyzed using LC–MS/MS.

Figure 8. LVAP test fixture.

## 6. RESULTS AND DISCUSSION

### 6.1 Contact Pressure Results

The I-Scan test pressure measurement system generated the pressure results graphically, as displayed in Figures 9 and 10. Both the LVAP weight and the MPPT device showed adequate contact on the flat surface.

On the featured surface, the LVAP weight created only three points of contact, of which only two were main contact points. This is shown in Figure 10 by the two large peaks on the graph. The LVAP weight did not show any contact across the rest of the featured surface.

Use of the MPPT device resulted in consistent contact on both the flat and the featured surfaces. Considerably more contact occurred on the featured surface using the MPPT device than was observed with use of the LVAP weight.

The LVAP weight averaged 1 lb of pressure across the flat surface; however, the system could not accurately measure the pressure due to the two individual points of pressure on the featured surface. The MPPT device averaged 0.1 lb of pressure across both the flat and the featured surfaces.

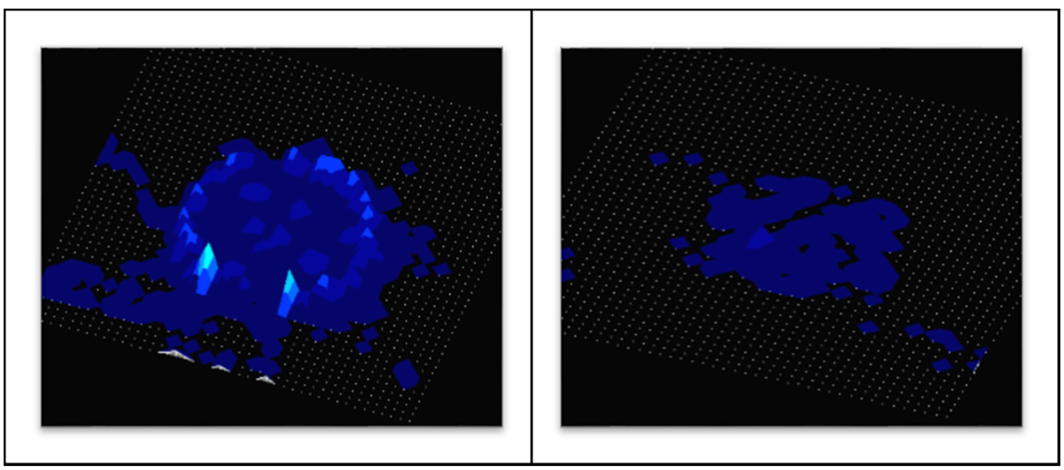

Figure 9. Contact pressure mapping on flat surface:
LVAP weight (left) and MPPT device (right).

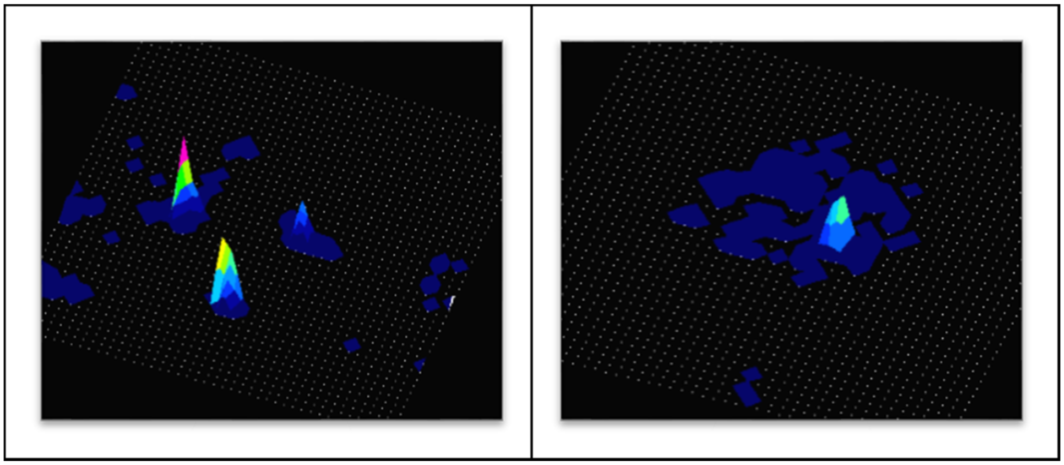

Figure 10. Contact pressure mapping on featured surface:
LVAP weight (left) and MPPT device (right).

## 6.2 Agent Contact Transfer Hazard Test Results

Experimental results are compiled in Table 1 and shown graphically in Figure 11. The MPPT device showed 98% VX recovery on the featured stainless panels. Note the non-detect result for the LVAP weight on the flat panel: 78% VX recovery was obtained after analysis of the acetone rinse from the stainless panel. All other solvent washes showed non-detect or ≤1%. This demonstrates proof of concept that the MPPT device generated adequate contact across featured surfaces.

The experiment was performed twice. Both tests were conducted under similar conditions except for the panel surface. For the first test, the panel surface was rolled, not polished. This allowed the VX droplet to spread across the surface instead of remaining as a sessile droplet. For the second test, the stainless steel panel surface was polished. This allowed the VX droplet to remain a sessile drop. This difference in panel surface is the reason for the increased recoveries from Test 1 to Test 2.

Table 1. Agent Contact Transfer Hazard VX Uptake Results

| Test Number (Panel Surface) | Device Type | Surface Condition | VX Recovery (%) |
|---|---|---|---|
| 1 (Rolled) | LVAP weight | Flat | 84 |
| | | Featured | 1 |
| | MPPT | Flat | 98 |
| | | Featured | 43* |
| 2 (Polished) | LVAP weight | Flat | 100 |
| | | Featured | ND |
| | MPPT | Flat | 97 |
| | | Featured | 98* |

*Test 1 was conducted on rolled stainless panels, and Test 2 was conducted on polished stainless panels.
ND, non-detect: 78% VX recovery from the stainless steel panel after a solvent rinse.

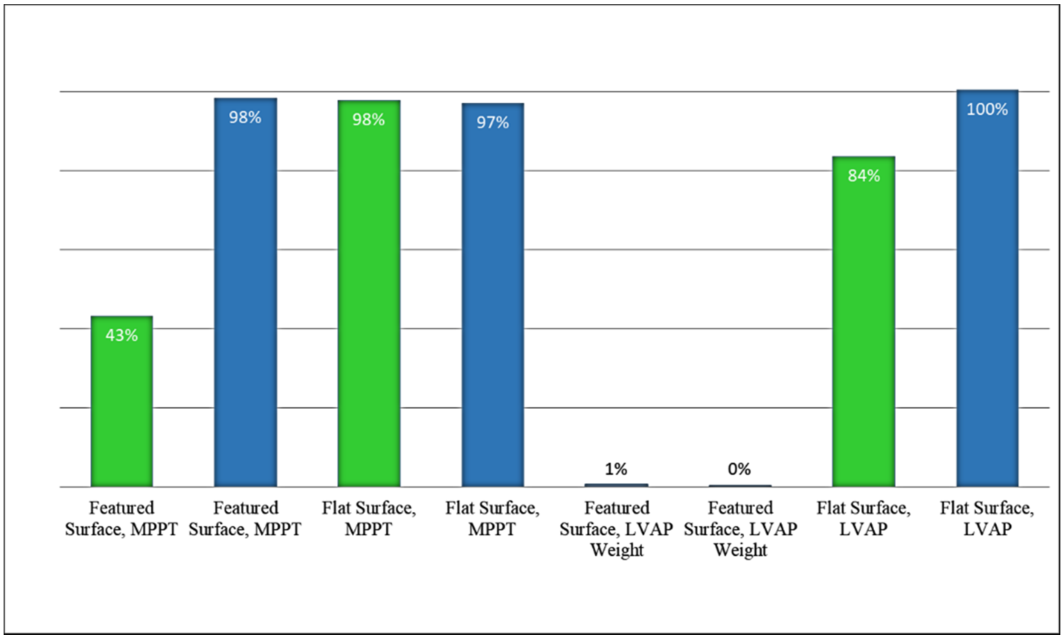


Figure 11. Agent contact transfer hazard: VX percent uptake from stainless steel panels.

## 6.3 LVAP Comparison Results

Experimental results for the LVAP comparison test are compiled in Table 2. Note that the relative standard deviation (RSD) for the flat surface with use of the LVAP weight was low, 2.69%. This was expected using the traditional LVAP methodology. The total VX permeation measured on the seamed material using the LVAP weight was significantly lower due to the lack of consistent pressure from the flat weight.

The RSD increased when the seamed test swatches were evaluated. The increased variability was due to the varying contact between the sampling media and the underside of the test swatch. The LVAP weight would not sit flat on the seamed surface, which caused uneven pressure across the surface. The MPPT device provided more uniform pressure across the test swatch.

The total permeation was lower on the featured surfaces due to the inconsistent contact generated by the LVAP weight set. The VX permeation increased on the seamed test swatch from the LVAP weight as compared to the MPPT device. This was because more consistent contact was generated by the MPPT device.

Table 2. VX Permeation: LVAP versus MPPT Comparison Results

| Material Type | Device Type | *n* | VX Permeation (µg) | Standard Deviation (µg) | RSD (%) |
|---|---|---|---|---|---|
| Flat surface | LVAP weight | 3 | 21.1 | 0.57 | 2.69 |
| Flat surface | MPPT | 3 | 17.58 | 2.08 | 11.84 |
| Featured/seam | LVAP weight | 3 | 6.85 | 2.71 | 39.59 |
| Featured/seam | MPPT | 3 | 14.17 | 7.37 | 51.98 |

## 7. SUMMARY AND CONCLUSION

The goal of this effort was to show proof of concept that the MPPT device would create adequate contact across a featured surface. The results of the pressure-mapping experiment provided confidence that the MPPT device could provide consistent pressure on both flat and featured surfaces. This hypothesis was confirmed with the results of the contact transfer hazard test, which showed the effectiveness of the MPPT device at creating contact across a complex surface. The MPPT device provided significantly more contact than the flat LVAP weight.

The results of the LVAP comparison test brought to light the need to increase the weight of the MPPT device. The current version of the MPPT device is considerably lighter than the LVAP weight. Heavier pins should be added to increase the overall contact pressure of the MPPT device. Increased weight would allow more consistent contact to be generated and, accordingly, more consistent results to be obtained.

## 8. FOLLOW-ON WORK

### 8.1 Electromagnetic Plate

Currently, the LVAP test method specifies that a 1 lb weight be used to generate a 1 psi pressure uniformly across the contamination area. The 1 psi used is similar to a "heavy touch" as discussed in industrial hygiene research journals.[1] The current version of the MPPT device does not provide 1 psi of pressure. Research is underway to identify a heavier pin that could increase the contact pressure.

U.S. Army Combat Capabilities Development Command Chemical Biological Center (Aberdeen Proving Ground, MD) is working on developing an electromagnetic plate that can assist with generating adequate contact pressure for the MPPT device. As planned, the electromagnetic plate will allow for instantaneous adjustment of pressure generated by the MPPT device or the LVAP weight.

Although this contact pressure has been sufficient for most customers, the question often arises as to whether pressure has any impact on agent permeation. Because the current weight condition is based on a "heavy touch", there has not been a characterization associated with sitting, kneeling, or other postures. Previous testing has been done (with limited resources) to characterize between 0.2 and 2.0 psi pressures on flat surfaces. The previous study did not show a statistical difference for the small pressure differential; however, much larger pressures have not been tested because of the material cost and space constraints within the current infrastructure. The conceptual Electromagnetic Multi-Point Pin Technology would use an electromagnetic plate to create pressure to augment the traditional 1 lb weight methodology. The plate would allow for sustained and uniform contact across featured surfaces with the use of the MPPT device. The addition of this electromagnetic plate would introduce the ability to easily adjust the contact pressure across all featured surfaces IAW customer requests. Introduction of the electromagnetic plate would also allow for the testing of pressures that directly correspond to those of a soldier sitting, kneeling, carrying a rucksack, leaning against a wall, and potentially, carrying a fellow soldier. Having the ability to efficiently test against PPE material effectiveness under these conditions will aid in making informed decisions regarding warfighter safety.

The current effort is to complete the design and build of an electromagnet plate that can be placed into the existing LVAP test chamber. A plate would sit on each shelf that houses an LVAP test cell. The LVAP cells would sit on top of the plates, as they normally would on shelves. The plate would not be activated for normal LVAP testing that uses traditional LVAP parameters, but it would be adjustable for those times when a customer requests a higher contact pressure. Through the adjustment of an electric current through the plate, the operator will have the ability to increase the pressure that the 1 lb weight creates against the sample. With use of a single large plate per shelf, uniform pressure will be applied among all samples during a single test. The plate will be characterized and programmed so that operators can quickly adjust to meet customer requirements. The I-Scan test pressure measurement system will be used to accurately map the pressures generated during the characterization of the plate. When the plate is used for testing, a contact pressure film will be used as a referee to ensure that the appropriate contact pressure was achieved during the test.

## 8.2 System Expansion

The current MPPT device is designed with a 1 in. contact area, as described in the LVAP test method requirements. This system design can be expanded to meet other testing needs. The system could be expanded to accommodate a 2 in. area that would better translate to current agent contact transfer hazard method requirements. The mechanism of the system would remain unchanged as the total contact area expands. This would create a very robust and adaptable device.

## 8.3 Real-Time MPPT

Research is underway to incorporate real-time sensors onto the end of each individual pin in the MPPT device. This would allow for the instantaneous transfer of information regarding agent contact from a surface, including real-time measurement for contact transfer hazard testing. The addition of a real-time MPPT would enable the generation of dose-response curves to support modeling and simulation. Extensive research has already begun in generating dosimetric sensors for low-volatility agents. This research may be leveraged and adapted to the current MPPT device, which would allow for a smoother transition.

## LITERATURE CITED


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## ACRONYMS AND ABBREVIATIONS

| | |
|---|---|
| CAD | computer-aided design |
| CWA | chemical warfare agent |
| DVB | divinyl benzene pad |
| IAW | in accordance with |
| LC–MS/MS | liquid chromatography–triple-quadrupole mass spectrometry |
| LVAP | low-volatility agent permeation |
| MPPT | Multi-Point Pin Technology |
| PPE | personal protective equipment |
| RSD | relative standard deviation |
| TECMIPT | Test and Evaluation Capabilities and Methodologies Integrated Process Team |
| TTOP | Test and Evaluation Capabilities and Methodologies Integrated Process Team (TECMIPT) Test Operating Procedure |
| VX | *O*-ethyl-*S*-(2-diisopropylaminoethyl) methyl phosphonothiolate |

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