Large Scale Tests of Vaporous Hydrogen Peroxide (VHP®) for Chemical and Biological Weapons Decontamination

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<th>a. REPORT</th>
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Vaporous Hydrogen Peroxide (VHP) has been used for more than a decade to sterilize clean rooms and pharmaceutical processing equipment and, more recently, to decontaminate anthrax-contaminated buildings. Recent studies at ECBC have shown that the addition of low-levels of ammonia gas renders VHP reactive towards GD, converting it to pinacolyl methylphosphonic acid. Thus, with suitable activation via ammonia gas, modified vaporous hydrogen peroxide (mVHP) affords the broad-spectrum decontamination of the chemical weapon (CW) agents VX, GD, and HD.* Studies at ECBC have also shown that mVHP is effective in decontamination of biological weapon (BW) surrogates, though mVHP does not at present appear to be substantially superior to standard VHP for biological decontamination. Potential applications of mVHP for military and civilian decontamination include: buildings, vehicle and aircraft interiors, and sensitive equipment.

Recent large scale tests of mVHP applicability for BW and CW decontamination were carried out in one of the ECBC Engineering Chambers, Building E3726. The study evaluated the use of mVHP to decontaminate BW agent surrogates and live CW agents on representative aircraft interior materials. These studies showed that at treatment levels of 250 ppm VHP and treatment times of 24 hours, residual contact hazard from chemical agents was reduced to the ORD required levels on all test materials, with the exception of decontamination of VX run at relatively low ammonia concentrations. These studies also showed that at treatment levels of 250 ppm VHP and treatment times of 24 hours, off-gas hazard from chemical agents was reduced to the ORD required levels for all test materials, with the exception of VX run at relatively low ammonia concentrations. The effect of ammonia concentration on BW and CW decontamination efficacy is currently under study.

*U.S. and international patents pending.
Images of mVHP® Generator and D-Box Used for Biological Decontamination Studies
ECBC Large Chamber Tests

Steris M1000 VHP® Generators

1000 ft³ Exposure Chamber
Large Chamber Test Design

- Agents: VX, GD, HD
- Surrogates: DEPPT, TEP, CEPS, MPS
- 2-4 µL agent deposited on test substrate coupon, allowed to age for one hour
- ca. 1000 ft³ size chamber constructed inside larger chamber (E3726)
- Flow rate ~60 CFM
- Ambient Temperatures
  - VHP® generated using 35 % H₂O₂ and two commercial M-1000 (Steris) VHP® Biodecontamination systems
  - H₂O₂ injection rate ~75 g/min, to maintain ~ 275 ppm [H₂O₂] (measured)
  - NH₃ gas introduced into VHP® stream just prior to its entering the chamber, to maintain ~ 20 ppm [NH₃] (calculated)
- Expose sample for 3, 8, or 24 hours
- Extract substrates using ethyl acetate
- Analyze extract by GC-MS (residual agent/products) (Detection limit ~1 ng)
Large Chamber Test
Substrates

- Stainless Steel (blank)
- Glass (control)
- Chemical Agent Resistant Coating (CARC) painted steel (MIL-P-53039A)
- Aluminum 2024 (Spec T3) (uncoated)
- Air Force Topcoat-coated aluminum (MIL-P-85285-PU)
- Butyl rubber-covered cloth (Boeing insulation cover)
- Kapton (Polyimide) (wiring insulation)
- Nylon Webbing (MIL-W-4088, T3, C3)
- Concrete (Structural)
Large Chamber Test Methods

• Contact Hazard = Natural Latex Rubber dental dam is placed in contact with contaminated substrate, held in contact by 1 kg weight for 15 minutes, then extracted with ethyl acetate and analyzed by GC-MS for amount of agent/surrogate transferred

• Residual Hazard = Contaminated substrate, after contact hazard test, is extracted with ethyl acetate and analyzed by GC-MS for amount of agent/surrogate remaining

• Headspace = Air sample taken directly over contaminated substrate plate and collected on solid sorbent tube, analyzed by GC-MS for mass of agent/surrogate captured
HD Background Data

- **Joint Portable Decon System ORD Requirements:**
  - HD Contact/Residual Hazard: Required = 3.0 mg/m²
  - HD Contact/Residual Hazard: Desired = 0.0 mg/m²
  - HD Headspace Hazard: Required = 2.3 x 10⁻² mg/m³
  - HD Headspace Hazard: Desired = 3.0 x 10⁻³ mg/m³

- **Minimum Detectable Limits for Tests:**
  - Contact/Residual Hazard: 4.93 x 10⁻⁵ mg/m²
  - Headspace Hazard: 8.33 x 10⁻⁵ mg/m³

- **Concentration x Time Values (ppm-hr)**

<table>
<thead>
<tr>
<th></th>
<th>24-hour</th>
<th>8-hour</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>VHP</td>
<td>NH₃</td>
</tr>
<tr>
<td>1HD:</td>
<td>956</td>
<td>177</td>
</tr>
<tr>
<td>2HD:</td>
<td>4657</td>
<td>337</td>
</tr>
<tr>
<td>3HD:</td>
<td>6716</td>
<td>472</td>
</tr>
<tr>
<td>4HD:</td>
<td>6484</td>
<td>721</td>
</tr>
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</table>
Contact Hazard Decontamination of HD by mVHP
24 Hour Exposure

- HD Recovered (mg/m²)
- ORD Req'd
- MDL

Test Run: 1HD, 2HD, 3HD, 4HD
Headspace Hazard Fumigation Data

Headspace Hazard Decontamination of HD by mVHP
24 Hour Exposure

HD Recovered (mg/m3)

Test Run

1HD  2HD  3HD  4HD
Agent Fate Baseline Data Shows ~0.7 mg/m³ HD in headspace after 24 hours weathering; HD in headspace after 24 hr mVHP treatment 3 orders of magnitude lower, ~0.0005 mg/m³

Lab/Wind Tunnel Data

5cm Tunnel Run#3, HD on Aluminum at near 30C

TGA #1 - HD on Al at 30C
Large Chamber Test Results – HD Reaction Products

VHP Decon of HD on Glass

- HD
- HDO
- HDO2

Hours of VHP Exposure

Micrograms recovered
• **Joint Portable Decon System ORD Requirements:**
  - GD Contact/Residual Hazard: Required = 1.7 mg/m²
  - GD Contact/Residual Hazard: Desired = 0.0 mg/m²
  - GD Headspace Hazard: Required = 1.3 x 10⁻³ mg/m³
  - GD Headspace Hazard: Desired = 2.0 x 10⁻⁴ mg/m³

• **Minimum Detectable Limits for Tests:**
  - Contact/Residual Hazard: 4.93 x 10⁻⁵ mg/m²
  - Headspace Hazard: 8.33 x 10⁻⁵ mg/m³

• **Concentration x Time Values (ppm-hr):**

<table>
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<tr>
<th></th>
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<th></th>
<th>8-hour</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>VHP</td>
<td>NH₃</td>
<td>VHP</td>
</tr>
<tr>
<td>1GD:</td>
<td>6549</td>
<td>560</td>
<td>2200</td>
</tr>
<tr>
<td>2GD:</td>
<td>6545</td>
<td>414</td>
<td>2138</td>
</tr>
<tr>
<td>3GD:</td>
<td>6358</td>
<td>379</td>
<td>2117</td>
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</tbody>
</table>
Contact Hazard Fumigation Data

Contact Hazard Decontamination of GD by mVHP
24 Hour Exposure

Graph showing the GD recovered (mg/m²) for different materials over 3 test runs (1GD, 2GD, 3GD).

- **JPDS ORD Contact Hazard Reqm't**: Indicates the threshold for contact hazard requirements.

The graph displays the MDL (minimum detectable limit) and shows the comparison between the recovered GD for each test run and the ORD required levels.
Headspace Hazard Fumigation Data

Headspace Hazard Decontamination of GD by mVHP
24 Hour Exposure

<table>
<thead>
<tr>
<th>Test Run</th>
<th>GD Recovered (mg/m³)</th>
</tr>
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<tbody>
<tr>
<td>1GD</td>
<td>Ord Req'd</td>
</tr>
<tr>
<td>2GD</td>
<td>AEL/TWA</td>
</tr>
<tr>
<td>3GD</td>
<td>MDL</td>
</tr>
</tbody>
</table>

Materials:
- Glass
- Aluminum
- CARC
- Topcoat
- Butyl
- Nylon
- Kapton
- Concrete

JPDS ORD
Headspace Hazard
Req'mt

JPDS ORD
Headspace Hazard
Des'd
Large Chamber Test Results – GD Reaction Products

VHP Decon of GD on Glass

- Micrograms Recovered
- Exposure Time (hrs)

GD
PMPA
**VX Background Data**

- **Joint Portable Decon System ORD Requirements:**
  - VX Contact/Residual Hazard: Required = 4.0 \times 10^{-2} \text{ mg/m}^2
  - VX Contact/Residual Hazard: Desired = 0.0 \text{ mg/m}^2
  - VX Headspace Hazard: Required = 1.7 \times 10^{-4} \text{ mg/m}^3
  - VX Headspace Hazard: Desired = 2.4 \times 10^{-5} \text{ mg/m}^3

- **Minimum Detectable Limits for Tests:**
  - Contact/Residual Hazard: 4.93 \times 10^{-5} \text{ mg/m}^2
  - Headspace Hazard: 3.33 \times 10^{-5} \text{ mg/m}^3

- **Concentration x Time Values (ppm-hr):**

<table>
<thead>
<tr>
<th></th>
<th>24-hour VHP</th>
<th>24-hour NH3</th>
<th>8-hour VHP</th>
<th>8-hour NH3</th>
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<tbody>
<tr>
<td>1VX</td>
<td>6352</td>
<td>488</td>
<td>2113</td>
<td>189</td>
</tr>
<tr>
<td>2VX</td>
<td>6182</td>
<td>320</td>
<td>1824</td>
<td>121</td>
</tr>
<tr>
<td>3VX</td>
<td>(Not Collected)</td>
<td></td>
<td>2038</td>
<td>103</td>
</tr>
<tr>
<td>4VX</td>
<td>6681</td>
<td>141</td>
<td>2109</td>
<td>47</td>
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</tbody>
</table>
Contact Hazard Decontamination of VX by mVHP
24 Hour Exposure

VX Recovered (mg/m²)

1.E+02
1.E+01
1.E+00
1.E-01
1.E-02
1.E-03
1.E-04
1.E-05

VX
1 VX
2 VX
4 VX
Test Run

JPDS ORD Contact Hazard Req'mt
Glass
Aluminum
CARC
USAF Topcoat
Butyl-coated Cloth
Nylon Cloth
Kapton
Concrete

OSTAG/USANCA Guidance
ORD Req'd
MDL
Headspace Hazard Decontamination of VX by mVHP
24 Hour Exposure

VX Recovered (mg/m³)

<table>
<thead>
<tr>
<th>Test Run</th>
<th>1VX</th>
<th>2VX</th>
<th>4VX</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass</td>
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<tr>
<td>Aluminum</td>
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<tr>
<td>CARC</td>
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<tr>
<td>Topcoat</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Butyl</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nylon</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Kapton</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Concrete</td>
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</table>

AEL/TWA
MDL
ORD Req’d

JPDS ORD
Headspace Hazard
Req’n’t
Glass

Aluminum
CARC
Topcoat
Butyl
Nylon
Kapton
Concrete

JPDS ORD
Headspace Hazard
Des’d
Kill Curves for VHP® and mVHP® vs. Avirulent anthrax

H₂O₂ & NH₃ concentration effects on kill rate of *B. anthracis* NNR1d1 Spores on glass coupons

- ~500 ppm H₂O₂, 25 ppm NH₃
- ~500 ppm H₂O₂, 0 ppm NH₃
- ~300 ppm H₂O₂, 25 ppm NH₃
- ~300 ppm H₂O₂, 8.2 ppm NH₃
- ~300 ppm H₂O₂, 0 ppm NH₃
Thorough Decontamination: BW Decontamination by VHP at Room Scale

Chamber mVHP studies using *B. anthracis* NNR1d1
Chambers study is the first demonstration of fumigation-style decontamination of CW agents at the room scale

- 24-hour mVHP® treatment with 250 ppm VHP® + 20 ppm NH₃ effectively reduces contact hazards of HD, GD, and VX to limits of detection
- 24-hour mVHP® treatment with 250 ppm VHP + 20 ppm NH₃ reduces vapor hazards of GD and VX to limits of detection
- 24-hr treatment with 250 ppm mVHP® + 20 ppm NH₃ reduces HD vapor hazard, but detectable amounts remain, especially on porous surfaces

- Porous surfaces may require longer mVHP® treatment times

- mVHP is marginally less effective as an anthrax decontaminant than VHP itself
Building Scale VHP Decontamination

- Internal Sensor Module
- Vaporizer Module
- Distribution Fan
- Filters / NAM
- Air Handling System
- Electrical Generator (All VHP System Components Powered by Generator)
• Demonstrated ability to obtain 125, 250 and 450 ppm treatment levels in 50,000 cu. ft. volume for 5 hour treatment cycle

• Used computational modeling to model air flow in rooms and lay out distribution system

• Confirmed model results with VHP sensor readings during readiness trials

• 100% Kill of G. strearothermophilus BI’s at 125ppm, 250 ppm and 450 ppm VHP treatment levels
mVHP Decontamination trial at Davis-Monthan AFB, November 2004; 100% BI kill in tests to date

C-141 with External Air Handler and NAM
QUESTIONS?