

Assessment of Fumigants for Decontamination of Surfaces Contaminated with Chemical Warfare Agents

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Disclaimer

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Foreword

Following the events of September 11, 2001, EPA's mission was expanded to address critical needs related to homeland security. Presidential Directives identify EPA as the primary federal agency responsible for the country's water supplies and for decontamination following a chemical, biological, and/or radiological (CBR) attack.

As part of this expanded mission, the National Homeland Security Research Center (NHSRC) was established to conduct research and deliver products that improve the capability of the Agency to carry out its homeland security responsibilities. One specific focus area of our research is on decontamination methods and technologies that can be used in the recovery efforts resulting from a CBR contamination event. In recovering from an event and decontaminating the area, it is critical to identify and implement appropriate decontamination technologies. The selection and optimal operation of an appropriate technology depends on many factors including the type of contaminant and associated building materials, temperature, relative humidity, fumigant concentration, fumigation time, and others. This document provides information on how two fumigant-based technologies performed in treatment of CWAs deposited on interior industrial building materials at various operational conditions.

These results, coupled with additional information in separate NHSRC publications (available at www.epa.gov/nhsrc), can be used to determine whether a particular decontamination technology can be effective in a given scenario. With these factors in consideration, the best technology or combination of technologies can be chosen that meets the clean up, cost and time goals for a particular decontamination scenario.

NHSRC has made this publication available to assist the response community to prepare for and recover from disasters involving chemical contamination. This research is intended to move EPA one-step closer to achieving its homeland security goals and its overall mission of protecting human health and the environment while providing sustainable solutions to our environmental problems.

Cynthia Sonich-Mullin, Acting Director
National Homeland Security Research Center

Notice

This report is submitted by CUBRC to Eastern Research Group, Inc. in fulfillment of Task Order 47 of the U.S. EPA STREAMS contract (Contract Number EP-C-05-059). This report covers work completed from January 2008 through June 2009.

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Abstract

The threat of a chemical agent release in a building or transportation hub is driving the U.S. Environmental Protection Agency's (EPA's) National Homeland Security Research Center (NHSRC) Decontamination and Consequence Management Division (DCMD) to conduct a research program that systematically evaluates available decontamination technologies against chemical agents. A program was designed to answer specific questions regarding the effectiveness of two decontamination technologies (steam and vaporous hydrogen peroxide modified with ammonia - mVHP®) against four selected chemical warfare agents (HD, GB, VX and thickened GD) applied to four different indoor building material surfaces (decorative laminate, industrial-grade carpet, galvanized metal, and ceiling tile). The technical objectives were to investigate the effects of environmental conditions (temperature and relative humidity), fumigant concentration, and contact time on decontamination efficacy as well as to determine the agent vapor concentration in the test chamber. A secondary objective was to make a qualitative visual assessment of the compatibility of the building materials with decontaminants: do the building materials decompose, dissolve, corrode, etc., when exposed to the decontaminants? A test chamber with appropriate controls and interfaces was designed and fabricated to accommodate the two decontamination systems under investigation. Known quantities of chemical warfare agent (CWA) were applied to sample coupons (with replicates, blanks, and positive controls) prior to treatment with the appropriate decontamination technology. Samples were removed from the chamber at specified time periods and analyzed for the amount of residual agent remaining on and/or within the sample. Chemical persistence as a function of time (without decontamination) was determined experimentally for HD to establish baseline information on the natural degradation of the CWA on the selected materials under specific operational conditions. Extraction methods were developed and extraction efficiencies were measured for the agent-material combinations.

Results from the efficacy testing indicated that the steam technology for both feed rates (1.5 and 3 kg/hr) removed the CWA surface contamination to below the method detection limit, on all of the building materials tested. The presence of GB, TGD and VX in the condensate, however, indicated that these agents may be re-deposited on other surfaces if the technology were used to fumigate a building or section of a building. Additionally, the steam impacted both the carpet and the ceiling tile materials, most significantly dissolving the ceiling tile.

The mVHP® results appear to indicate that increasing fumigant concentration slightly improved the HD decontamination efficacy for most of the material-exposure time combinations in the test matrix. The best mVHP® decontamination efficacies were observed for the full flow conditions, yielding efficacies of 99% or better for all materials, except for ceiling tile, at the 350 ppmv target concentration conditions.

The most significant findings of the mVHP® study were related to the effect of the mVHP® generator output flow on the decontamination efficacy. Increased output flow (100% versus 10% of the generator output flow) resulted in increased efficacy for fumigant (vaporous hydrogen peroxide and ammonia) concentrations that were the same as or lower than the 10% flow test fumigant concentrations. This effect was seen for all HD and VX-material combinations. For example, at the 10 % flow condition, decontamination efficacies were all less than or equal to 32% for the VX-material combinations at the 400 min exposure time while the efficacies at the full flow condition were 81-89% for these same material-agent combinations and exposure time. Finally, the mVHP® fumigant did not permanently impact the appearance of most of the materials, only causing a white residue to form on the galvanized metal ductwork.

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Abbreviation and Acronym List

AATCC - American Association of Textile Chemists and Colorists
AMC - Army Materiel Command
CA - carpet
CASARM - Certified Analytical Standard Agent Reference Material
CAS RN - Chemical Abstracts Service Registry Number
CCV - continuing calibration verification
COTS – commercial off-the-shelf
CT - ceiling tile
CWA(s) - chemical warfare agent(s)
DAIG - Department of Army Inspector General
DC - diffuser configuration
DCMD - Decontamination and Consequence Management Division
DL - decorative laminate
ECBC - Edgewood Chemical Biological Center
EI - electron ionization
EPA – U.S. Environmental Protection Agency
GB - Sarin
GC – gas chromatograph
GC/MS - gas chromatograph / mass spectrometer
GD - Soman
GM - galvanized metal
IBM(s) – Interior Building Material(s)
HD - sulfur mustard
HVAC - heating, ventilation and air conditioning
ISO - International Organization for Standardization
LB - laboratory blank
LPM - liters per minute
MDL - method detection limit
mVHP® - modified Vaporous Hydrogen Peroxide
NHSRC - National Homeland Security Research Center
PB - procedural blank
PC- positive control
PFTE - polytetrafluoroethylene
PVC - polyvinyl chloride
QAPP - Quality Assurance Project Plan
r - correlation coefficient

RDECOM – U.S. Army Research, Development and Engineering Command

RDTE – Research, Development, Test and Evaluation

RH – relative humidity

SD – standard deviation

TGD - thickened Soman

TIC – Total Ion Current

VHP® – vaporous hydrogen peroxide

Unit List

amu - atomic mass unit

cm – centimeter

ft – foot, feet

g - gram

g/m³ - grams per cubic meter

hr - hour

kg/hr - kilograms per hour

L - liter

L/min - liters per minute

L/hour – liters per hour

LPM - liters per minute

m – meter

mm - millimeter

m³ - cubic meter

min - minute

mL - milliliter

ng - nanogram

ppmv - parts per million by volume

°C - degrees Celsius

µg - microgram

µL - microliter

µm – micrometer

1.0

Introduction

The U.S. Environmental Protection Agency's (EPA's) mission is to protect human health and the environment. Following the terrorist attacks of September 11, 2001, and the subsequent mailing of anthrax-tainted letters, EPA's role with respect to homeland security was expanded. Presidential Directives identified EPA as the primary federal agency responsible for protecting public water supplies and remediation following an attack on indoor or outdoor areas. In recognition of this expanded role, EPA established a homeland security research program. This research program is charged with developing and delivering reliable, responsive expertise and products based on scientific research and evaluations of technology. The imminent threat of a chemical agent release in a building or transportation hub is driving the EPA's National Homeland Security Research Center (NHSRC) Decontamination and Consequence Management Division (DCMD) to develop a research program that systematically evaluates available decontamination technologies against chemical agents on interior surfaces. Exterior surface materials are also of critical importance and should be studied in future research efforts.

1.1 Objectives

This test program was designed to answer specific questions regarding the effectiveness of two decontamination technologies against four selected chemical warfare agents (CWAs); 2-(fluoromethylphosphoryl)oxypropane (GB; CAS RN 77-81-6), O-ethyl S-[2-(diisopropylamino)ethyl] methylphosphonothiolate (VX; CAS RN 50782-69-9), thickened GD 2-(fluoromethyl-phosphoryl)oxy-3,3-dimethylbutane (TGD; CAS RN 96-64-0) and bis(2-chloroethyl) sulfide (HD; CAS RN 505-60-2) deposited onto four different interior building material (IBM) surfaces. The IBMs were decorative laminate, industrial-grade carpet, galvanized metal ductwork, and ceiling tile. The technical objective was to investigate the effects of environmental conditions (temperature and relative humidity), fumigant concentration, and contact time on decontamination efficacy. A secondary objective was to make a qualitative visual assessment of the compatibility of the building materials with decontaminants: do the building materials decompose, dissolve, corrode, etc., when exposed to the decontaminants?

This research program addressed the following specific questions:

- What is the decontamination efficacy of steam for

removal of CWAs on IBMs as compared to controls at ambient environmental conditions?

- What is the decontamination efficacy of modified Vaporous Hydrogen Peroxide(VHP®) (mVHP®), modified in that ammonia is added to VHP®, for removal of CWAs on IBMs under various environmental and operational conditions as compared to controls at ambient environmental conditions?
- What are the physical effects of the decontaminants on the IBMs?

1.2 General Approach

A test chamber with controls and interfaces was designed and fabricated to accommodate the two decontamination systems under investigation. Known quantities of CWAs were applied to sample coupons (with replicates, blanks, and positive controls) prior to subsequent treatment with the appropriate decontamination technology. Samples were removed from the chamber at specified time periods and analyzed for the amount of residual agent remaining on or within the sample in the pores or crevices.

Chemical persistence as a function of time (without decontamination) was determined experimentally to establish baseline information on the natural decay of the CWAs on the selected materials under specific operational conditions. Extraction methods were developed and extraction efficiencies were measured for the agent-material combinations.

1.3 Test Facilities

Testing was performed at the CUBRC Chemical Agent Research, Development, Test and Evaluation (RDT&E) facility located near Buffalo, New York. The facility is certified by the U.S. Army Research, Development and Engineering Command (RDECOM) under a Bailment Agreement to receive, store, handle, and consume chemical warfare agents. The facility is inspected for compliance by the Edgewood Chemical Biological Center (ECBC), the Army Materiel Command (AMC), and the Department of Army Inspector General (DAIG). All chemical agent work performed at this test site falls under CUBRC's International Organization for Standardization (ISO) 9001 quality system.

2.0

Experimental Methods

2.1 Chemical Agents

The chemical agents used to evaluate the efficacy of decontamination were Sarin (GB), thickened Soman (TGD), VX and sulfur mustard (HD). The purity of the chemical agents was greater than 85%. In addition, CASARM-certified agents of higher purity and of a separate lot were used as analytical reference standards. The thickened GD was prepared by adding Acryloid K125 polymer (Rohm and Haas, Philadelphia, PA) to neat GD to achieve a 4.5% weight percent of GD in the total mass of thickened agent. All chemical agents were supplied by the U.S. Army at the request of the EPA.

2.2 Equipment and Instrumentation

2.2.1 Test Chamber

The fumigants under evaluation were passed through a test chamber (Figure 2.2.1), which consisted of a commercial glove box (Cole-Parmer®, Vernon Hills, Illinois) with the physical dimensions of 32"W x 20"H x 24"D. The chamber was modified to allow for temperature control, sampling and ventilation to meet the requirements of the test plan. Modifications to the test chamber included the following:

Heat blankets (SSH-1212-360-120, Omega, Stamford, CT 06907) were installed on the enamel finished side walls, back wall and base of the chamber. They were wired to a six-zone controller (CN616TC1 Six Zone Temp Controller, Omega, Stamford, CT 06907) and independently set, feedback-controlled, and monitored using a temperature controller (CN616, Omega, Stamford, CT 06907). Each blanket was connected to a solid state relay (SSr33DC25, Omega, Stamford, CT 06907) and maintained a set point temperature of $\pm 2^\circ\text{C}$. The temperature controller (CN616, Omega, Stamford, CT 06907) was equipped with an interface (NI USB-232/2 2-port RS232 Serial interface for USB, National Instruments, Austin Texas) allowing data to be logged to a computer.

A ventilation fan (90 CFM, Radio Shack, Springville NY 14141) with a valve (21083 3" gate valve, US Plastics, Lima Ohio) was installed at the top of the chamber to allow ventilation at a rate of up to 1840 LPM. After testing the chamber was vented with air from the laboratory for the steam testing and air from the mVHP® generator (Vaporous Hydrogen Peroxide (VHP®) 1000-ARD Biodecontamination Unit modified for mVHP® Chem/Bio Decontamination, STERIS Corporation, Mentor, OH 44060) for the mVHP® testing. The ventilation configurations used during the mVHP®

testing are discussed in more detail in Section 2.4.6.

The chamber, as received, was configured with a Plexiglas® window (K-34788-00 Economical Glove Box, Cole-Parmer, Vernon Hills, IL 60061). This window was replaced with 0.25" thick plate glass (1/4 " x 26 7/8" x 16" plate glass, Advanced Glass, Williamsville, NY 14221) to reduce agent absorption to the window surface. The same gasket that sealed the Plexiglas® window was used to seal the glass window.

A rack was placed within the chamber to support the test samples. The rack used during the steam testing was made of perforated stainless steel. A polypropylene rack was used for the mVHP® tests.



Figure 2.2-1 - Test Chamber

2.2.2 Steam Fumigant Instrumentation

Steam fumigant was generated using a Reimers® steam boiler (ABA8ZE1Z 8 KW press steam boiler with AR series pump, Reimers® Electra Steam, Incorporated, Clear Brook, VA).

A 0.25" (0.635 cm) stainless steel tube was used to duct steam into the chamber. During dry runs (i.e., no agent), the steam flow had a tendency to form water droplets when injected into a saturated chamber. To mitigate this problem, a diffuser was designed and installed to remove water droplets before they could enter the chamber. A three-inch length of one-inch diameter copper tubing was flattened at one end to create a flared nozzle. The interior of the nozzle was packed with copper wool and a small drain port was built into the nozzle to extract the collected droplets from the copper wool. The design allowed enough steam to enter the chamber to maintain the desired steady-state atmosphere of 600 g/m³ at 100 °C. Excess droplets were drained from the nozzle

through a short section of stock ¼"ID Tygon® tubing to the floor of the chamber interior. A peristaltic pump (Masterflex, Cole-Parmer, Vernon Hills, IL 60061) was used to efficiently remove condensate from the base of the chamber during the experiments.

A steam-condensing column was fabricated and installed on the test chamber to serve two major purposes: 1) to collect water generated by the steam so an output could be measured; and 2) to condense and collect the effluent steam from the chamber exhaust for CWA analysis. The ethylene glycol-jacketed condensing column was plumbed to a refrigerated circulating bath (temperature range of -25°C to 150°C) (NESLAB RTE-10, Thermo Scientific, Waltham, MA) to control its temperature to roughly 15 °C. A chiller-circulator bath (Model 1160 Polyscience Circulating Chiller Bath Recirculating Heater Chilling Bath, VWR, Bridgeport, NJ 08014) was connected to the liquid jacket to circulate chilled ethylene glycol (Prestone® antifreeze, Wal-Mart, Springville NY). This setup allowed the volume of the all the steam produced by the generator to condense, collect, and be measured.

The steam generator (maximum output rated at 1.8 L/min) was calibrated to produce the desired amount of steam for the experiments.

During the steam fumigant tests the test, chamber heat blankets were set at 100 °C to minimize water condensation on the chamber walls.

2.2.3 Modified Vaporous Hydrogen Peroxide (mVHP®) Fumigant Instrumentation

A VHP® 1000-ARD Biodecontamination Unit (STERIS Corporation, Mentor, OH) modified for mVHP® Chem/Bio Decontamination was used to generate the mVHP® fumigant. The unit was modified by STERIS to allow injection of ammonia gas into the stream of VHP®, and also to run as an open loop system. This modification of the 1000-ARD unit affects the system setup, the decontamination cycle and the system controls. Additionally a COTS Munter desiccant dehumidifier (MG90 provided as test equipment by STERIS Corporation, Mentor, OH 44060) for low air volumes was integrated into the system to dehumidify input air to the 1000-ARD unit. STERIS provided CUBRC with a supplemental operating procedure¹ for the modified unit.

The matrix for tests included two fumigant flow rates through the test chamber, one at 100% of the preset mVHP® unit output (340 L/min) and one at 10% (34 L/min) of the mVHP® unit. The 10 % output condition was created by directing 10% of the preset unit output to the test chamber.

2.3 Interior Building Materials

The interior building materials selected for the project were chosen to represent both porous and non-porous materials used in commercial construction. All test coupons were cut to dimensions of 3.5 cm x 1.5 cm from the stock materials supplied by the manufacturer. Specific information for each material is presented below:

Decorative laminate (Formica®, Cincinnati, OH), white matte finish, grade 10, nominal thickness of 1.2 mm); manufactured by Solid Surface Design (Barcelona, Spain); no material preparation prior to testing, (designated as DL).

Industrial grade carpet (style #M7978, color 910; Shaw Industries, Inc., Ringgold, GA) supplied by Carpet Corporation of America (Rome, GA); no material preparation prior to testing (designated as CA).

Galvanized metal duct (standard 24-gauge galvanized steel HVAC duct; Adept Products, Inc., West Jefferson, OH) supplied by Accurate Fabrication, Inc. (Columbus, OH); material cleaned with acetone (99.4% purity by vendor assay Baker Ultra Resi-analyzed, VWR, Bridgeport, NJ 08014) prior to testing (designated as GM).

Ceiling tile (Armstrong® 954; Lancaster, PA), Classic Fine Textured, or equivalent; manufactured by Armstrong; no material preparation prior to testing (designated as CT).

Thickness measurements were made on a randomly selected set of ten samples for each material type. A summary of these data is presented in Table 2.3-1.

Table 2.3-1 - Sample Thickness Measurements

IBM	Thickness, mm	
	mean	standard deviation
Decorative Laminate (DL), n=10	1.19	0.03
Industrial Grade Carpet (CA), n=10	5.82	0.10
Galvanized Metal Duct (GM), n=10	0.58	0.03
Ceiling Tile (CT), n=10	18.64	0.10

2.4 Experimental Design

Tables 2.4-1 and Table 2.4-2 present a summary of the specific experiments performed using the steam and mVHP® fumigants, respectively.

The mVHP® test matrix was compressed; no tests were run with GB or TGD due to the limited availability of the generator.

GB was not tested against steam for the GM and DL samples. Previous studies² have shown GB to be non-

persistent on DL and GM. Studies at CUBRC have confirmed the non-persistence of this agent on these two surfaces as detailed in Section 3.3.

The following are designations and descriptions of the types of samples used to perform the experimental effort.

Test coupons: IBM samples that were contaminated with CWA and exposed to fumigants for specified periods of time.

Table 2.4-1 - Test Matrix for Steam Fumigant Experiments

Agent	Steam output, kg/hr	Materials	Elapsed Exposure Time, minutes	CUBRC Test ID
HD	1.5	GM, DL	120, 180, 240, 400	T-1
		CT, CA	120, 180, 240, 400	T-2
	3	GM, DL	60, 120, 180, 240	T-9
		CT, CA	60, 120, 180, 240	T-10
GB	1.5	CT, CA	60, 120, 180, 240	T-4
	3	CT, CA	60, 120, 180, 240	T-12
VX	1.5	GM, DL	60, 120, 180, 240	T-5
		CT, CA	60, 120, 180, 240	T-6
	3	GM, DL	60, 120, 180, 240	T-13
		CT, CA	60, 120, 180, 240	T-14
TGD	1.5	GM, DL	60, 120, 180, 240	T-7
		CT, CA	60, 120, 180, 240	T-8
	3	GM, DL	60, 120, 180, 240	T-15
		CT, CA	60, 120, 180, 240	T-16

Table 2.4-2 - Test Matrix for mVHP® Fumigant Experiments

Agent	STERIS output	Peroxide target, ppm	Materials	Elapsed Exposure Time, minutes	CUBRC Test ID
HD	10%	250	GM, DL	120, 240, 400, 468	T-18
			CT, CA	120, 240, 400, 510, 600	T-17
		350	GM, DL	120, 240, 400, 468	T-26
			CT, CA	120, 240, 400, 510	T-25
	100 %	250	GM, DL	60, 120, 150, 180	T-19
			CT, CA	60, 120, 180, 400	T-20
		350	GM, DL	60, 120, 150, 180	T-28
			CT, CA	60, 120, 150, 180	T-27
VX	10%	250	GM, DL	120, 180, 240, 400	T-24
			CT, CA	Aborted	T-23
			CT, CA	120, 180, 240, 400	T-23 R
	100 %	250	GM, DL	120, 180, 240, 400	T-21
			CA, CT	120, 180, 240, 400	T-22

Procedural blanks: uncontaminated IBM samples exposed to the fumigant along with the test coupons; used to determine if sample-to-sample cross-contamination occurs during the fumigant testing.

Laboratory blanks: uncontaminated IBM samples not processed with the decontaminants.

Positive controls: IBM samples that in separate trials are contaminated but not exposed to fumigant and allowed to remain at ambient environmental conditions for the periods of time equal to decontamination exposure times.

A single test run consisted of 56 total coupons and eight procedural blanks for each of two material types. For each material type, five test coupons and two procedural blanks were evaluated at four different sampling points. Five laboratory blanks for each material type were processed at the same time separately from these samples. The 56 coupons were divided equally into four separate sample trays as shown in the example in Figure 2.4-3, below.

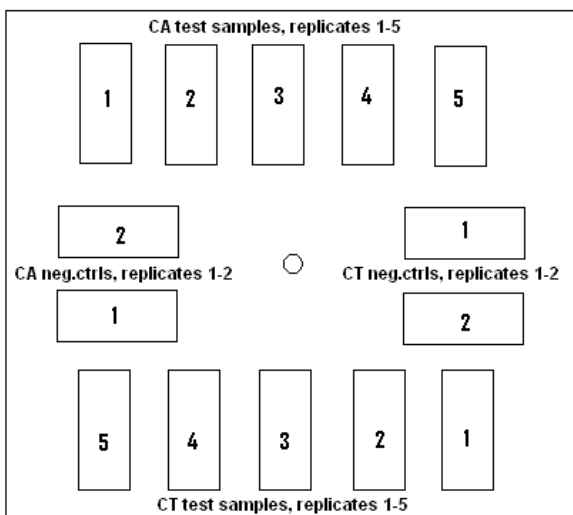


Figure 2.4-3 - Coupon Configuration in Sample Tray

Decontamination efficacy was calculated as:

$$E = (C_0 - C_F) / C_0 \cdot 100\% \quad [1]$$

where C_0 is the average concentration of agent before decontamination (determined from the positive control coupons of each surface material) and C_F is the average concentration on the test coupons after decontamination.

Separate efficacy calculations were performed for each of the material-agent combinations at each exposure time. In addition, since each of these test matrix points was represented by multiple sample coupons, a mean and standard deviation of the efficacy values were reported. The percent efficacy is an indicator of relative efficacy and values stated as >99% do not indicate that the surface is clean. These values simply indicate that more than 99% of the initial contamination is no longer

present in the extracts of the test samples.

2.5 Experimental Procedures

2.5.1 Sample Treatment

The galvanized metal ductwork samples were cleaned with acetone (99.4% purity by vendor assay Baker Ultra Resi-analyzed, VWR, Bridgeport, NJ 08014) prior to testing. All test coupons were allowed to equilibrate at room temperature and relative humidity for a minimum of 30 min before chemical agent was applied.

2.5.2 Chemical Agent Application

Agent droplets were applied to coupons using a gas tight syringe (80201 25µl syringe with a 22 gauge needle for agents HD, VX and GB and a 81020 100µl syringe with a 18 gauge needle for TGD, Hamilton Company, Reno, Nevada) equipped with a repeatable dispenser (Model PB600-1; Hamilton Company, Reno, Nevada). A separate syringe was used for each agent to avoid cross-contamination. A total of 2 µL of agent was applied to each test sample as four 0.5 µL droplets for agents HD, GB, and VX; and as a single 2 µL droplet for agent TGD. This yielded deposition of 2.0 to 2.5 mg of chemical agent on each coupon. Once a full tray of coupons (10 contaminated test coupons and four procedural blanks) was prepared, that tray was immediately placed into the test chamber. The laboratory blanks, which received no application of agent, were placed into a 125 mL tall wide-mouth (05-719-54 4, I-Chem Laboratory Glassware, Fisher Scientific Atlanta, GA) jar containing extraction solvent (EM-HX0296-6 Omnisolv HR- Pesticide Residue Analysis grade hexane, VWR).

2.5.3 Efficacy Experiments – General Method

Before the application of agents to coupons was initiated, the test chamber was pre-conditioned to the desired operational conditions for use of the steam or mVHP® system. The first tray placed into the test chamber corresponded to the longest sampling time point (exposure time). The second tray placed into the test chamber corresponded to the next-to-longest sampling time point and this process was repeated for all of the sample trays.

At each specified sampling time, the appropriate test coupons and corresponding procedural blanks were removed from the test chamber and placed into 125 mL tall wide-mouth glass I-Chem extraction jars (IR121-0125 wide mouth jars, VWR) containing extraction solvent (hexane 99.8% purity by vendor assay). Ten mL of extraction solvent was used for the galvanized metal, decorative laminate, and carpet material coupons and 20 mL was used for the ceiling tile material coupons to effectively cover the material because of the thickness of the coupon.

Once the final tray was removed from the chamber, the decontamination system was turned off and the chamber was ventilated to remove residual decontaminant and/or agent.

2.5.4 Reference Samples (Dose Confirmation)

Using agent application procedures identical to the procedures described in Section 2.5.2 above, four reference dose confirmation samples were prepared (during the agent application process for each test) to provide a normalized contamination level (a 100% value) for use in the decontamination efficacy calculations (Sections 3.7 and 3.11). The appropriate volume of agent was applied to the inner sidewall of a 125 mL glass jar containing 10 mL of solvent. The jar was gently swirled to mix the agent and solvent recovery was calculated based upon the theoretical mass applied. This calculated mass was normalized and used to represent the 100% challenge mass for each experiment. When a test using CT was performed, a second set of dose confirmation samples was prepared using 20 mL of solvent to match the specific extraction conditions used for the ceiling tile coupons.

2.5.5 Ambient Positive Control Experiments

A series of positive control experiments was performed to allow accurate determination of decontamination efficacy for the steam and mVHP® decontaminants. This is necessary because natural attenuation may reduce the amount of agent remaining on the coupons and this could be attributed to decontaminant related reduction. The ambient positive controls were conducted at 24 °C and 40% RH and at the same airflow, 2.35 LPM (0.016 air exchanges/minute), as the persistence test flow rate (Section 2.5.9). This flow rate is similar to the flow rate used in previous EPA testing³. The positive control coupons resided in the test chamber for times parallel to the decontamination test contact times.

Both steam and mVHP® produce environmental conditions (temperature and relative humidity) different from ambient conditions; additionally, steam generates condensation. The decontamination efficacy for both technologies is calculated based upon ambient positive controls. Ambient positive controls were used with all four agents.

2.5.6 GC/MS Method for the Analysis of CWAs in Coupon Extracts and Vapor Samples

The test coupon and the vapor tube extracts were analyzed and quantified for each CWA using GC/MS with electron ionization (EI), under the conditions listed in Table 2.5-1. The GC/MS was operated in the full-scan mode (total ion current, TIC) for a mass range of 50 to 500 daltons. The GC/MS data were acquired and processed using Agilent ChemStation software (see Tables 2.5.1 and 2.5.2) (Agilent Technologies, Santa

Clara, CA). The CWAs were identified by comparison of the retention time and mass spectra against the retention time and mass spectra of calibration standards. Each instrument was calibrated prior to the analysis of samples from each test run using a nine-point calibration curve spanning the range of 0.98 – 291 nanograms (ng). Calibration curves were generated in Microsoft Excel® using a second-order polynomial fit, and correlation coefficients ($r^2 > 0.99$) were calculated from the regression fit. The concentration of the agent was calculated by external standardization using calibration standards analyzed with each data set. Analytical results within the calibration range established for the instrument were reported in ng. Due to the wide calibration range, two separate calibration curves (one with high values and one with low values) were occasionally employed to generate an improved fit to the calibration data. Continuing calibration verification (CCV) standards were inserted into the sample series every ten samples at a minimum to identify any calibration drift. The acceptance criterion for the CCV was $\pm 25\%$ of the initial calibrated response or amount. Maintenance of the instrument was performed in accordance with manufacturer's recommendations. All maintenance was recorded in a dedicated GC/MS Maintenance Log Book for each instrument.

Samples generated during the vapor collection method characterization (Section 2.5.10) were analyzed using a thermal desorption unit (Markes Ultra TD Unity, Markes International, Ltd, Gwaun Elai Medi Science Campus, Llantrisant, RCT, CF72 8XL, UK) interfaced to an Agilent Model 5973/6890 GC/MS (Agilent Technologies, Santa Clara, CA). For this study, vapor calibration standards and test samples were generated by direct injection of liquid calibration standards into stainless steel thermal desorption tubes (0.25 inch (6.35 mm) OD and 3.5 inches (88.9 mm) long) containing 200 mg of sorbent Tenax TA (35/60 mesh - Markes International Limited, Llantrisant, RCT, CF72 8XL, UK). The sorbent tubes were thermally desorbed and analyzed using the instrumental conditions shown in Table 2.5-2 and Table 2.5-3.

**Table 2.5-1 - Description of Gas Chromatograph/
Mass Spectrometer Conditions for the Analysis of
Liquid Extracts**

Parameter	Condition
Instrument	Agilent 5973/5975 Mass Spectrometer Model with electron ionization (EI) ion source, interfaced to a 6890/7980 Gas Chromatograph equipped with a Model 7673A Automatic Sampler and Agilent Enhanced MSD ChemStation Software version D.02.00.275.
Column	30 m x 0.25 mm i.d. Restek RTx-5 MS (cross-linked methyl silicone), fused silica capillary column, 0.5 µm film thickness (Restek No. 12638)
Carrier Gas Flow Rate	1.2 mL/min helium in Constant Flow Mode
Column Temperature (GB)	40 °C initial temperature, hold 1 min, 8 °C/min to 90 °C, hold 0 min, 25 °C/min to 260 °C.
Column Temperature (GD)	50 °C initial temperature, hold 1 min, 10 °C/min to 240 °C, hold 2 min, 20 °C/min to 260 °C.
Column Temperature (HD)	50 °C initial temperature, hold 1 min, 10 °C/min to 150 °C, hold 0 min, 15 °C/min to 240 °C
Column Temperature (VX)	50 °C initial temperature, hold 1 min, 25 °C/min to 240 °C, hold 2 min, 20 °C/min to 280 °C
Injection Volume/Type	1 µL splitless injection (4 mm i.d. double goose neck splitless insert) (20785-214.5, Restek, Bellefonte, PA 16823-8812) with 2 min purge activation time. Split vent flow rate @ 50 mL /min.
Quad Temperature	250 °C
MS Source temperature	230 °C
Solvent Delay	5 min
Data were collected from 50 to 550 daltons at a scan rate of 2.91 scans/sec, threshold of 50 and sampling of 2. Sampling of 2, a GCMS scan parameter, is the number of times the abundance of each mass is recorded before proceeding to the next mass.	

**Table 2.5-2 - Description of Gas Chromatograph/
Mass Spectrometer Conditions for Vapor Samples**

Parameter	Condition
Instrument	Agilent Model 5973 Network Mass Spectrometer equipped with electron ionization (EI) ion source, interfaced to a 6890N Gas Chromatograph and Agilent Enhanced MSD ChemStation Software version E.02.00.
Column	30 m x 0.25 mm i.d. Restek RTx-5 MS (cross-linked methyl silicone), fused silica capillary column, 0.5 µm film thickness (Restek No. 12638)
Carrier Gas Flow Rate	1.2 mL/min helium in Constant Flow Mode
Column Temperature	60 °C initial temperature, hold 1 min, 20 °C/min to 280 °C, hold 1 min
Injection Volume/Type	Direct interface to Markes Unity Thermal Desorption Unit
Quad Temperature	250 °C
MS Source temperature	230 °C
Solvent Delay	5 min
Data were collected from 50 to 550 daltons at a scan rate of 2.91 scans/sec, threshold of 50 and sampling of 2. Sampling of 2, a GCMS scan parameter, is the number of times the abundance of each mass is recorded before proceeding to the next mass.	

Table 2.5-3 - Description of Thermal Desorption Unit Conditions

Parameter	Condition
Instrument	Markes Ultra-Unity automated thermal desorption system interfaced directly to Agilent Model 5973/6890 GC/MS.
Sorbent Tubes	Tenax TA, (35/60 mesh) 200 mg, 3.5 inch (89 mm) x ¼ inch (6.4 mm) or HaySep D (60/80 mesh), 300 mg, 3.5 inch (89 mm) x ¼ inch (6.4 mm) (Markes International Limited, Llantrisant, RCT, CF72 8XL, UK)
Tube Desorption	280 °C for 4 min
Desorption Flow	40 mL/min
Flow Path Temperature	180 °C
Cold Trap	Unity “Chemical Weapons” trap (U-T10CW) (Markes International Limited, Llantrisant, RCT, CF72 8XL, UK)
Trapping Temperature	0 °C
Trap Desorption	300 °C for 4 min
Trap Split Flow	10 mL/min

2.5.7 Chemical Warfare Agent Purity

Certified Analytical Standard Agent Reference Material (CASARM) certified agents were used to prepare analytical standards for the calibration of instruments. The agents used for the decontamination testing were required to have a purity of greater than 85%. These testing agents were analyzed using GC/MS (injection of dilute agent) to verify purity and all agents met or exceeded this criterion.

2.5.8 Extraction Efficiency Determinations

Baseline chemical agent extraction efficiencies were determined using hexane (EM-HX0296-6 Omnisolv HR- Pesticide Residue Analysis grade hexane, VWR) for the four agents (HD, GB, VX and TGD) deposited onto the decorative laminate and galvanized metal ductwork materials. Two µL of the appropriate neat chemical agent was applied using the procedures outlined in Section 2.5.2 onto triplicates of each sample type. To minimize any effects of evaporation, individual coupon samples were placed into 125 mL tall wide-mouth I-Chem extraction jars with PFTE-lined caps containing 10 mL of pesticide-grade hexane within 30 seconds of contamination. The jars were placed into an ultrasonic bath (33995-548 Branson Model 5510-DTH, VWR,

Bridgeport, NJ 08014) for 10 minutes. Once the jar was removed from the ultrasonic bath an aliquot was removed and transferred to a GC autosampler vial for GC/MS analysis. These results were compared to results from a previous EPA effort,² in which samples were placed in hexane and soaked overnight (14 hours) to determine if acceptable extraction efficiencies could be achieved (i.e., 40-120 % recovery with a <30 % coefficient of variance).

Extraction method development and validation was performed to determine optimal procedures for extracting HD, VX, and TGD from porous materials (CT and CA). Four solvents/combinations of solvents were evaluated: 1) hexane; 2) a 1:1 mixture of hexane and acetone; 3) methylene chloride; and 4) ethyl acetate. Hexane and methylene chloride have been used in previous EPA CWA decontamination studies.² These recoveries are listed in Appendix A, Tables A.1-A.4.

2.5.9 Agent Persistence on IBMs

Experiments were performed to generate an agent-substrate persistence model as a function of time to aid in the selection of appropriate sampling points (exposure times) for the fumigant efficacy tests. The first study was conducted using GB on GM and DL. Triplicate coupons of each of the two materials were contaminated with GB, placed into the test chamber, and held at 22 ± 2 °C, 40 ± 10% RH, and with a ventilation rate of one air exchange per hour. Triplicate samples were removed at 5, 15, 30, 60 and 120 minutes, immediately extracted and analyzed by GC/MS.

A separate persistence study was performed using HD on all four building materials under the same experimental exposure conditions. Three samples were removed and extracted at time points of one hour, four hours, one day, two days, and seven days and analyzed by GC/MS. From these studies, specific exposure times were determined and are represented in the test matrices presented in Table 2.4-1 and Table 2.4-2.

2.5.10 Vapor Collection Method Characterization

A brief investigation was conducted using GD, GB and HD to determine the feasibility of collecting vapor samples during mVHP® testing. VX was not evaluated because the Markes Thermal Desorption /Autosampler unit does not lend itself to the direct analysis of VX vapor.

Sorbent tubes were spiked with the respective agent in triplicate at a level of 5 µg. The spiked tubes were then connected to the vapor sampling ports on the test chamber and sampled the decontaminant atmosphere (1.2 CFM of mVHP®) at a flow rate of approximately 250 mL/min for one hour. Calibration tubes were spiked at levels of 0.05, 0.5, and 5 µg as a reference. Following

vapor sampling, the spiked tubes were analyzed by direct thermal desorption to a GC/MS system (Section 2.5.6).

The GD recoveries were less than 20% with one outlier and the GB recoveries were 0, 0 and 2%. HD recoveries at one and four hrs were near 100%.

2.6 Detailed Procedures

2.6.1 Steam

The operational conditions for the steam testing specified in the QAPP⁴ were based upon previous similar decontamination work. In a similar study conducted by Battelle (1995)⁵, high decontamination efficacies were observed when a steam generation rate of 20.8 kg/m³/hr was used for decontamination of stainless steel, concrete and unglazed porcelain coupons contaminated with HD, VX or GB. Using this rate and adjusting the rate proportionally to the volume of the test chamber (5 ft³ = 0.148 m³), a steam generation rate of three kg/hr would be required to duplicate the conditions in the previous work. Based upon this calculation, two test conditions were selected for the steam experiments:

Steam Condition 1 - 3 kg/hr at time points of 60, 120, 180 and 240 minutes

Steam Condition 2 - 1.5 kg/hr at time points of 120, 180, 240 and 400 minute.

The rate of steam generation was controlled by an outlet valve. The steam output was measured as a function of predetermined valve opening (number of turns). A series of tests using varying steam injection rates was performed to establish the outlet valve settings required to meet the desired experimental steam conditions. These experiments demonstrated that after one hour of continuous operation with the outlet valve open one complete turn, a total of 4 L of water were collected. Because the largest flow rate for our series of experiments would only generate 3 L/hr, a second series of tests with valve settings at ¼, ½ and ¾ were conducted to determine which valve settings would yield the desired rates of 3 kg/hr and 1.5 kg/hr. A calibration curve for outlet valve position versus steam injection rate was generated and the appropriate outlet valve positions were selected for the experiments.

During the testing, 1L of steam condensate was collected, beginning at the start of each test for CWA analysis. Most of the agent was assumed to be removed during the first hour of steam decontamination and potentially recovered in the condensate solution.

2.6.2 Steam Tests

Test coupons were pre-conditioned in the test fume hood under ambient conditions prior to application of the chemical agent while the test chamber was heated to 100 °C and pre-conditioned with steam. Once the desired chamber conditions were achieved, the chemical

agent was deposited onto the IBM coupons, one tray at a time, and the tray was placed into the chamber. The decontamination exposure time was measured independently for each tray, and started at the time each tray was placed into the test chamber.

During the steam exposure period, approximately 1L of water condensate from the heated gas exiting the chamber was collected for analysis. Approximately 30 minutes was required to collect this volume of water at a steam generation rate of 3 kg/hr and about an hour was required for the 1.5 kg/hr tests. A 500 mL aliquot of this condensate sample was extracted twice with 10 mL of methylene chloride and analyzed by GC/MS using the same conditions as the steam test samples presented in Table 2.5 1. At the appropriate sampling time periods, the chamber was briefly opened and the test coupons and procedural blanks were removed, extracted in solvent with ultrasonication, and the extract was analyzed by GC/MS.

2.6.3 Modified Vaporous Hydrogen Peroxide (mVHP®)

A STERIS VHP® 1000-ARD was used to generate the mVHP® fumigant during this project. The unit was leased from STERIS Corporation. The 1000-ARD generates the mVHP® fumigant by injecting a liquid solution of hydrogen peroxide (Vaprox®, 35% hydrogen peroxide) (STERIS Corporation, Mentor, OH 44060) onto a heated vaporizer plate resulting in a heated hydrogen peroxide vapor. This gas is then mixed with a low concentration (ppmv) of ammonia. Recent studies at ECBC⁶ have shown that the addition of low levels of ammonia renders VHP® reactive towards GD, converting GD to pinacolyl methylphosphonic acid. The study concluded that mVHP® affords broad-spectrum decontamination of the CWAs including VX, GD and HD.

The STERIS technology employs four phases of activity:

Dehumidification: The 1000-ARD provides dry, heated air that was introduced into the test chamber to achieve a temperature of 30 °C and a relative humidity of less than 40%. A temperature and relative humidity probe, provided by manufacturer of the 1000-ARD, was placed inside the test chamber along with a VHP® sensor and an ammonia sensor. All sensors operated via a feedback loop with the 1000-ARD generator to ensure that all environmental and operational conditions were achieved, held constant, and recorded.

Conditioning: The 1000-ARD generates VHP® and adds ammonia to reach the desired concentration as detected by the sensors. Because the ammonia sensor cannot operate in the presence of hydrogen peroxide vapors, ammonia was introduced first and allowed to stabilize at a fixed percentage of the intended target peroxide

concentration. Once the ammonia concentration was stable, the ammonia sensor was turned off and the addition of vaporous hydrogen peroxide was started.

To avoid contaminating the 1000-ARD generator, the typical practice of vapor regeneration (cycling back through the 1000-ARD) was not performed. Instead, all fumigant vapor was exhausted after it was sent into the test chamber and appropriately filtered. This was not expected to affect the fumigant concentration or composition and in turn the efficacy of the fumigant.

Decontamination: The 1000-ARD system supplied a steady concentration of mVHP® throughout the experiments.

Because the ammonia concentration could not be monitored continuously during the experiments, Dräger indicator tubes (Model Ammonia 5/b, SKC Inc., Eighty Four, PA) were used to measure the ammonia concentration during this phase but prior to inserting the test and procedural blank coupons. A manually operated bellows pump (EW-86514-14 Dräger accuro® Pump Kit and Gas Detection Cole-Parmer, Vernon Hills, IL 60061) drew calibrated 100 mL samples through the Dräger Tubes.

Aeration: Once the test was complete, dry heated air was forced through the test chamber to remove the hydrogen peroxide and ammonia vapors. The chamber effluents were scrubbed using a catalytic converter (supplied by STERIS Corporation, Mentor, OH 44060).

2.6.4 Determination of Fumigant Flow Rate

According to the STERIS web site, the mVHP® ARD Biodecontamination System can provide high-volume biological decontamination for enclosures having a volume up to 10,000 ft³ (~280 m³) depending on the application. The technology has been tested in several test programs under various test configurations. A 2006 test program at ECBC utilized a STERIS mVHP® prototype system with an output of 40 CFM (~1100 L/min) VHP® into a 112 ft³ (3.17 m³) test chamber⁷ resulting in 2.8 air exchanges per minute.

STERIS training personnel indicated that the minimum reproducible volumetric output of the 1000-ARD was 340 L/min. Therefore, in cooperation with EPA, two test conditions were established for the 0.148 m³ (5.23 ft³) chamber used for this program. The first condition was at a full flow rate of 340 L/min and the second was at 10% of full flow, or 34 L/min. The 340 L/min flow rate equates to an air-exchange rate of 2.4 exchanges per minute, considerably higher than what is operationally achievable in the field and close to the values achieved during the previous programs referenced above. The 10% flow rate (34 L/min) equates to 0.24 air exchanges per minute and is more representative of real world facility decontamination applications.

To achieve the 10% output of 34 L/min, the 1000-ARD generator effluent was sent through a rotameter (FL 1653 3.17 SCFM, Omega, Stamford, CT 06907) allowing 90% of the flow to be diverted to vent. In order to accommodate the lower flow rate, dilute solutions of the hydrogen peroxide were prepared ranging from 2% to 5% by volume. Many dry runs were performed to test the system at the lower flow rates to ensure the system could generate the target peroxide concentration (250 ppmv ± 10%) under these conditions. Variables that were investigated and optimized included the hydrogen peroxide injection rate and concentration. During the dry runs the temperature within the chamber was not the same for the 340 and 34 L/min conditions. The heat output from the 1000-ARD at the 340 CFM flow rate increased the chamber temperature to almost 40 °C, while the chamber temperature at the lower flow rate remained around 24 °C. The sensors were biased high at the 340 CFM flow rate possibly due to the higher temperature in the test chamber. All of the results are presented as a function of hydrogen peroxide target concentration and not the actual concentrations. The actual concentrations are reported in Section 4.5.3.

Use of the 340 L/min flow rate caused a pressure increase in the test chamber that was alleviated by installing a STERIS auxiliary blower into the exhaust vent of the chamber and using the auxiliary blower was controlled by the onboard computer. It was run at 12 SCFM instead of the ventilation fan originally installed onto the test chamber. The pressure of one atmosphere at the 100% flow of 340 L/min ventilation rate was monitored with a differential pressure gauge (Series 2000 0-25" Magnehelic®, Dwyer, Michigan City, Indiana, 46361). The auxiliary blower was not needed for the 34 L/min flow rate. The original ventilation fan was, therefore, used for these tests.

Additional positive control sets were run at these two different flow rates for the entire mVHP® test matrix and these tests were designated as follows: PC-5 and PC-10 full flow HD tests, PC-6 and PC-9 reduced flow HD tests, PC-7 full flow HD tests, and PC-8 reduced flow HD tests. The sorbent tube data from these tests can be found in Section 3.1.4 while the coupon extract data from these tests can be found in Appendix D.

2.6.5 mVHP® Diffuser

Turbulence and non-uniformity of fumigant distribution were observed during some early experiments. As a result, it was necessary to build and install a diffuser to modify the manufacturer-provided chamber in order to 1) improve fumigant distribution; and 2) reduce airflow velocity over the samples. Several iterations of design improvements were required to achieve better mixing and fumigant distribution and to significantly reduce the flow velocity directly over the surfaces of the samples.

Following the installation of the diffuser and during one of the positive control tests, uneven evaporation of agent from the test samples was observed. The evaporation pattern indicated that the diffuser was the cause of this problem, so the diffuser design was modified. Table 2.6-1 shows the evolution of the diffuser design and the tests that were performed under each iteration.

The Configuration 1 design consisted of a section of PVC tube extending the entire length of the upper back corner of the glove box with a series of staggered holes drilled along the entire length pointing downward at a 45-degree angle from the back of the glove box. Because many of these holes were located close to the chamber's exit tube, Configuration 2 was implemented. Configuration 2 moved the diffuser to the far end of the fumigant inlet tube and holes were added to the PVC tube. These holes reduced the fumigant velocities and yielded a more evenly distributed the flow pattern. In addition, a capped end piece was added to the terminus of the inlet tube. This configuration generated a better fumigant distribution and also reduced the velocity of the fumigant exiting the diffuser. A third configuration (Configuration 3) was constructed to further improve the distribution. In this configuration, the number of holes along the length of the PVC tubing was reduced and the end was oriented away from the sensor unit. A baffle was also added above the diffuser to isolate the sensor package located above the diffuser and allow the STERIS fumigant input to be more thoroughly mixed with the glove box atmosphere prior to measurement. This configuration further improved the homogeneity of the fumigant distribution.

Additional experiments were performed with Configuration 3 to ensure that the following could be achieved: 1) uniform droplet evaporation; 2) optimal distribution of fumigant within the test chamber; and 3) stable concentration measurements for mVHP® and RH. The results of these experiments led to one final modification, termed Configuration 4. Configuration 4 featured holes in the cap. This final diffuser configuration, with the baffle installed, is shown in Figure 2.6-1. Although perfect uniformity of droplet evaporation was not achieved, the evaporation pattern was greatly improved.



Figure 2.6-1 - Test Final Diffuser Configuration

Table 2.6-1 - Peroxide Diffuser Configurations

Peroxide Diffuser Configuration	Dates	Test(s)
Configuration 1 - open, pointed straight down	5/14 - 5/19	PC-5
Configuration 2 - new design, capped and drilled	5/20 - 5/21	T17
Configuration 3 - reduced holes, different orientation, baffle	5/22 - 5/25	T18 and PC-6
Configuration 4 - holes drilled in cap (final)	5/26 - 6/12	T19, T20, T21, T22, T23, T23R, T24, T25, T26, T27, and T28 PC-7, PC-8, PC-9, and PC-10

3.0 Results and Discussion

3.1 Analytical Method Development Results – Determination of Extraction Efficiency of CWAs from IBMs

Studies were conducted initially to verify the extraction efficiencies of the CWAs from the IBMs that were to be used in the decontamination studies. Hexane with a 10 minute ultrasonication was used to extract HD, GB, VX and TGD from decorative laminate and galvanized metal ductwork. Hexane, 1:1 acetone hexane (v/v), methylene chloride and ethyl acetate were used to extract HD, GB, VX and TGD from ceiling tile and carpeting. These recoveries are listed in Appendix A, Tables A.1-A.4.

Acceptable extraction efficiencies (well within the QAPP criterion of 40-120% with < 30 % coefficient of variance) were achieved for all agents using 10 mL of hexane and 10 minutes of ultrasonication for laminate, galvanized metal ductwork, and carpet. Ceiling tile required 20 mL of hexane to effectively cover the material with solvent in the 125 mL extraction jar. (Appendix A, Tables A.1-A.4). Therefore, hexane was used at these volumes for the majority of the persistence testing, positive controls, and decontamination testing. Better extraction efficiencies were observed using 1:1 acetone hexane (v/v) for VX and TGD deposited on ceiling tile. This solvent system was therefore used for all persistence testing, positive controls, and decontamination testing done with this agent-material combination.

Relatively lower extraction recoveries were obtained for GB, possibly due to loss of the GB prior to extraction. An additional time study was conducted to determine how quickly the GB was lost either due to reaction on the IBM surface and/or evaporation. During the extraction efficiency study, the agent residence time prior to extraction was approximately 30 seconds for all agents. For this study, coupon samples were extracted after residence times of 0, 0.5, 1 and 2 minutes. The mean results (n=3) were 100%, 79.1%, 67.6% and 34.4% recovery, respectively (Appendix A, Table A.5).

3.2 Determination of Method Detection Limits (MDLs)

Method detection limits (MDLs) were determined using the single concentration design estimator recommended by the EPA. The single concentration design estimator is defined as the minimum concentration which can be measured and reported with 99% confidence that the

analyte concentration is greater than zero, determined from the analysis of a sample in a given matrix containing the analyte (40 CFR 136¹, Appendix B). This study was performed by spiking replicate low level matrix samples (7) at identical concentrations 2-5 X the expected MDL. The MDL was calculated by multiplying the sample standard deviation by the correct Student's t-value (3.143).

The resulting method detection limits are shown in Appendix B, Tables B.1 – B.4. Based on these results a detection limit of 5 µg (based on 10 mL extraction) was established for the recovery of CWAs from the DL, GM and CA IBMs, and 15 µg (based on 15 mL extraction) for CT.

3.3 Persistence of GB on Galvanized Metal Ductwork and Decorative Laminate without Decontamination

A study was conducted to determine the persistence of GB on galvanized metal ductwork and decorative laminate using triplicate samples under environmental conditions of a temperature of 22 °C ± 2 °C, air exchange rate of one air change/hour (flow rate ± 10% of flow rate) and RH of 40% ± 10%. The test was conducted in the fumigation chamber and coupon samples were taken and extracted after 5, 15, 30, 60 and 120 min of contact time with the agent. A summary of the results follows in Table 3.3-1.

Table 3.3-1 - Persistence of GB as Indicated by Agent Recovery

IBM	GB, Mean % Recovery ± SD				
	T= 5 min	T = 15 min	T =30 min	T=60 min	T=120 min
Decorative Laminate (n=3)	33 ± 20	0.48 ± 0.51	<0.25	<0.25	<0.25
Galvanized Metal Ductwork (n=3)	52 ± 2.2	4.3 ± 1.0	<0.25	<0.25	<0.25

3.4 Persistence of HD on IBMs without Decontamination

A study was conducted to determine the persistence of HD on all the IBMs using triplicate samples under environmental conditions of a temperature of 22 °C ± 2 °C, air exchange rate of one air change/hour (flow rate ±

10% of flow rate) and RH of 40% ± 10%. The test was conducted in the fumigation chamber and coupon samples were taken and extracted after one hour, four

hours, one day, two days, and seven days of contact time with the agent. A summary of the results follows (Table 3.4-1).

Table 3.4-1 - Persistence of HD as Indicated by Agent Recovery

Building Material	HD, Mean % Recovery ± SD				
	T = 1 Hr	T = 4 Hr	T = 1 day	T = 2 day	T = 7 day
Decorative Laminate (n=3)	97 ± 1	62 ± 2	<0.25	<0.25	<0.25
Galvanized Metal Ductwork (n=3)	97 ± 2	54 ± 6	<0.25	<0.25	<0.25
Carpet (n=3)	98 ± 5	67 ± 6	23 ± 3	14 ± 1	7.3 ± 0.3
Ceiling Tile (n=3)	97 ± 2	42 ± 5	5 ± 1	1.6 ± 2.E-01	2.8 E-01 ± 8 E-02

3.5 Ambient Positive Controls for Determination of Fumigant Efficacy

The positive control tests were run with the test chamber being held under ambient environmental conditions (24 °C, 40% RH, 2.35 LPM flow). Three replicates of each of the IBM and agent combinations were run. The times the coupons resided in the positive control chamber paralleled the times the coupons resided in the fumigation chamber (Table 2.4-1). The ambient

positive control data, shown in Table 3.5-1, illustrate that the agents persisted throughout the anticipated decontamination exposure times at levels above the method detection limits. The G-agents, TGD and GB, were not as persistent as VX and HD, confirming other positive control data generated in previous EPA efforts.³

Table 3.5-1 - Ambient Positive Control Data Summary

Ambient Positive Controls, % Recovery ± Standard Deviation (n=3)						
Agent	IBM	60 min	120 min	180 min	240 min	400 min
HD	GM	84 ± 2	76 ± 3	64 ± 3	51 ± 2.2	24 ± 4
	DL	85 ± 1	70 ± 2	55 ± 3	44 ± 2.6	20 ± 3
	CA	83 ± 3	75 ± 1	54 ± 2	47 ± 0.93	33 ± 3
	CT	75 ± 3	52 ± 3	29 ± 2	20 ± 1.7	7.9 ± 1
GB	CA	2.3 ± 0.6	1.6 ± 0.2	1.8 ± 0.2	1.3 ± 0.27	0.8 ± 0.1
	CT	12 ± 1	10 ± 1	9.9 ± 0.8	7.1 ± 1.8	4.1 ± 0.5
VX	GM	97 ± 2	91 ± 2	91 ± 2	86 ± 5	76 ± 6
	DL	93 ± 7	90 ± 3	82 ± 2	82 ± 2	76 ± 2
	CA	88 ± 8	88 ± 6	75 ± 6	74 ± 2	76 ± 6
	CT	97 ± 4	92 ± 3	85 ± 9	92 ± 3	91 ± 4
TGD	GM	48 ± 7	18 ± 9	3.6 ± 2.8	0.7 ± 0.1	0.6 ± 0.0
	DL	52 ± 3	29 ± 5	8.5 ± 1.9	0.8 ± 0.1	0.4 ± 0.2
	CA	40 ± 7	30 ± 4	14 ± 4	15 ± 4	9.3 ± 2
	CT	83 ± 2	59 ± 3	55 ± 4	54 ± 2	40 ± 2

3.6 Recovery over Time of CWAs on IBMs with Steam Fumigant Technology

The results from the steam decontamination tests to assess the recovery of CWAs applied to IBM samples conducted at both 1.5 and 3 kg/hr are shown in Table 3.6.1.

The IBMs (n=5) with CWAs applied and procedural blanks (PB) (n=2) were removed at four time periods as indicated in the tables below. The percent recovery results are based upon the amount of agent determined in the dose confirmation reference samples analyzed with each test (Section 2.5.4).

HD was not detected in any of the IBM extracts from the samples collected after 120 min for the 1.5 kg/hr steam test and after 60 min for the 3 kg/hr steam test.

GB was not detected in any of the extracts from the IBM samples collected after 60 min from the 1.5 kg/hr steam test. TGD was not detected in any of the extracts from the IBM samples collected after 60 min for the 1.5 or 3kg/hr steam tests, except for the carpeting samples at 60 min for which an average of 0.5% (10 µg) was recovered. A bar graph plot of the µg/sample of TGD recovered for the 1.5 kg/hr test condition is shown in

Appendix C.

VX was detected in all extracts from the IBM samples collected at 60 min, except for the 1.5 kg/hr CT samples. At 120 min, 1-2% VX was present in the GM, DL, and CA extracts for both the 1.5 and 3 kg/hr steam tests. At 180 and 240 minutes, the extracts from the 1.5 kg/hr decontaminated CA samples were the only sample extracts containing detectable VX, recovering 1.0% (20 µg) of the dose reference sample mass. VX was not recovered in any of the extracts from IBM samples collected beyond 180 min for the 3 kg/hr steam test. Statistical analysis of these recoveries could not be completed because all of test coupon recoveries (n=5) were calculated using the masses recovered from the test coupon (n=5) and the agent mass from the single dose confirmation reference sample analyzed (n=1) with each test (Section 2.5.4). Bar graphs of the µg/sample of VX recovered from the IBM coupons can be found in Appendix C.

Extracts of laboratory blanks (LB) (n=5) and PB (n=2) were also analyzed for each test and none of these extracts contained CWAs above the detection limits.

Table 3.6-1 - Recovery of HD on IBM Decontaminated with Steam at 1.5 and 3 kg/hr

Sample Description	Steam Rate kg/hr	HD, Mean Recovery, %			
		120 min	180 min	240 min	400 min
GM (n=5)	1.5	<0.25	<0.25	<0.25	<0.25
DL (n=5)	1.5	<0.25	<0.25	<0.25	<0.25
CA (n=5)	1.5	<0.25	<0.25	<0.25	<0.25
CT (n=5)	1.5	<0.75	<0.75	<0.75	<0.75
PB GM (n=2)	1.5	<0.25	<0.25	<0.25	<0.25
PB DL (n=2)	1.5	<0.25	<0.25	<0.25	<0.25
PB CA (n=2)	1.5	<0.25	<0.25	<0.25	<0.25
PB CT (n=2)	1.5	<0.75	<0.75	<0.75	<0.75
		60 min	120 min	180 min	240 min
GM (n=5)	3	<0.25	<0.25	<0.25	<0.25
DL (n=5)	3	<0.25	<0.25	<0.25	<0.25
CA (n=5)	3	<0.25	<0.25	<0.25	<0.25
CT (n=5)	3	<0.75	<0.75	<0.75	<0.75
PB GM (n=2)	3	<0.25	<0.25	<0.25	<0.25
PB DL (n=2)	3	<0.25	<0.25	<0.25	<0.25
PB CA (n=2)	3	<0.25	<0.25	<0.25	<0.25
PB CT (n=2)	3	<0.75	<0.75	<0.75	<0.75

Table 3.6-2 - Recovery of GB on IBM Decontaminated with Steam at 1.5 and 3 kg/hr

Sample Description	Steam Rate kg/hr	GB, Mean Recovery, %			
		60 min	120 min	180 min	240 min
CA (n=5)	1.5	<0.25	<0.25	<0.25	<0.25
CT (n=5)	1.5	<0.75	<0.75	<0.75	<0.75
PB CA (n=2)	1.5	<0.25	<0.25	<0.25	<0.25
PB CT (n=2)	1.5	<0.75	<0.75	<0.75	<0.75
		60 min	120 min	180 min	240 min
CA (n=5)	3	<0.25	<0.25	<0.25	<0.25
CT (n=5)	3	<0.75	<0.75	<0.75	<0.75
PB CA (n=2)	3	<0.25	<0.25	<0.25	<0.25
PB CT (n=2)	3	<0.75	<0.75	<0.75	<0.75

Table 3.6-3 - Recovery of VX on IBM Decontaminated with Steam at 1.5 and 3 kg/hr

IBM		Steam Rate, Kg/hr	VX Mean Recovery, % ± Standard Deviation			
			60 min	120 min	180 min	240 min
GM	(n=5)	1.5	25 ± 9	1 ± 2 E-01	<0.25	<0.25
DL	(n=5)	1.5	3 ± 1	1 ± 1 E-01	<0.25	<0.25
CA	(n=5)	1.5	5.8 ± 1.4	2 ± 2 E-01	2 ± 8 E-02	7.0 E-01 ± 6 E-02
CT	(n=5)	1.5	<0.75	<0.75	<0.75	<0.75
PB GM	(n=2)	1.5	<0.25	<0.25	<0.25	<0.25
PB DL	(n=2)	1.5	<0.25	<0.25	<0.25	<0.25
PB CA	(n=2)	1.5	<0.25	<0.25	<0.25	<0.25
PB CT	(n=2)	1.5	<0.75	<0.75	<0.75	<0.75
			60 min	120 min	180 min	240 min
GM	(n=5)	3	12 ± 1	<0.25	<0.25	<0.25
DL	(n=5)	3	5.7 ± 0.7	<0.25	<0.25	<0.25
CA	(n=5)	3	6.0 ± 1.9	1.2 ± 2 E-01	8 E-01 ± 9 E-02	<0.25
CT	(n=5)	3	<0.25	<0.25	<0.75	<0.75
PB GM	(n=2)	3	<0.25	<0.25	<0.25	<0.25
PB DL	(n=2)	3	<0.25	<0.25	<0.25	<0.25
PB CA	(n=2)	3	<0.25	<0.25	<0.25	<0.25
PB CT	(n=2)	3	<0.75	<0.75	<0.75	<0.75

Table 3.6-4 - Recovery of TGD on IBM Decontaminated with Steam at 1.5 and 3 kg/hr

Sample Description	Steam Rate kg/hr	TGD, Mean Recovery, % +/- SD			
		60 min	120 min	180 min	240 min
GM (n=5)	1.5	<0.25	<0.25	<0.25	<0.25
DL (n=5)	1.5	<0.25	<0.25	<0.25	<0.25
CA (n=5)	1.5	<0.25	<0.25	<0.25	<0.25
CT (n=5)	1.5	5 E-01± 2 E-01	<0.75	<0.75	<0.75
PB GM (n=2)	1.5	<0.25	<0.25	<0.25	<0.25
PB DL (n=2)	1.5	<0.25	<0.25	<0.25	<0.25
PB CA (n=2)	1.5	<0.25	<0.25	<0.25	<0.25
PB CT (n=2)	1.5	<0.75	<0.75	<0.75	<0.75
		60 min	120 min	180 min	240 min
GM (n=5)	3	<0.25	<0.25	<0.25	<0.25
DL (n=5)	3	<0.25	<0.25	<0.25	<0.25
CA (n=5)	3	<0.25	<0.25	<0.25	<0.25
CT (n=5)	3	<0.75	<0.75	<0.75	<0.75
PB GM (n=2)	3	<0.25	<0.25	<0.25	<0.25
PB DL (n=2)	3	<0.25	<0.25	<0.25	<0.25
PB CA (n=2)	3	<0.25	<0.25	<0.25	<0.25
PB CT (n=2)	3	<0.75	<0.75	<0.75	<0.75

3.7 Steam Efficacy Results

The computed efficacies for steam decontamination are shown in Tables 3.7.1, 3.7.2, 3.7.3 and 3.7.4. The decontamination efficacy for steam decontamination of HD was greater than 99 % for all materials for all exposure periods and the decontamination efficacy for steam decontamination of GB on carpet and ceiling tile coupons was greater than 99% for all exposure periods. The decontamination efficacy for steam decontamination of TGD was equal to or greater than 99% for all materials and exposure periods.

The decontamination efficacy for steam decontamination of VX on ceiling tile was greater than 99% for all exposure periods. For CT, DL and GM samples, the minimum decontamination efficacy for steam decontamination of VX was 82% at the 1.5 kg/hr steam feed rate and 88% at the 3 kg/hr steam rate. Efficacy increased over time and reached or exceeded 99% after the first exposure period.

Table 3.7-1 - HD Steam Decontamination (Decon) Efficacy

IBM	Min	Ambient Positive Controls (n=3), % Recovery ± SD	HD 1.5 kg/hr		HD 3 kg/hr	
			Samples, HD % Recovery ± SD (n=5)	Decon Efficacy	Samples, HD % Recovery ± SD (n=5)	Decon Efficacy
GM	60	84 ± 2	-	-	<0.25	>99%
	120	76 ± 3	<0.25	>99%	<0.25	>99%
	180	64 ± 4	<0.25	>99%	<0.25	>99%
	240	51 ± 2	<0.25	>99%	<0.25	>99%
	400	24 ± 4	<0.25	>99%	-	-
DL	60	85 ± 1	-	-	<0.25	>99%
	120	70 ± 2	<0.25	>99%	<0.25	>99%
	180	55 ± 3	<0.25	>99%	<0.25	>99%
	240	44 ± 3	<0.25	>99%	<0.25	>99%
	400	20 ± 3	<0.25	>99%	-	-
CA	60	83 ± 3	-	-	<0.25	>99%
	120	75 ± 1	<0.25	>99%	<0.25	>99%
	180	54 ± 3	<0.25	>99%	<0.25	>99%
	240	47 ± 1	<0.25	>99%	<0.25	>99%
	400	33 ± 3	<0.25	>99%	-	-
CT	60	75 ± 3	-	-	<0.75	>99%
	120	52 ± 3	<0.75	>99%	<0.75	>99%
	180	29 ± 2	<0.75	>99%	<0.75	>99%
	240	20 ± 2	<0.75	>99%	<0.75	>99%
	400	7.9 ± 0.6	<0.75	>99%	-	-

- indicates that an efficacy test was not completed for this material-exposure time combination

Table 3.7-2 - GB Steam Decontamination (Decon) Efficacy

IBM	Min	Ambient Positive Controls (n=3), % Recovery ± SD	GB 1.5 kg/hr		GB 3 kg/hr	
			Samples, GB % Recovery ± SD (n=5)	Decon Efficacy	Samples, GB % Recovery ± SD (n=5)	Decon Efficacy
CA	60	2 ± 6 E-01	<0.25	>99%	<0.25	>99%
	120	2 ± 2 E-01	<0.25	>99%	<0.25	>99%
	180	2 ± 2 E-01	<0.25	>99%	<0.25	>99%
	240	1 ± 3 E-01	<0.25	>99%	<0.25	>99%
CT	60	12 ± 1	<0.75	>99%	<0.75	>99%
	120	10 ± 1	<0.75	>99%	<0.75	>99%
	180	9.9 ± 0.8	<0.75	>99%	<0.75	>99%
	240	7.1 ± 1.8	<0.75	>99%	<0.75	>99%

Table 3.7-3 - VX Steam Decontamination (Decon) Efficacy

IBM	Mins	Ambient Positive Controls (n=3)	VX 1.5 Kg/hr			VX 3 Kg/hr		
			Samples, VX % Recovery			Samples, VX % Recovery		
			± SD (n=5)		Decon Efficacy	± SD (n=5)		Decon Efficacy
GM	60	97 ± 2	25 ± 9		74%	12 ± 1		88%
	120	91 ± 2	1 ± 2.E-01		99%	<0.25		>99%
	180	91 ± 2	<0.25		>99%	<0.25		>99%
	240	86 ± 5	<0.25		>99%	<0.25		>99%
DL	60	93 ± 7	3 ± 1		97%	6 ± 7.E-01		94%
	120	90 ± 3	9.E-01 ± 1.E-01		99%	<0.25		>99%
	180	82 ± 2	<0.25		>99%	<0.25		>99%
	240	82 ± 2	<0.25		>99%	<0.25		>99%
CA	60	88 ± 8	6 ± 1		93%	6 ± 2		93%
	120	88 ± 6	2 ± 2.E-01		98%	1 ± 2.E-01		99%
	180	75 ± 6	2 ± 8.E-02		98%	8.E-01 ± 9.E-02		>99%
	240	74 ± 2	7.E-01 ± 6.E-02		99%	<0.25		>99%
CT	60	97 ± 4	<0.75		>99%	<0.75		>99%
	120	92 ± 3	<0.75		>99%	<0.75		>99%
	180	85 ± 9	<0.75		>99%	<0.75		>99%
	240	92 ± 3	<0.75		>99%	<0.75		>99%

Table 3.7-4 - TGD Steam Decontamination (Decon) Efficacy

IBM	Min	Ambient Positive Controls (n=3), % Recovery ± SD	TGD 1.5 kg/hr			TGD 3 kg/hr	
			Samples, TGD % Recovery			Samples, TGD % Recovery	
			± SD (n=5)		Decon Efficacy	± SD (n=5)	Decon Efficacy
GM	60	48 ± 7	<0.25		>99%	<0.25	>99%
	120	18 ± 9	<0.25		>99%	<0.25	>99%
	180	4 ± 3	<0.25		>99%	<0.25	>99%
	240	7 E-01 ± 1 E-01	<0.25		>99%	<0.25	>99%
DL	60	52 ± 3	<0.25		>99%	<0.25	>99%
	120	29 ± 5	<0.25		>99%	<0.25	>99%
	180	9 ± 2	<0.25		>99%	<0.25	>99%
	240	8 E-01 ± 1 E-01	<0.25		>99%	<0.25	>99%
CA	60	40 ± 7	5 E-01 ± 2 E-01		87%	<0.25	>99%
	120	30 ± 4	<0.25		>99%	<0.25	>99%
	180	14 ± 4	<0.25		>99%	<0.25	>99%
	240	15 ± 4	<0.25		>99%	<0.25	>99%
CT	60	83 ± 2	<0.25		>99%	<0.75	>99%
	120	59 ± 3	<0.75		>99%	<0.75	>99%
	180	55 ± 4	<0.75		>99%	<0.75	>99%
	240	54 ± 2	<0.75		>99%	<0.75	>99%

3.8 CWAs in Condensate Samples Collected during Steam Decontamination

At the start of the each steam decontamination test, 1L of condensate was collected from the test chamber and analyzed by GC/MS for CWAs. The elevated temperature and humidity within the test chamber were expected to have the combined effects of both evaporation and hydrolysis of the CWAs. HD was not detected (<0.02 µg/mL) in any of the 1L condensate samples collected from either the 1.5 or 3 kg/hr steam tests. VX was detected in the 1L condensate samples collected from both the 1.5 and 3 kg/hr steam tests and the concentration of VX ranged from 0.36 to 1.8 µg/mL. GB was detected at 5.4 and 0.9 µg/mL in the 1L condensate collected from 1.5 and 3 kg/hr steam tests, respectively. TGD (analyzed as GD) was detected in the condensate collected from both the 1.5 and 3 kg/hr steam tests and the concentration of GD ranged from 1.5 to 7.5 µg/mL. Due to the lack of replicate samples statistical analysis of these data could not be completed. Results are presented in Table 3.8-1.

Table 3.8-1 - Results from the GC/MS Analysis of Condensate Samples Collected during Steam Decontamination Tests

Sample Description	Steam Rate, kg/hr	HD, µg/mL
HD Condensate - Test 1	1.5	<0.02
HD Condensate - Test 2	1.5	<0.02
HD Condensate - Test 9	3	<0.02
HD Condensate - Test 10	3	<0.02
		GB, µg/mL
GB Condensate - Test 4	1.5	5.4
GB Condensate - Test 12	3.0	0.9
		VX, µg/mL
VX Condensate - Test 5	1.5	1.8
VX Condensate - Test 6	1.5	0.4
VX Condensate - Test 13	3	0.4
VX Condensate - Test 14	3	0.4
		GD, µg/mL
GD Condensate - Test 7	1.5	1.5
GD Condensate - Test 8	1.5	2.4
GD Condensate - Test 15	3	7.5
GD Condensate - Test 16	3	2.8

3.9 Steam – IBM Compatibility

The coupons exposed to the fumigant for the longest time were visually inspected and digitally photographed

upon removal from the chamber. Unexposed IBM coupons were placed side by side with the coupons exposed to agent/fumigant for comparison. The IBM coupons were inspected for physical changes such as discoloration, crumbling, warping, or blistering.

Ceiling tile and carpeting exhibited the most change. The carpeting samples were saturated with moisture and the edges of the ceiling tiles also appeared to have absorbed moisture. The most significant material effect was that the edges of the tile exhibited some crumbling. Visual inspection did not identify any discernible difference in the appearance of the IBM with varying steam fumigant flow rates (1.5 and 3 kg/hr).

3.10 Recovery over Time of CWAs on IBMs Using mVHP® Fumigant Technology

The first set of tests compared the different output flows of the generator (340 L/min versus 34 L/min) for two target concentrations (250 and 350 ppmv) against recovery of HD. These results are shown in Tables 3.11.1 through 3.11.4. The actual VHP® concentration for each of the tests can be found next to the test number in Tables 3.11.1 through 3.11.4. For the 250 ppmv tests the recoveries were lowest for the tests conducted at the 340 L/min output flow. The 350 ppmv full flow (340 L/min) tests also showed lower recoveries for the test coupons relative to the 34 L/min flow tests. The higher target concentration of mVHP® (350 ppmv versus 250 ppmv) resulted in lower recoveries in both the 34 and 340 L/min tests. The 34 L/min tests at 350 ppmv showed non-detectable levels of agent on almost all materials except CA after 180 minutes.

The presence of HD on the CA and CT procedural blanks indicate that HD was desorbing from the surface of the test coupons and adsorbing to the CA and CT procedural blank coupons in the chamber during the decontamination testing. The highest masses of HD (reflected in the recoveries) were detected at the 34 L/min flow configuration. During these lower flow tests a more stagnant atmosphere existed in the chamber, allowing the HD to adsorb to the porous surfaces (CA, CT) instead of exiting the test chamber. These results indicate that CA and CT serve as sinks for HD in indoor environments and, because these materials are sinks, they could later serve as sources of HD through gas-phase emission of this agent.

The effect of the two different output flows was also studied for VX and the results are shown in Tables 3.11-5 to 3.11-6. The actual VHP® concentration for each of the tests can be found next to the test number in Tables 3.11-5 to 3.11-6. The full flow (340 L/min) test samples had lower recoveries than the one-tenth flow (34 L/min) test samples indicating again that the increased output flow

Table 3.10-1 - mVHP® Decontamination of HD, 10%, 250 ppmv Recoveries

10% output, 250 ppmv		HD Mean Recovery, % ± Standard Deviation					
IBM	Test ID, actual VHP® concentration ppmv	120 min	240 min	400 min	468 min	510 min	600 min
GM (n=5)	18, 246 ppmv	74 ± 6	40 ± 6	1 ± 2	<0.25	-	-
DL (n=5)	18, 246 ppmv	71 ± 1	41 ± 5	2 ± 2	<0.25	-	-
CA (n=5)	17, 210 ppmv	62 ± 5	41 ± 11	29 ± 4	29 ± 6.9	22 ± 3	3
CT (n=5)	17, 210 ppmv	38 ± 5	7.4 ± 1.5	4 ± 1	1 ± 3 E-01	9 E-01 ± 5 E-01	5 E-01
PB GM (n=2)	18, 246 ppmv	<0.25	<0.25	<0.25	<0.25	-	-
PB DL (n=2)	18, 246 ppmv	<0.25	<0.25	<0.25	<0.25	-	-
PB CA (n=2)	17, 210 ppmv	4 ± 4 E-01	6.9 ± 1.1	4 ± 2 E-01	3 ± 7 E-03	6 ± 3	3
PB CT (n=2)	17, 210 ppmv	7.3 ± 0.9	5 ± 1 E-01	3 ± 7 E-02	8 E-01 ± 2 E-01	9 E-01 ± 3 E-01	3 E-01

Note: These tests were run with a diffuser configuration that was different from the final configuration.

- Decontamination tests were not completed for this exposure time-material-agent combination.

Table 3.10-2 - mVHP® Decontamination of HD, 10%, 350 ppmv Recoveries

10% output, 350 ppmv		HD Mean Recovery, % ± Standard Deviation					
IBM	Test ID, actual VHP® concentration ppmv	120 min	240 min	400 min	468 min	510 min	
GM (n=5)	26, 316 ppmv	59 ± 2	7.3 ± 3	<0.25	<0.25	-	-
DL (n=5)	26, 316 ppmv	51 ± 4	3 ± 4	<0.25	<0.25	-	-
CA (n=5)	25, 394 ppmv	60 ± 5	40 ± 6	33 ± 5	-	23 ± 3	3
CT (n=5)	25, 394 ppmv	35 ± 3	9.4 ± 3	1 ± 9 E-01	-	<0.75	
PB GM (n=2)	26, 316 ppmv	<0.25	<0.25	<0.25	<0.25	-	-
PB DL (n=2)	26, 316 ppmv	<0.25	<0.25	<0.25	<0.25	-	-
PB CA (n=2)	25, 394 ppmv	8.8 ± 0.4	13 ± 6	6.6 ± 1.7	-	5.1 ± 1.8	
PB CT (n=2)	25, 394 ppmv	4.8 ± 0.8	5.1 ± 0.8	8 E-01 ± 5 E-01	-	<0.75	

- Decontamination tests were not completed for this exposure time-material-agent combination.

Table 3.10-3 - mVHP® Decontamination of HD, 100%, 250 ppmv Recoveries

100% output, 250 ppmv		HD Mean Recovery, % ± Standard Deviation				
IBM	Test ID, actual VHP® concentration ppmv	60 min	120 min	150 min	180 min	400 min
GM (n=5)	19, 157 ppmv	6.5 ± 4.4	<0.25	<0.25	<0.25	-
DL (n=5)	19, 157 ppmv	2 ± 3	<0.25	<0.25	<0.25	-
CA (n=5)	20, 158 ppmv	15 ± 4	10 ± 2.4	-	8.5 ± 1.6	5.6 ± 1.9
CT (n=5)	20, 158 ppmv	<0.75	<0.75	-	<0.75	<0.75
PB GM (n=2)	19, 157 ppmv	<0.25	<0.25	<0.25	<0.25	-
PB DL (n=2)	19, 157 ppmv	<0.25	<0.25	<0.25	<0.25	-
PB CA (n=2)	20, 158 ppmv	6 E-01 ± 6 E-01	4 E-01 ± 4 E-01	-	<0.25	<0.25
PB CT (n=2)	20, 158 ppmv	<0.75	<0.75	-	<0.75	<0.75

- Decontamination tests were not completed for this exposure time-material-agent combination

Table 3.10-4 - mVHP® Decontamination of HD, 100%, 350 ppmv Recoveries

100% output, 350 ppmv		HD Mean Recovery, % ± Standard Deviation			
IBM	Test ID, actual VHP® concentration ppmv	60 min	120 min	150 min	180 min
GM (n=5)	28, 251 ppmv	17 ± 3	<0.25	<0.25	<0.25
DL (n=5)	28, 251 ppmv	21 ± 6	<0.25	<0.25	<0.25
CA (n=5)	27, 219 ppmv	17 ± 2	8.6 ± 2.7	8.9 ± 3.1	5.3 ± 1.5
CT (n=5)	27, 219 ppmv	<0.75	<0.75	<0.75	<0.75
PB GM (n=2)	28, 251 ppmv	<0.25	<0.25	<0.25	<0.25
PB DL (n=2)	28, 251 ppmv	<0.25	<0.25	<0.25	<0.25
PB CA (n=2)	27, 219 ppmv	<0.25	<0.25	<0.25	<0.25
PB CT (n=2)	27, 219 ppmv	<0.75	<0.75	<0.75	<0.75

Table 3.10-5 - mVHP® Decontamination of VX, 10%, 250 ppmv Recoveries

10% output, 250 ppmv		VX Mean Recovery, % ± Standard Deviation			
IBM	Test ID, actual VHP® concentration ppmv	120 min	180 min	240 min	400 min
GM (n=5)	24, 272 ppmv	97 ± 2	87 ± 4	86 ± 16	62 ± 5
DL (n=5)	24, 272 ppmv	95 ± 1	100 ± 20	91 ± 23	55 ± 13
CA (n=5)	23R, 261 ppmv	96 ± 8	89 ± 7	71 ± 10	51 ± 11
CT (n=5)	23R, 261 ppmv	93 ± 3	87 ± 9	74 ± 11	61 ± 5
PB GM (n=2)	24, 272 ppmv	<0.25	<0.25	<0.25	<0.25
PB DL (n=2)	24, 272 ppmv	<0.25	<0.25	<0.25	<0.25
PB CA (n=2)	23R, 261 ppmv	<0.25	<0.25	<0.25	<0.25
PB CT (n=2)	23R, 261 ppmv	<0.75	<0.75	<0.75	<0.75

Table 3.10-6 - mVHP® Decontamination of VX, 100%, 250 ppmv Recoveries

100% output, 250 ppmv		VX Mean Recovery, % ± Standard Deviation			
IBM	Test ID, actual VHP® concentration ppmv	120 min	180 min	240 min	400 min
GM (n=5)	21, ND	43 ± 8	32 ± 3	19 ± 3	8.1 ± 0.8
DL (n=5)	21, ND	32 ± 3	23 ± 3	18 ± 2	11 ± 1
CA (n=5)	22, 154 ppmv	48 ± 11	32 ± 7	21 ± 5	9.7 ± 2.0
CT (n=5)	22, 154 ppmv	62 ± 3	51 ± 3	29 ± 5	17 ± 1
PB GM (n=2)	21, ND	<0.25	<0.25	<0.25	<0.25
PB DL (n=2)	21, ND	<0.25	<0.25	<0.25	<0.25
PB CA (n=2)	22, 154 ppmv	<0.25	<0.25	<0.25	<0.25
PB CT (n=2)	22, 154 ppmv	<0.75	<0.75	<0.75	<0.75

ND – Actual concentration of VHP™ was not determined for this test.

resulted in less of the agent being recovered from the test samples. Statistical analysis of these recoveries could not be completed because all of test coupon recoveries (n=5) were calculated using the masses recovered from the test coupon (n=5) and the agent mass from the single dose confirmation reference sample analyzed (n=1) with each test (Section 2.5.4).

Extracts of the laboratory blanks did not contain any CWAs above the detection limits.

agent on the coupon surface.

3.11 mVHP® Efficacy Results

Efficacies for application of the mVHP® decontamination technology were determined using the ambient positive controls. The efficacies for the HD tests are shown in Table 3.11-1. This table also displays the actual VHP® concentrations for the HD tests. Most of the efficacies for the 350 ppmv test conditions were higher than those seen for the 250 ppmv test conditions. These results appear to indicate that increasing fumigant concentration slightly improved the HD decontamination efficacy for most of the material-exposure time combinations in these tests. More testing is needed to determine if there is a significant difference in decontamination efficacies for these two different fumigant concentrations.

Exposure times of 180 min, at the 350 ppmv test condition, are sufficient for achieving 99% efficacy or better; however, carpet was the exception with only 90% efficacy being obtained at the 180 min exposure time. Due to compression of the test schedule, further decontamination exposure times could not be studied. Increased contact times at this concentration may result in a higher decontamination efficacy.

The decontamination efficacy for all HD-material combinations was affected by the output flow from the generator. For example, under mVHP® 250 ppmv full flow conditions, the DL was decontaminated to > 99 % after 120 min whereas at the 10% output, an efficacy of only 88% was observed after 400 min. The efficacies for the non-porous surfaces were better than those for the porous surfaces with DL and GM having a greater than 99 % decontamination efficacy even at the reduced flow 350 ppmv conditions after 400 min.

These test results, along with the corresponding environmental data (shown in Appendix D), indicate that two factors significantly affect test results – output flow and temperature. The individual effect of these variables could not be ascertained from this testing. The resulting chamber temperature differed with each of the output flows. The 10% flow test chamber temperature was typically around 24 °C while the chamber temperature was around 40 °C during the full flow tests. Agent evaporation is enhanced by elevated temperatures and increased flow resulting in reduction of the amount of

Table 3.11-1 - mVHP® HD Decontamination (Decon) Efficacy

IBM	Mins	Ambient Positive Controls (n=3)	HD 10% output, 250 ppmv**, actual VHP® concentration GM, DL (246 ppmv) and CT, CA (210 ppmv)		HD 100% output, 250 ppmv, actual VHP® concentration GM, DL (157 ppmv) and CT, CA (158 ppmv)		HD 10% output, 350 ppmv, actual VHP® concentration GM, DL (316 ppmv) and CT, CA (394 ppmv)		HD 100% output, 350 ppmv, actual VHP® concentration GM, DL (215 ppmv) and CT, CA (219 ppmv)	
			Samples, % Recovery ±SD (n=5)	Decon Efficacy	Samples, % Recovery ±SD (n=5)	Decon Efficacy	Samples, % Recovery ±SD (n=5)	Decon Efficacy	Samples, % Recovery ±SD (n=5)	Decon Efficacy
GM	60	84 ± 2	-	ND	6.5 ± 4.4	92%	-	ND	17 ± 3	80%
	120	76 ± 3	74 ± 6	2.3%	<0.25	>99%	59 ± 2	23%	<0.25	>99%
	180	64 ± 3	-	ND	<0.25	>99%	-	ND	<0.25	>99%
	240	51 ± 2	40 ± 6	21%	-	ND	7.3 ± 2.6	86%	-	ND
	400	24 ± 4	1 ± 2	95%	-	ND	<0.25	99%	-	ND
DL	60	85 ± 1	-	ND	2 ± 3	97%	-	ND	21 ± 6	76%
	120	70 ± 2	71 ± 1	#	<0.25	>99%	51 ± 4	28%	<0.25	>99%
	180	55 ± 3	-	ND	<0.25	>99%	-	ND	<0.25	>99%
	240	44 ± 3	41 ± 5	7.4%	-	ND	3 ± 4	92%	-	ND
	400	20 ± 3	2 ± 2	88%	-	ND	<0.25	99%	-	ND
CA	60	83 ± 3	-	ND	15 ± 4	82%	-	ND	17 ± 2	80%
	120	75 ± 1	62 ± 5	17%	10 ± 2	86%	60 ± 5	20%	8.6 ± 2.7	89%
	180	54 ± 2	-	ND	8.5 ± 1.6	84%	-	ND	5.3 ± 1.5	90%
	240	47 ± 1	41 ± 11	12%	-	ND	40 ± 6	15%	-	ND
	400	33 ± 3	29 ± 4	11%	5.6 ± 1.9	83%	33 ± 5	#	-	ND
CT	60	75 ± 3	-	ND	<0.75	>99%	-	ND	<0.75	99%
	120	52 ± 3	38 ± 5	28%	<0.75	99%	35 ± 3	34%	<0.75	99%
	180	29 ± 2	-	ND	<0.75	97%	-	ND	<0.75	97%
	240	20 ± 2	7.4 ± 1.5	63%	-	ND	9.4 ± 3.2	53%	-	ND
	400	7.9 ± 0.6	4 ± 1	55%	<0.75	91%	1.5 ± 9 E-01	81%	-	ND

** These tests were run with a different diffuser configuration.

ND denotes that while positive control test was completed for this agent-material combination at this time point a decontamination test was not completed.

Indicates that the positive control recoveries were less than the decontamination test recoveries.

Actual concentration as determined through titration (see Section 4.5.3).

Efficacies for the VX tests are shown in Table 3.11-2. Actual VHP® concentrations are also shown in the tables. These results indicate that even after 400 min the greatest efficacy observed was 89% for the VX deposited on galvanized metal. Due to compression of the test schedule further decontamination exposure times could not be studied.

The decontamination efficacy for all VX-material combinations was also affected by the output flow/temperature. The decontamination efficacies for the 34 L/min flow were all less than or equal to 32% at 400 min while the efficacies for the full flow condition were 81-89%. More research is needed to determine if increased exposure times and/or increased fumigant concentration will result in improved decontamination efficacies.

Table 3.11-2 - mVHP® VX Decontamination (Decon) Efficacy

IBM	Mins	Ambient Positive Controls (n=3)	VX 10% output, 250 ppmv, actual VHP® concentration GM, DL (272 ppmv) and CT, CA (261 ppmv)		VX 100% output, 250 ppmv, actual VHP® concentration GM, DL (ND) and CT, CA (154 ppmv)	
			Samples, % Recovery ± SD (n=5)		Samples, % Recovery ± SD (n=5)	
				Decon Efficacy		Decon Efficacy
GM	120	91 ± 2	97 ± 2	#	43 ± 8	53%
	180	91 ± 2	87 ± 4	4.7%	32 ± 3	65%
	240	86 ± 5	86 ± 16	#	19 ± 3	77%
	400	76 ± 6	62 ± 5	18%	8.1 ± 0.8	89%
DL	120	90 ± 3	95 ± 1	#	32 ± 3	65%
	180	82 ± 2	100 ± 20	#	23 ± 3	72%
	240	82 ± 2	91 ± 23	#	18 ± 2	79%
	400	76 ± 2	55 ± 13	28%	11 ± 1	85%
CA	120	88 ± 6	96 ± 8	#	48 ± 11	46%
	180	75 ± 6	89 ± 7	#	32 ± 7	57%
	240	74 ± 2	71 ± 10	3.5%	21 ± 5	72%
	400	76 ± 6	51 ± 11	32%	9.7 ± 2.0	87%
CT	120	92 ± 3	93 ± 3	#	62 ± 3	33%
	180	85 ± 9	87 ± 9	#	51 ± 3	41%
	240	92 ± 3	74 ± 11	20%	29 ± 5	69%
	400	91 ± 4	61 ± 5	32%	17 ± 1	81%

indicates that the positive control recoveries were less than the decontamination test recoveries.

ND – Actual concentration of VHP™ was not determined for this test.

3.12 Discussion of Effect of mVHP® Decontamination Diffuser Configuration on Test Results and Additional Positive Controls

During the initial mVHP® tests (PC 5 Test 17 and 18 and PC 6), uneven evaporation of agent drops and positive control test results indicated a location bias within the chamber. The bias was suspected to be a result of the diffuser configuration (design and orientation). A series of studies was conducted and modifications

were made to the diffuser configuration (Section 2.6.5). The effect of the diffuser configuration on the test was characterized by applying distilled water droplets to the surface of a stainless steel pan to simulate the evaporation of agent droplets. Three modifications to the diffuser configuration were evaluated and a final modification was incorporated for the remaining tests.

All mVHP® tests but Test 17 (diffuser Configuration 2) and Test 18 (Configuration 3) were conducted with the final diffuser configuration (Configuration 4). Tests 17

and 18 consisted of HD 250 ppmv challenges at 10% flow. The corresponding tests (at 100 % flow at 250 ppmv - Tests 19 and 20) and (10% flow at 350 ppmv - Tests 25 and 26) have a degree of uncertainty regarding the potential effect of the different diffuser configurations. The absolute effect of the different diffuser configurations is not clear.

As stated above in Section 2.5.6, additional positive control experiments were run with the mVHP® generator operating with DI water instead of hydrogen peroxide solution where either 100% of the generator flow was pushed through the test chamber (Tests 6, 10 for HD and Test 7 for VX) and or 10% of the flow (Tests 5, 9 for HD and Test 8 for VX) was pushed through the test chamber. PC-5 and PC-6 were run with the first diffuser configuration and both of these tests had lower observed average temperatures and relative humidities, as shown in Tables D.1, D.2, D.7, and D.8, than the repeated positive controls (PC-9 and PC-10, respectively, also shown in the same Tables). These lower average temperatures and relative humidities may have been due to uneven distribution of both the conditioning input air as well as the fumigant. A larger percent recovery of HD was observed for most of the positive controls run with the earlier diffuser configuration for the reduced flow tests (PC-5), as seen in Tables D.1, D.2, D.7, and D.8, indicating that evaporative loss was likely occurring during the positive control test run with the final diffuser configurations (PC-9 and PC-10). In addition, the positive control samples run at the full flow condition in the final diffuser configuration (PC-9 and PC-10) experienced higher average temperatures than the corresponding ambient positive control test samples as shown in Tables D-4 and D5. This diffuser configuration was used during the HD decontamination testing and similar average temperatures were observed during this testing. Therefore, the full flow positive controls indicate that some of the reduction in the HD agent mass on the coupons during the full flow tests was likely due to evaporation.

The VX positive control and decontamination tests were all run with the final diffuser configuration and the results from these tests are shown in Tables D-13 through D-18. The positive control samples run at the full flow condition experienced higher average temperatures than the corresponding ambient positive control test samples as shown in Tables D-16 and D17. The VX recoveries on all four building materials for the full flow positive control samples also appear to be lower than the VX recoveries observed for the ambient positive control samples indicating that there were some losses due to evaporation during the full flow positive control tests. The positive controls were run at the same diffuser configuration as the VX decontamination tests and had a similar average temperature during the

decontamination phase, indicating that some evaporative losses may have occurred during the VX mVHP® full flow decontamination tests.

As discussed in Section 4.9, procedural blank coupons (data shown in Tables 3.10-1 through 3.10-4) placed in the chamber along with CWA-contaminated coupons showed that HD was found on the CA and CT coupons for many of the exposure periods. This demonstrates that the chemical CWA vapor cross-contaminated the “clean” samples. Non-porous procedural blank extracts (GM and DL) contained less agent mass than the extracts of the porous materials (CA and CT).

3.13 CWAs in Chamber Vapor Samples Collected during mVHP® Decontamination

During the mVHP® tests, vapor from the test chamber was collected onto sorbent tubes to determine whether vaporized CWA was present in the atmosphere of the chamber during decontamination. Vapor samples were collected onto solid sorbent tubes with a metered flow rate for a known period of time. The sampling covered the initial time period during the decontamination or corresponding positive control test. Samples were collected for a shorter period of time (60 versus 120 min) at the higher air exchange rates (100% versus 10%) in the test chamber. The contents of the sorbent tubes were extracted with ethyl acetate and the resulting extract analyzed using GC/MS.

The initial vapor study (Section 2.4.10) was conducted using thermal desorption of the sorbent tubes prior to GC/MS analysis. During the actual tests, the vapor adsorbent tubes were extracted with 2 mL of ethyl acetate to allow reanalysis or dilution of the samples (it is not possible to reanalyze sorbent tubes following thermal desorption), as needed.

3.13.1 HD Vapor Sample Results

HD was detected in all the vapor samples but the mass collected (0.001 to 0.625 mg) was relatively low in comparison to the total mass of agent applied to the coupons (80 to 100 mg). Vapor concentrations based on the volume of vapor sampled over the periods of time are presented in Table 3.13-1. The vapor sample concentrations taken during the positive control tests (PC-9 and PC-10) were higher than the test vapor sample concentrations; however, a proper statistical analysis of these data, to determine if this difference was due to the absence of the fumigant, could not be completed because the type of IBMs in the test chamber during the positive control and test runs was different. There was a significant difference (t- test of unequal variances, 95% confidence level, $n=3$, $t_{crit} 4.3 > t_{stat} 3.5$) between the vapor samples collected during Tests 27 and 28 (100% flow rate, target 350 ppmv VHP®) indicating a difference in the amount of agent available in the gas phase for the two different groups of samples. The Test 28 samples taken with GM and DL in the test chamber showed higher values of agent than the Test 27 samples taken with CT and CA in the test chamber indicating the agent was evaporating at a slower rate from the porous materials. There were no significant differences between Tests 25 and 26 indicating that the material type did not affect the amount of available agent for this lower flow rate. The two flow rates could not be compared because their sampling times were different.

3.13.2 VX Vapor Sample Results

VX was not detected in any of the vapor samples. The VX may have been present but the actual detection limit for VX was much higher (0.036 mg/m³) than the detection limit for HD because the VX sorbent tubes

required extraction with a solvent creating a significant dilution factor (2 mL = 2000 x dilution factor). Lower vapor concentrations were expected, at least for the positive control vapor samples, because VX has a lower vapor pressure than HD and as observed in the positive controls, VX is more persistent on these IBMs than HD.

3.14 mVHP® - IBM Physical Compatibility

No observable changes were noted for the physical properties of the IBM coupons exposed to mVHP®. The galvanized metal ductwork exhibited a white residue at the agent application sites. There did not appear to be any discernible differences in the appearance of the coupons with different mVHP® fumigant flow rates.

Table 3.13-1 - mVHP® HD Vapor Results

CUBRC Test ID	IBMs	Agent	ppmv H ₂ O ₂	STERIS Output	Sampling period, min	Mean Concentration and Standard Deviation, mg/m ³		Number of replicates
PC-9	GM DL CA CT	HD	0	10%	0-120	13.2 ± 2.3		(n=3)
T-18	GM DL	HD	250	10%	0-300	8.4 ± ND		(n=1)
T-26	GM DL	HD	350	10%	0-120	7.4 ± 0.3		(n=3)
T-25	CA CT	HD	350	10%	0-120	6.8 ± 0.3		(n=3)
PC-10	GM DL CA CT	HD	0	100%	0-60	5.4 ± 0.8		(n=3)
T-19	GM DL	HD	250	100%	0-60	2.4 ± ND		(n=1)
T-20	CA, CT	HD	250	100%	0-60	2.5 ± 0.3		(n=3)
T-28	GM DL	HD	350	100%	0-60	3.6 ± 4.E-03		(n=3)
T-27	CA CT	HD	350	100%	0-60	2.4 ± 0.6		(n=3)

4.0

Quality Assurance

4.1 ISO 9001 Audit

There were four findings during the ISO 9001 audit. All findings were classified as minor and none were found to affect the quality of the data.

4.2 Deviations from the QAPP

The mVHP® test matrix was modified as an additional VHP® test concentration was added (350 ppmv) and GB and TGD were not tested. The mVHP® test flow output

was changed from 425.0 L/min and 42.5 L/min to 340 L/min and 34 L/min.

4.3 Quality Assurance Indicators

Table 4.3-1 contains data quality indicators that were monitored in accordance with the QAPP. No findings required corrective actions.

Table 4.3-1 - Measurements and Data Quality Indicators for Decontamination and Persistence Testing

Parameter	Measurement Method	Data Quality Indicators
Temperature	NIST-traceable thermometer	Compare against calibrated thermometer before and after experiment, agree $\pm 10\%$
Relative humidity	NIST-traceable hygrometer	Compare against calibrated hygrometer before and after experiment, agree $\pm 10\%$
Air exchange rate in chamber	Mass flow controller	Compare to second NIST-traceable calibrated flow meter before and after experiment, agree $\pm 10\%$
Hydrogen peroxide concentration	mVHP® electrochemical sensor	Compare to AATCC Test Method 102-2007 (AATCC, 1957), agree $\pm 10\%$
Ammonia concentration	NH ₃ electrochemical sensor	Run NH ₃ calibration gas, agree $\pm 10\%$
Agent on Positive Control	Extraction/GC	The relative standard deviation of the percent recoveries at each time point should be $\leq \pm 25\%$.
Agent on Laboratory Blank	Extraction/GC	Laboratory blanks should have less than 1% of the amount of analyte compared to that found on positive controls.
Agent on Procedural Blank	Extraction/GC	Procedural blanks should have less than 10% of the amount (recovery corrected) compared to that found on positive controls.

4.4 Temperature and Relative Humidity

During the ambient positive control tests the RH and temperature were measured with an internally chamber-mounted calibrated VWR® hygrometer/temperature probe (35519-020 VWR®, Bridgeport, NJ 08014). During the steam shakedown tests a bare wire thermocouple (CHAL-020 Type K, Omega, Stamford, CT 06907) was inserted into the chamber verifying that the temperature was 100 °C. During the remainder of the testing the temperature was monitored on the exterior of the chamber between the heating blankets and the chamber exterior wall. This temperature was maintained at ≥ 100 °C throughout the testing. The VWR® hydrometer (VWR.com, DH-011 – Cal dates 1/09 – 1/10) was also used to monitor the laboratory

temperature and humidity.

During the mVHP® experiments (decontamination tests and positive controls), the RH and temperature were monitored by the vendor-provided sensors. The STERIS unit was plumbed into the glovebox along with a STERIS remote sensor box containing the temperature, humidity, hydrogen peroxide and ammonia sensors. The first STERIS sensor bundle did not work properly and STERIS had to reconfigure the unit. The STERIS reconfigured unit's sensors had the following calibration dates: 5/07-5/08 and 6/07-6/08. A replacement sensor unit that was within calibration did not arrive in time for incorporation into the testing ($>50\%$ of the test program was complete). Neither the hydrometer nor the STERIS Humidity and Temperature Sensor were compared to a

NIST-calibrated thermometer and hygrometer.

4.5 Air Exchange Rate

4.5.1 Steam Air Exchange

The steam air exchange rate was 0.016 exchanges/min. This air exchange rate was dictated by the feed rate of the steam into the test chamber.

4.5.2 mVHP® Air Exchange

There were two mVHP® air exchange rates; one for the high flow (100% - 340 L/min) = 2.3 air exchanges per min and one for the low flow (10% - 34 L/min) = 0.23 air exchanges per minute. The flow output was controlled by a Venturi Flow Meter (STERIS ARD 1000, STERIS, Mentor OH 44060)with a differential pressure sensor. STERIS reported that the unit was calibrated on July 27, 2008. This flow meter was not compared to a NIST-calibrated flow meter.

The ammonia output was controlled by an AALBORG 0-100 mL/min flow controller supplied by STERIS (calibrated June 26, 2008). This flow meter was not compared to a NIST-calibrated flow meter.

4.5.3 Hydrogen Peroxide Concentrations

The stock hydrogen peroxide solutions used to generate

VHP® were titrated following AATCC Test Method 102-2007⁸ The intention of this QA indicator was to verify the sensor-determined VHP® vapor concentrations by pulling an air sample from the test chamber through an impinger filled with acidified potassium permanganate and then titrating this solution. Because the hydrogen peroxide stock solutions used for generation of the VHP® were titrated instead, these titration results along with the test duration, air flow rate and mean rate of delivery of the titrated solution through the STERIS unit (information obtainable from the data file generated by the STERIS unit) were used to calculate a mean theoretical hydrogen peroxide vapor concentration for the duration of a test. In addition, condensation was not observed in any of the tests and the RH and temperatures achieved in the test chamber also did not indicate that condensation occurred during any of the testing. No losses of the vapor to the condensation phase should therefore be occurring. The calculated concentrations were compared to the concentrations reported by the STERIS unit sensor (used in the feedback loop). In Table 4.5-2 the calculated concentrations and the average sensor concentrations are shown. The sensors were biased high at the higher flow tests, likely due to the increased temperature during these runs.

Table 4.5-2 - VHP™ Concentrations and Ammonia Concentrations for Each of the Test Runs

Test ID	Agent	STERIS output - CFM	Target VHP® - ppmv	Average VHP® sensor reading - ppmv	Average RH - %	Average Temperature - °C	Delta Temperature (from ambient 25 °C) - °C	Average Calculated H ₂ O ₂ concentration	% Error
17	HD	34	250	268	47	28	4	210	28%
18	HD	34	250	259	60	26	2	246	5%
19	HD	340	250	257	36	37	13	157	64%
20	HD	340	250	247	43	37	13	158	56%
21	VX	340	250	254	38	37	13	x	x
22	VX	340	250	250	37	36	12	154	63%
23 R	VX	34	250	264	50	26	2	261	1%
24	VX	34	250	262	55	26	2	272	-4%
25	HD	34	350	353	54	29	5	394	-10%
26	HD	34	350	355	62	30	6	316	12%
27	HD	340	350	347	37	42	18	219	59%
28	HD	340	350	340	41	40	16	215	58%

X – Peroxide solution not titrated.

4.6 Ammonia Concentration

4.6.1 Ammonia Concentration Control

During testing the ammonia sensor was observed to be saturated even at the 16 ppmv concentration. This saturation did not allow the conditioning phase of the fumigant generation to be completed successfully. Software control of the flow controller was therefore overridden and manual control was implemented by connecting the flow controller to a Hastings flow control box (Model 400, Teledyne Hastings Instruments, Hampton, VA 23669) for all remaining tests (Table 4.6-1 – manual control tests are highlighted in yellow).

Two distinct levels of operation were programmed into the unit: 250 and 350 ppmv hydrogen peroxide concentration. The generator-selected ammonia levels were 6% of the target hydrogen peroxide concentration. All of the above parameters were set up and tested at the 340 L/min output flow. The ammonia levels recorded in full flow checkout runs were then used to establish the flow rates programmed into the external flow controller.

The following ammonia flow rates were established for each test type:

100% flow = ammonia flow rate of 5.5 g/min

10% flow = ammonia flow rate 4.1 g/min.

The STERIS unit was pre-programmed to set the ammonia concentration during the ammonia characterization cycle prior to the conditioning cycle which starts the introduction of hydrogen peroxide. The ammonia levels established by the STERIS unit's logic always came in above the 6% target value (typically ~10%). In order to maintain some order of consistency in the testing, the levels targeted for the 350 ppmv runs were adjusted to the actual higher ratios (~10%) defined in the 250 ppmv test and not the software-targeted value of 6 %.

These values closely approximate the average values generated on previous runs with valid “ammonia characterization” phase operation.

4.6.2 Verification of Ammonia Concentration

The results from the Dräger tube measurements, taken during Tests 17, 18 and 19, are shown in Table 4.6-2. The Dräger measurements consistently indicated a lower ammonia concentration (40-50%) than the 1000-ARD sensor. The presence of the VHP® may have affected the Dräger measurement, but from the offline measurements by the Dräger tubes the concentration measured during the experiments was relatively consistent from measurement to measurement and test to test. Dräger sampling was therefore discontinued after Test 19.

Table 4.6-1 - Ammonia Generation Data for the Various Tests

CUBRC Test ID	Date	Agent	Ammonia ppmv	Ammonia g/min	STERIS output
PC-5	05/19/09	HD	na	na	10%
17	05/21/09	HD	28	4.18	10%
18	05/22/09	HD	21	3.27	10%
PC-6	05/25/09	HD	na	na	100%
19	05/26/09	HD	23	4.18	100%
20	05/27/09	HD	43	6.18	100%
PC-7	05/28/09	VX	na	na	100%
21	05/29/09	VX	43	6.2	100%
22	06/01/09	VX	37	5.29	100%
PC-8	06/02/09	VX	na	na	10%
23	06/03/09	VX	ABORTED		10%
23 R	06/04/09	VX	41	5.54	10%
24	06/05/09	VX	33	4.18	10%
25	06/08/09	HD	39	4.63	10%
26	06/09/09	HD	40	4.54	10%
PC-9	06/10/09	HD	na	na	10%
PC-10	06/11/09	HD	na	na	100%
27	06/12/09	HD	41	4.71	100%
28	06/12/09	HD	38	4.41	100%

na = Indicates that these tests are the positive controls. The fumigant was not introduced into the chamber during these tests.

Yellow highlight indicates manual control of NH₃ flow controller.

Table 4.6-2 - Dräger Tube Ammonia Measurements

Test	Source	Range (ppmv)	Batch #	Dräger Reading (ppmv)	STERIS Reading (ppmv)
Test 17	Tube 1	5 - 100	ARZH-0551	5	15
Test 17	Tube 2	5 - 70	ARZL-3361	5	15
Test 18	Tube 1	0 - 70	ARZL-3361	12 - 15	26
Test 18	Tube 2	0 - 100	ARZH-0551	10	22
Test 19	Tube 1	0 - 70	ARZL-3361	7 - 10	17
Test 19	Tube 2	0 - 100	ARZH-0551	<5	12

4.7 Agent on Positive Controls

The data quality indicator for the positive controls is that the relative standard deviation of the triplicate set of positive control percent recoveries at each time point should be ≤ 25%. This criterion was met for most of the triplicate positive control sets except for the GB on carpet and the TGD on galvanized metal, decorative laminate, and carpet. The relative standard deviations appear to be highest for the GB and TGD.

Table 4.8-1 - Relative Standard Deviation for Ambient Positive Controls

Relative Standard Deviation for Ambient Positive Controls						
HD	GM	2.7	3.5	5.4	4.2	17
	DL	1.2	2.9	4.7	5.9	17
	CA	3.1	1.6	4.5	2.0	10
	CT	3.4	6.0	5.9	8.4	7.6
GB	CA	27	9.4	13	20	16
	CT	5.5	5.1	8.2	26	12
VX	GM	1.7	2.2	2.2	6.2	7.6
	DL	7.2	3.1	2.3	1.9	2.6
	CA	9.4	7.3	7.5	2.1	7.6
	CT	4.5	3.7	10	2.9	4.2
TGD	GM	15	48	76	14	5.3
	DL	6.7	17	22	18	44
	CA	17	14	31	23	18
	CT	2.7	4	6.5	4.5	3.9

were analyzed every 10 samples. The daily check of the calibration curve at one midpoint level had to be within $\pm 25\%$ of the actual concentration. The method detection limit for the CWAs is typically 5 ng depending upon the substrate being analyzed.

Table 4.10-1 - Equipment Calibration Schedule

Equipment	Responsible Group	Frequency
RH probe	Manufacturer	Prior to testing
Gas Sensors	Manufacturer	Prior to testing
Volumetric Flow Controller	Teledyne Hastings	Prior to testing
Thermocouples	CUBRC Personnel	One-time two point calibration

4.8 Agent on Laboratory Blanks

Agent was not detected in any of the mVHP® or steam laboratory blank extracts.

4.9 Agent on Procedural Blanks

Procedural blank coupons placed in the chamber along with CWA-contaminated coupons showed that in only a few cases (HD on CA and CT) the CWA vapor cross-contaminated the “clean” samples. Non-porous procedural blank extracts (GM and DL) contained less agent mass than the extracts of the porous materials (CA and CT).

4.10 Equipment Calibration

The instrumentation and QA/QC procedures used to determine chemical agents are identified in Section 2.5.6. The analytical equipment needed for the analytical methods was maintained and operated according to the quality requirements and documentation of the Ashford Test Facility. All equipment was calibrated with the appropriate standards. Table 4.10-1 contains the equipment calibration schedule.

The Agilent GC/MS used for the analysis of the coupon extracts and vapor samples collected on sorbents was checked prior to beginning the analysis of each batch of samples using a minimum of five calibration reference standards. The GC/MS was recalibrated if the square of the correlation coefficient (r^2) from the regression analysis of these standards was <0.99 . With each batch of samples continuing calibration verification standards

5.0

Conclusions

This research program addressed the following specific questions:

- What is the decontamination efficacy of steam for removal of CWAs on IBMs as compared to controls at ambient environmental conditions?
- What is the decontamination efficacy of modified Vaporous Hydrogen Peroxide(VHP®) (mVHP®), modified in that ammonia is added to VHP®, for removal of CWAs on IBMs under various environmental and operational conditions as compared to controls at ambient environmental conditions?
- What are the physical effects of the decontaminants on the IBMs?

Prior to addressing these questions, persistence studies were completed. The persistence studies, completed for HD, indicate that the agent remained on the coupons for time periods equivalent to the selected decontamination exposure times; but HD did not persist beyond the 4-hour time point on the non-porous surfaces (DL and GM). The HD persistence was further studied in the ambient positive controls, which were run under the identical environmental conditions. These controls indicate that HD persisted for 400 min on all of the IBMs.

Persistence studies for GB deposited onto GM and DL demonstrate that GB falls below detectable limits within 30 minutes. These results led to these material-agent combinations being dropped from the decontamination efficacy and corresponding ambient positive control test matrices.

The steam results indicate a decontamination efficacy of greater than 99% for HD, GB and TGD within 60 minutes for all IBM types at both feed rates (1.5 and 3 kg/hr). The efficacy of steam decontamination against VX was >99% after 180 min exposure time for all materials except CA. Increased steam output appears to lead to higher decontamination efficacy for the VX on the carpet (98% efficacy at 180 min for 1.5 kg/hour output versus >99% at 180 min for 3 kg/hr). Due to normalization of the recovery masses to the single dose confirmation mass, statistical treatments of these data to discern if this difference is significant was not possible. Detectable concentrations of VX, GB, and GD were found in the condensate, indicating that these agents are not completely hydrolyzing during application of the steam and that the IBM decontamination process is a

combination of physical removal and hydrolysis. Finally, the steam fumigant did impact both porous materials (CA and CT) causing permanent damage to the CT.

The mVHP® results appear to indicate that increasing fumigant concentration slightly improved the HD decontamination efficacy for most of the material-exposure time combinations in the test matrix. The best efficacies were observed for the full flow conditions, with efficacies of 99% or better for all materials, except for CT, at the 350 ppmv target concentration conditions.

The most significant findings of this study were related to the effect of the generator output flow. This effect was seen for all HD-material combinations. For example, under mVHP® 250 ppmv full flow conditions, the DL was decontaminated to > 99% after 120 min whereas at the 10% output, an efficacy of only 88% was observed after 400 min. This effect was also seen for porous surfaces where 99% efficacy was observed at 120 min for CT, at the 350 ppmv full flow condition, while only 34% efficacy was observed at 120 min for the 10% flow condition. This same effect was also seen for VX where the decontamination efficacies for the 34 L/min flow were all less than or equal to 32% at 400 min while the efficacies for the full flow condition were 81-89%.

These test results, along with the corresponding environmental data (shown in Appendix D), indicate that two factors significantly affect test results – output flow and temperature. The individual effect of these variables could not be ascertained from this testing. The resulting chamber temperature differed with each of the output flows. The 10% flow test chamber temperature was typically around 24 °C while the chamber temperature was around 40 °C during the full flow tests. Agent evaporation is enhanced by elevated temperatures and increased flow resulting in reduction of the amount of agent on the coupon surface. Evaporation of HD was confirmed by the presence of the agent in all of the sorbent tube samples taken during decontamination testing.

The various diffuser configurations also complicated the analysis of the mVHP® data because these configurations may have altered the amount of fumigant each of the individual coupons was exposed to during a test. The standard deviations in the percent recoveries in the test samples were not higher than the standard deviations in the percent recoveries in the ambient positive controls indicating that these deviations were due to the dosing and extraction of the agent from the IBMs. These results

show that there was at least some homogeneity (within the area occupied by the 5 replicate coupons) in the fumigant-agent interactions and possibly the fumigant concentration.

An additional variation in the mVHP® test parameters was the gas concentration data generated by the vendor-provided sensors that were operated in a feedback loop with the fumigant generator. These sensors did not allow the VHP® or ammonia concentrations to reach and maintain their target concentrations. The VHP® sensor exhibited a positive bias during the tests where the test chamber temperature was elevated (the full flow tests) and the ammonia sensors saturated at their target concentration levels. During the reduced flow test runs, the VHP® concentrations were within 28% of the target concentrations yielding at least one set of test data that was generated using the vendor-recommended VHP® concentrations. The target ammonia concentration during all of the mVHP® tests exceeded the vendor-recommended concentration (10% of the target VHP® concentration versus the vendor-recommended 6%) but the generator typically operates at this higher concentration. This excess of ammonia should not affect the HD and VX test results because the purpose of the ammonia is to make the fumigant reactive towards the G-agents. Finally, the mVHP® fumigant did not impact the materials causing only a white residue to form where the agent droplet was applied onto the GM.

Scaling of these fumigation technologies was the largest technical challenge encountered during implementation of this testing program. For the interior of buildings, the air exchange rate is a significant factor in the reduction of contaminants. Any efficacy testing must consider the impact of air exchange (created by the generator output flow) on the results and this testing attempted to mimic the air exchange rate that is typical in a building or building section undergoing decontamination. As the testing progressed, the limitations of trying to adapt a system designed for use in large spaces to a small volume test chamber became increasingly apparent. Considerable effort was expended in trying to mitigate artifacts from testing discussed above (design changes to VHP® diffuser and STERIS sensor interface). The STERIS peroxide and ammonia flow controllers and software were not designed for small areas. Several “patches” were made over time to resolve issues as they were identified. The test schedule also impacted the program as several delays occurred, and the STERIS unit had limited availability which did not allow an extension to the test schedule. As a result, high priority was given to running tests within a short period of time resulting in delayed review of test data and test conditions. The consequence of this accelerated test schedule was that any potential test issues that negatively impacted the results could not be addressed before further testing

proceeded.

Conversely, scaling-up of the steam fumigation technology could be problematic. The presence of the GB, TGD and VX in the condensate indicated that if the steam fumigation were used to decontaminate the interior of a facility or section of a facility there would likely be re-deposition of these agents on other surfaces. However, the non-detectable levels of the agents on the procedural blanks directly adjacent to the test coupons indicate that the steam might be a suitable decontamination method if it were used in small areas where the condensate could be collected, such as a steam cleaner.

There are many different research questions that remain unanswered related to these two fumigants. First, the efficacies of these fumigants against the agents in vapor form are likely different from the efficacies observed for the liquid forms as determined under this effort. Future testing with the mVHP® and steam should include efficacy testing against vapor-contaminated samples. In addition, the effects of agent dwell time, especially for the persistent agents like VX, on the decontamination efficacy for these fumigants should be studied. Any future mVHP® testing should also incorporate the final diffuser configuration as well as adjustment of the VHP® target levels in the software to allow the concentrations to reach the vendor-recommended levels. Lastly, additional testing with the mVHP® fumigant should also include longer decontamination exposure times and additional materials.

6.0 Appendices

Appendix A - Determination of Extraction Efficiency of CWAs From IBMs

Table A.1 – Results of Solvent Extraction of VX from IBMs

Table A.2 – Results of Solvent Extraction of TGD from IBMs

Table A.3 – Results of Solvent Extraction of HD from IBMs

Table A.4 – Results of Solvent Extraction of GB from IBMs

Table A.5 – GB Persistence Time Study

Table A.1 - Results of Solvent Extraction Study for Extracting VX from IBMs

Sample ID	Hexane		Ethyl Acetate		Methylene Chloride		1:1 Hexane:Acetone	
	mg/ sample	% recovery	mg/ sample	% recovery	mg/ sample	% recovery	mg/ sample	% recovery
Reference in 10 mL solvent, R1	1.84	91	1.94	96	2.08	103	2.08	103
Reference in 10 mL solvent, R2	1.95	97	2.05	101	2.14	106	2.18	108
Reference in 10 mL solvent, R3	1.69	84	2.21	110	1.98	98	1.85	92
Mean		91		102		102		101
Blank decorative laminate	0.00	0	-	-	-	-	-	-
Decorative laminate, R1	2.18	108	-	-	-	-	-	-
Decorative laminate, R2	2.16	107	-	-	-	-	-	-
Decorative laminate, R3	2.14	106	-	-	-	-	-	-
Mean		107	-	-	-	-	-	-
Blank metal ductwork	0.00	0	-	-	-	-	-	-
Metal ductwork, R1	1.86	92	-	-	-	-	-	-
Metal ductwork, R2	2.14	106	-	-	-	-	-	-
Metal ductwork, R3	1.94	96	-	-	-	-	-	-
Mean		98						
Blank industrial grade carpet	0.00	0	0.00	0	0.00	0	0.00	0
Industrial grade carpet, R1	2.06	102	2.11	105	1.98	98	2.09	104
Industrial grade carpet, R2	2.19	109	2.18	108	2.12	105	2.43	121
Industrial grade carpet, R3	2.18	108	2.54	126	2.15	106	2.30	114
Mean		106		113		103		
Blank ceiling tile	0.00	0	0.00	0	0.00	0	0.00	0
Ceiling tile, R1	1.76	87	2.17	108	1.67	83	2.15	107
Ceiling tile, R2	1.51	75	1.94	96	1.47	73	1.87	93
Ceiling tile, R3	1.53	76	1.89	94	1.43	71	1.96	97
Mean		79		99		75		99

Agent was applied as four 0.5 µL droplets on each sample to result in 2.016 mg VX per coupon.

Laminate, ductwork and carpet samples were extracted in 10 mL of solvent. Ceiling tile was extracted in 20 mL solvent.

Coupons were extracted in solvent by ultrasonication for 10 minutes.

Table A.2 - Results of Solvent Extraction Study for Extracting TGD from IBMs

Sample ID	Hexane		Ethyl Acetate		Methylene Chloride		1:1 Hexane:Acetone	
	mg/ sample	% recovery	mg/ sample	% recovery	mg/ sample	% recovery	mg/ sample	% recovery
Reference in 10 mL solvent, R1	1.78	87	1.61	79	1.94	95	1.99	98
Reference in 10 mL solvent, R2	1.85	91	1.82	89	1.79	87	1.76	86
Reference in 10 mL solvent, R3	1.84	90	1.89	93	1.80	88	2.17	106
Mean		89		87		90		97
Blank decorative laminate	0.00	0	-	-	-	-	-	-
Decorative laminate, R1	1.74	85	-	-	-	-	-	-
Decorative laminate, R2	1.84	90	-	-	-	-	-	-
Decorative laminate, R3	1.87	91	-	-	-	-	-	-
Mean		89	-	-	-	-	-	-
Blank metal ductwork	0.00	0	-	-	-	-	-	-
Metal ductwork, R1	1.54	75	-	-	-	-	-	-
Metal ductwork, R2	1.99	98	-	-	-	-	-	-
Metal ductwork, R3	2.00	98	-	-	-	-	-	-
Mean		90						
Blank industrial grade carpet	0.00	0	0.00	0	0.00	0	0.00	0
Industrial grade carpet, R1	1.79	88	1.96	96	1.49	73	1.83	90
Industrial grade carpet, R2	1.75	86	1.68	82	1.88	92	1.82	89
Industrial grade carpet, R3	1.84	90	1.92	94	1.81	89	1.71	84
Mean		88		91		85		87
Blank ceiling tile	0.00	0	0.00	0	0.00	0	0.00	0
Ceiling tile, R1	1.22	60	1.89	93	1.65	80	1.74	85
Ceiling tile, R2	1.52	75	1.87	92	1.96	96	1.98	97
Ceiling tile, R3	1.40	69	2.05	100	1.64	80	1.85	91
Mean		68		95		86		91

Agent was applied as four 0.5 µL droplets on each sample to result in 2.04 mg GD per coupon.

Laminate, ductwork and carpet samples were extracted in 10mL of solvent. Ceiling tile was extracted in 20 mL solvent.

Coupons were extracted in solvent by ultrasonication for 10 minutes.

Table A.3 - Results of Solvent Extraction Study for Extracting HD from IBMs

Sample ID	Hexane		Ethyl Acetate		Methylene Chloride		1:1 Hexane:Acetone	
	mg/ sample	% recovery	mg/ sample	% recovery	mg/ sample	% recovery	mg/ sample	% recovery
Reference in 10 mL solvent, R1	2.63	104	2.82	111	2.88	113	2.62	103
Reference in 10 mL solvent, R2	2.67	105	2.71	107		99	a/	a/
Reference in 10 mL solvent, R3	2.65	104	2.77	109	2.97	117	3.00	118
Mean		104		109		110		111
Reference in 20 mL solvent, R1	2.68	106	-	-	-	-	-	-
Reference in 20 mL solvent, R2	2.60	103	-	-	-	-	-	-
Reference in 20 mL solvent, R3	2.62	103	-	-	-	-	-	-
Mean		104						
Blank decorative laminate	0.00	0	-	-	-	-	-	-
Decorative laminate, R1	2.83	112	-	-	-	-	-	-
Decorative laminate, R2	2.77	109	-	-	-	-	-	-
Decorative laminate, R3	2.73	107	-	-	-	-	-	-
Mean		109						
Blank metal ductwork	0.00	0	-	-	-	-	-	-
Metal ductwork, R1	2.77	109	-	-	-	-	-	-
Metal ductwork, R2	2.72	107	-	-	-	-	-	-
Metal ductwork, R3	2.69	106	-	-	-	-	-	-
Mean		107						
Blank industrial grade carpet	0.00	0	0.00	0	0.00	0	0.00	0
Industrial grade carpet, R1	2.62	103	2.82	111	2.52	99	2.75	108
Industrial grade carpet, R2	2.47	97	2.58	102	2.64	104	2.59	102
Industrial grade carpet, R3	2.79	110	2.59	102	2.66	105	2.51	99
Mean		103		105		103		103
Blank ceiling tile	0.00	0	0.00	0	0.00	0	0.00	0
Ceiling tile, R1	2.71	107	2.78	109	2.83	111	2.96	117
Ceiling tile, R2	2.78	109	2.78	109	2.64	104	2.77	109
Ceiling tile, R3	2.75	108	2.75	108	2.77	109	2.60	102
Mean		108		109		108		109

a/Sample Lost

Agent was applied as four 0.5 µL droplets on each sample to result in 2.54 mg HD per coupon.

Laminate, ductwork and carpet samples were extracted in 10 mL of solvent. Ceiling tile was extracted in 20 mL solvent.

Coupons were extracted in solvent by ultrasonication for 10 minutes

Table A.4 - Results of Solvent Extraction Study for Extracting GB from IBMs

Sample ID	Hexane		Ethyl Acetate *		Methylene Chloride		1:1 Hexane:Acetone	
	mg/ sample	% recovery	mg/ sample	% recovery	mg/ sample	% recovery	mg/ sample	% recovery
Reference in 10 mL solvent, R1	1.73	79	1.91	86	1.73	79	1.90	86
Reference in 10 mL solvent, R2	1.78	81	1.84	84	1.70	77	1.62	73
Reference in 10 mL solvent, R3	1.61	73	1.21	55	1.68	76	1.27	58
Mean		77		75		77		73
Blank decorative laminate	0.00	0	-	-	-	-	-	-
Decorative laminate, R1	1.59	72	-	-	-	-	-	-
Decorative laminate, R2	1.44	65	-	-	-	-	-	-
Decorative laminate, R3	1.76	80	-	-	-	-	-	-
Mean		72						
Blank metal ductwork	0.00	0	-	-	-	-	-	-
Metal ductwork, R1	1.91	87	-	-	-	-	-	-
Metal ductwork, R2	1.86	84	-	-	-	-	-	-
Metal ductwork, R3	1.82	83	-	-	-	-	-	-
Mean		84						
Blank industrial grade carpet	0.00	0	0.00	0	0.00	0	0.00	0
Industrial grade carpet, R1	1.99	90	2.25	102	2.02	92	1.77	80
Industrial grade carpet, R2	1.73	79	2.77	126	1.83	83	1.80	82
Industrial grade carpet, R3	1.70	77	2.72	124	1.88	85	1.84	83
Mean		82		117		87		82
Blank ceiling tile	0.00	0	0.00	0	0.00	0	0.00	0
Ceiling tile, R1	1.46	66	2.74	124	1.69	76	1.57	71
Ceiling tile, R2	1.39	63	2.76	125	1.69	77	1.56	71
Ceiling tile, R3	1.26	57	2.78	126	1.84	84	1.44	65
Mean		62		125		79		69

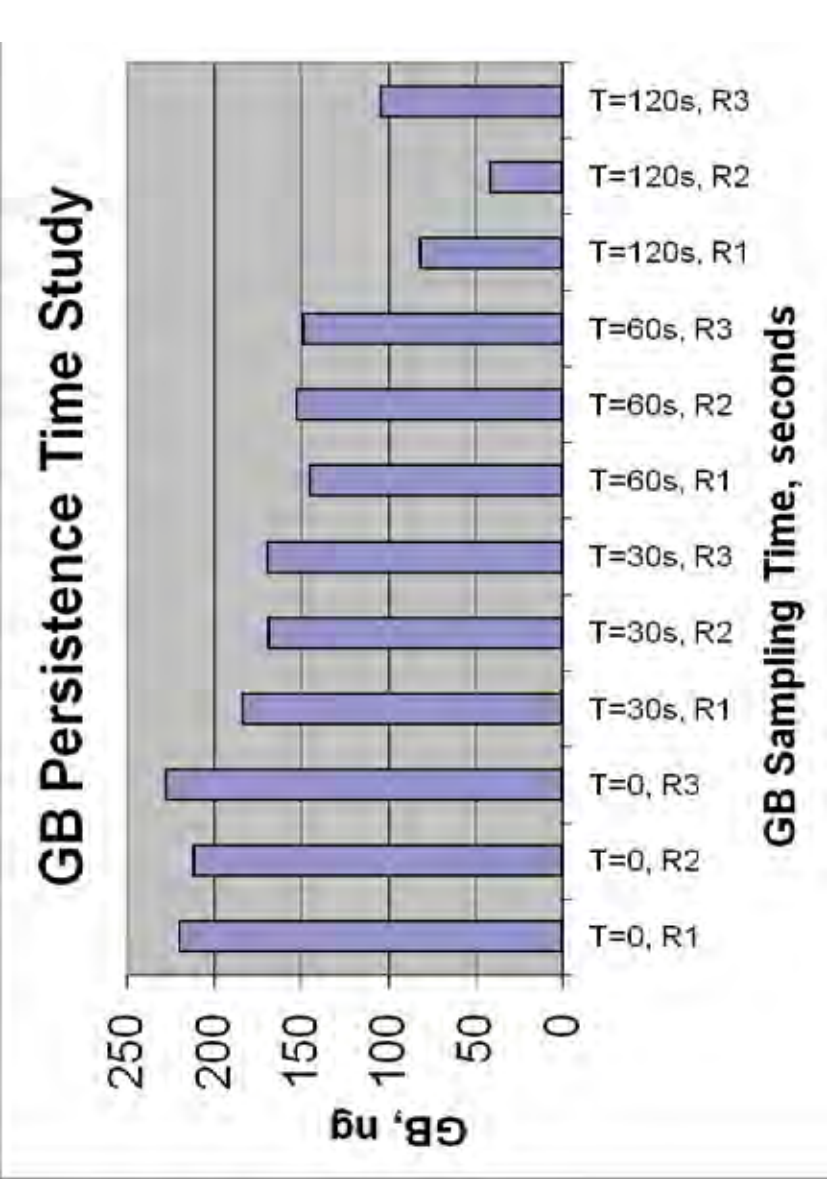
* Chromatography of GB in ethyl acetate is not suitable for testing due to extreme tailing.

Agent was applied as four 0.5 μ L droplets on each sample to result in 2.18 mg GB per coupon.

Laminate, ductwork and carpet samples were extracted in 10 mL of solvent. Ceiling tile was extracted in 20 mL solvent.

Coupons were extracted in solvent by ultrasonication for 10 minutes

Table A.5 - Results of GB Persistence Study on Glass



Sampling Time	GB, ng	% Recovery
T=0, R1	220	99.7
T=0, R2	212	96.2
T=0, R3	228	103.5
T=30s, R1	184	83.7
T=30s, R2	169	76.6
T=30s, R3	170	77.1
T=60s, R1	145	65.9
T=60s, R2	153	69.2
T=60s, R3	149	67.7
T=120s, R1	82	37.2
T=120s, R2	41	18.8
T=120s, R3	104	47.3

Purpose: Determine whether GB evaporates measurably in the time period between when the agent is applied to the coupon and when the coupon is placed in the extraction solvent.

During the extraction study, the agent residence time was approximately 30 seconds for all agents. This study compares extraction recoveries at 0, 0.5, 1 and 2 minute recovery times.

Agent was applied as four 0.5 µL droplets on glass at the time intervals indicated per sample (2.18 mg).

Samples were sonicated in hexane for 10 minutes.

Appendix B - Determination of Method Detection Limits (MDLs)

Table B.1 – Determination of Method Detection Limit (MDL) – Decorative Laminate Coupons

Table B.2 – Determination of Method Detection Limit (MDL) – Galvanized Steel Coupons

Table B.3 – Determination of Method Detection Limit (MDL) – Ceiling Tile Coupons

Table B.4 – Determination of Method Detection Limit (MDL) – Carpet Coupons

Table B.1 - Determination of Method Detection Limit (MDL) – Laminate Coupons

Sample Description	GB, ng	GD, ng	HD, ng	VX, ng
Laminate Rep 1	0.68	0.77	0.83	0.61
Laminate Rep 2	0.81	0.76	0.93	0.59
Laminate Rep 3	0.81	0.76	0.89	0.55
Laminate Rep 4	0.80	0.45	0.80	0.49
Laminate Rep 5	0.80	0.46	0.76	0.53
Laminate Rep 6	0.99	0.50	0.79	0.58
Laminate Rep 7	0.81	0.52	0.83	0.47
STD Dev.	0.09	0.15	0.06	0.05
GC/MS MDL (STDDEV x 3.143)	0.28	0.48	0.18	0.16
<i>Coupon DL, µg based upon 10 mL extract</i>				
	2.8	4.8	1.8	1.6

Table B.2 - Determination of Method Detection Limit (MDL) – Galvanized Steel Coupons

Sample Description	GB, ng	GD, ng	HD, ng	VX, ng
Galvanized Steel Rep 1	0.86	0.46	0.78	0.40
Galvanized Steel Rep 2	0.90	0.53	0.86	0.49
Galvanized Steel Rep 3	0.78	0.44	0.66	0.50
Galvanized Steel Rep 4	0.91	0.45	0.70	0.57
Galvanized Steel Rep 5	0.70	0.45	0.68	0.53
Galvanized Steel Rep 6	0.74	0.46	0.83	0.60
Galvanized Steel Rep 7	1.12	0.47	0.81	0.55
STD Dev.	0.14	0.03	0.08	0.06
MDL (STDDEV x 3.143)	0.44	0.10	0.25	0.20
<i>Coupon DL, µg based upon 10 mL extract</i>				
	4.4	1.0	2.5	2.0

Table B.3 - Determination of Method Detection Limit (MDL) – Carpet Coupons

Sample Description	GB, ng	GD, ng	HD, ng	VX, ng
Carpet Rep 1	0.71	0.63	0.67	0.75
Carpet Rep 2	0.85	0.62	0.58	1.02
Carpet Rep 3	0.70	0.60	0.51	0.96
Carpet Rep 4	0.79	0.59	0.67	1.02
Carpet Rep 5	0.94	0.68	0.60	0.92
Carpet Rep 6	0.88	0.61	0.76	1.07
Carpet Rep 7	0.83	0.68	0.72	1.19
STD Dev.	0.09	0.03	0.09	0.14
MDL (STDDEV x 3.143)	0.28	0.11	0.27	0.43
<i>Coupon DL, µg based upon 10 mL extract</i>				
	2.8	1.1	2.7	4.3

Table B.4 - Determination of Method Detection Limit (MDL) – Ceiling Tile Coupons

Sample Description	GB, ng	GD***, ng	HD, ng	VX***, ng
Ceiling Tile Rep 1	0.91	0.64	1.19	0.33
Ceiling Tile Rep 2	0.80	0.68	1.01	0.27
Ceiling Tile Rep 3	0.86	0.57	0.88	0.35
Ceiling Tile Rep 4	0.71	0.52	0.56	0.30
Ceiling Tile Rep 5	0.75	0.59	0.59	0.39
Ceiling Tile Rep 6	0.67	0.61	0.71	0.45
Ceiling Tile Rep 7	0.67	0.45	0.68	0.31
STD Dev.	0.09	0.08	0.23	0.06
MDL (STDDEV x 3.143)	0.29	0.24	0.73	0.19
<i>Coupon DL, µg based upon 20 mL extract</i>				
	5.9	4.9	14.6	3.8

*** 50% hexane/acetone solvent system was used for these extractions

Appendix C - Steam Results Graphs

Figure C.1 Bar graph of μg of VX recovered per sample for the 3 kg/hr steam tests.

Figure C.2 Bar graph of μg of VX recovered per sample for the 1.5 kg/hr steam tests.

Figure C.3 Bar graph of μg of HD recovered per sample for the 1.5 kg/hr steam tests.

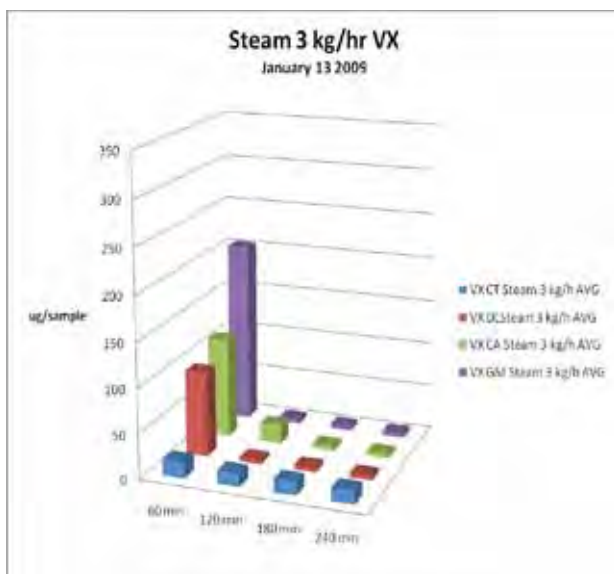


Figure C.1 - Bar graph of μg of VX recovered per sample for the 3 kg/hr steam tests.

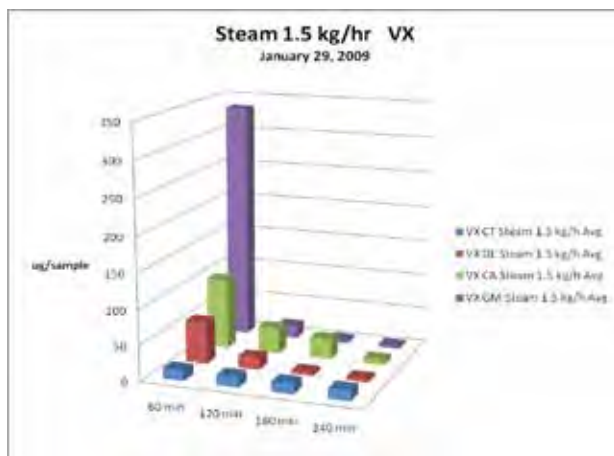


Figure C.2 - Bar graph of μg of VX recovered per sample for the 1.5 kg/hr steam tests.

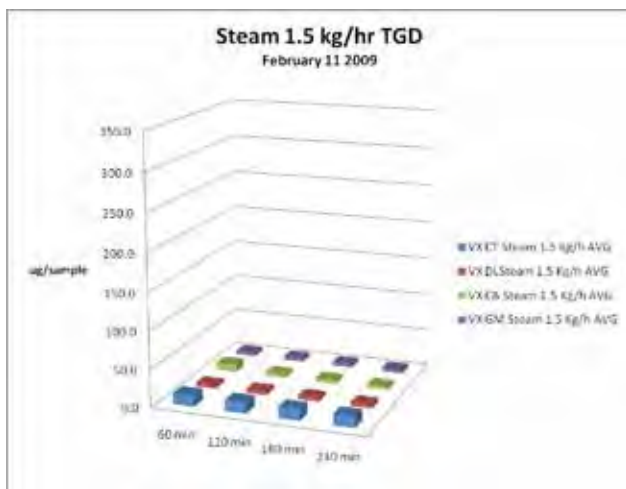


Figure C.3 - Bar graph of μg of HD recovered per sample for the 1.5 kg/hr steam tests.

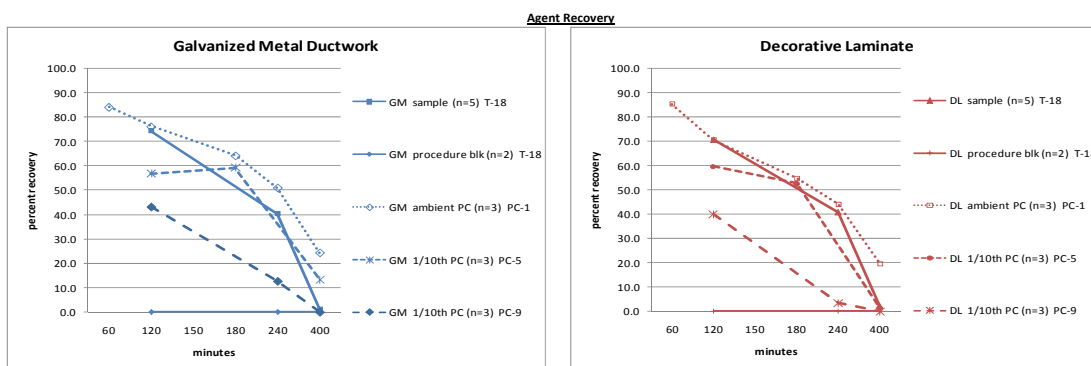
Appendix D - mVHP® Recovery Graphs

Samples, Positive Controls, Procedural Blanks and Environmental Conditions

Table D.1	HD, GM/DL, 10%, 250 ppmv
Table D.2	HD, CA/CT, 10%, 250 ppmv
Table D.3	Temperature, RH and Flow – HD, 10%, 250 ppmv
Table D.4	HD, GM/DL, 100%, 250 ppmv
Table D.5	HD, CA/CT, 100%, 250 ppmv
Table D.6	Temperature, RH and Flow – HD, 100%, 250 ppmv
Table D.7	HD, GM/DL, 10%, 350 ppmv
Table D.8	HD, CA/CT, 10%, 350 ppmv
Table D.9	Temperature, RH and Flow – HD, 10%, 350 ppmv
Table D.10	HD, GM/DL, 100%, 350 ppmv
Table D.11	HD, CA/CT, 100%, 350 ppmv
Table D.12	Temperature, RH and Flow – HD, 100%, 350 ppmv
Table D.13	VX, GM/DL, 10%, 250 ppmv
Table D.14	VX, CA/CT, 10%, 250 ppmv
Table D.15	Temperature, RH and Flow – VX, 10%, 250 ppmv
Table D.16	VX, GM/DL, 100%, 250 ppmv
Table D.17	VX, CA/CT, 100%, 250 ppmv
Table D.18	Temperature, RH and Flow – VX, 100%, 250 ppmv

Table D.1 - HD, GM/DL, 10%, 250 ppmv

Environmental Conditions & HD Test Results - Galvanized Metal and Decorative Laminate
10% Output With a Peroxide Level of 250 ppmv



Diffuser Configuration (DC): T-18 = DC 3, PC-5 = DC 1, PC-9 = DC 4, PC1 = NA

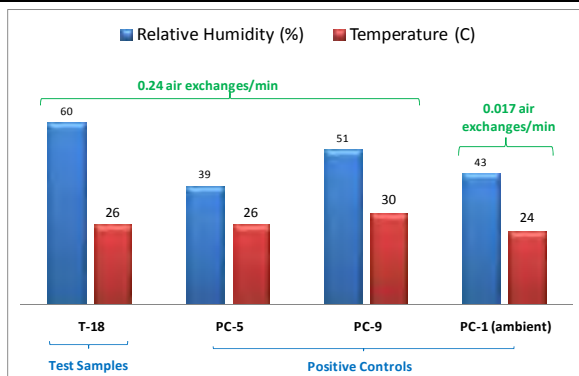
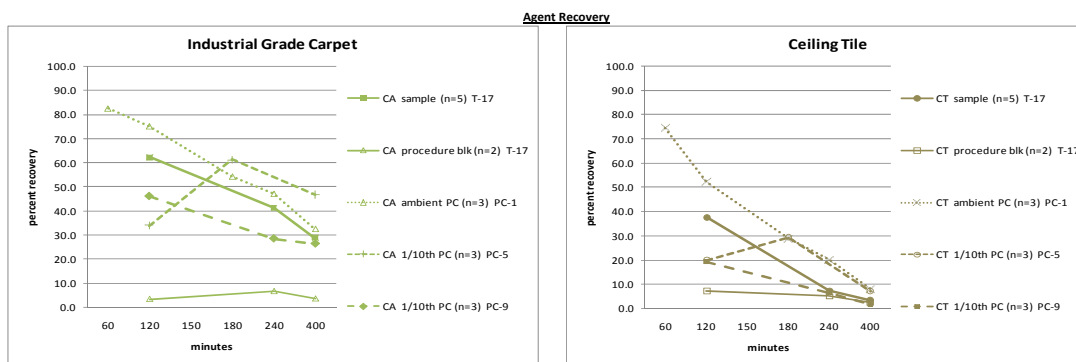


Table D.2 - HD, CA/CT, 10%, 250 ppmv

Environmental Conditions & HD Test Results - Industrial Grade Carpet and Ceiling Tile
10% Output With a Peroxide Level of 250 ppmv



Diffuser Configuration (DC): T-17 = DC 2, PC-5 = DC 1, PC-9 = DC 4, PC1 = NA

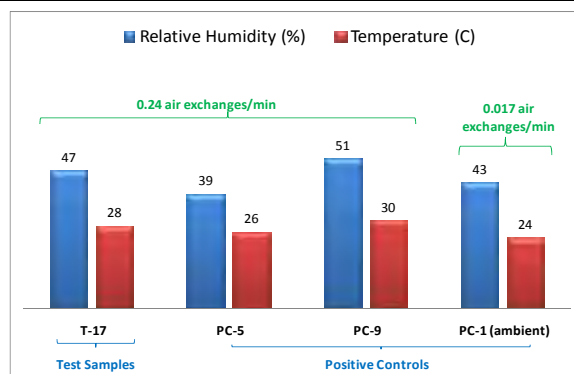
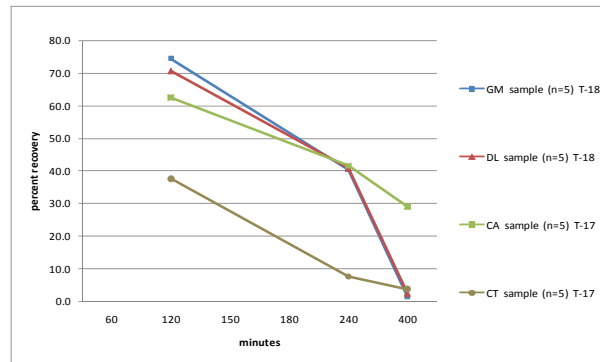


Table D.3 - Temperature, RH and Flow – HD, 10%, 250 ppmv

Temperature, RH and Flow Results for Testing of mVHP® Fumigant for HD
10% Flow With a Peroxide Level of 250 ppmv



Diffuser Configuration (DC): T-18 = DC 3, T-17 = DC 2

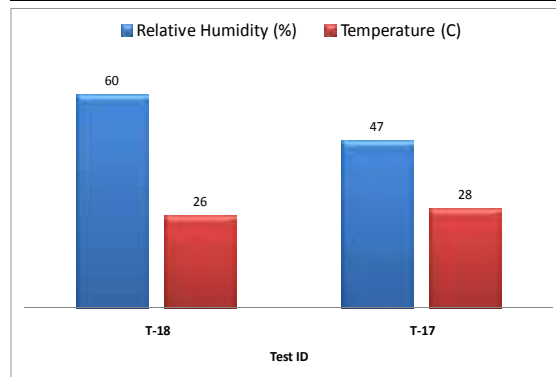
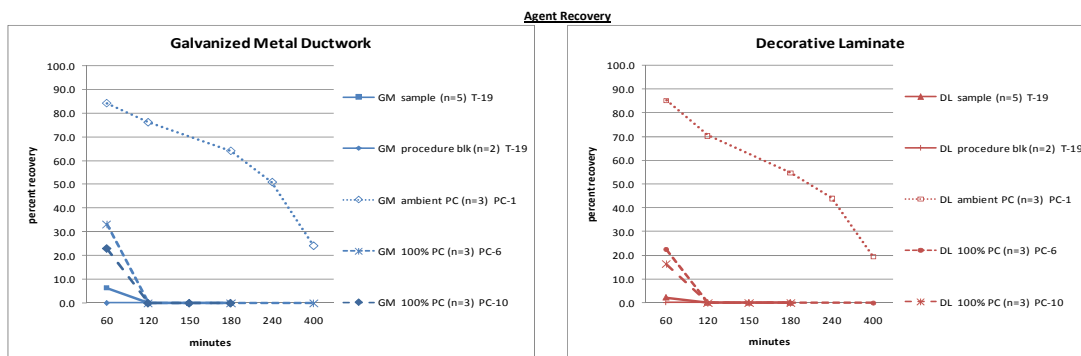


Table D.4 - HD, GM/DL, 100%, 250 ppmv

Environmental Conditions & HD Test Results - Galvanized Metal and Decorative Laminate
100% Output With a Peroxide Level of 250 ppmv



Diffuser Configuration (DC): T-19 = DC 4, PC-6 = DC 3, PC-10 = DC 4, PC1 = NA

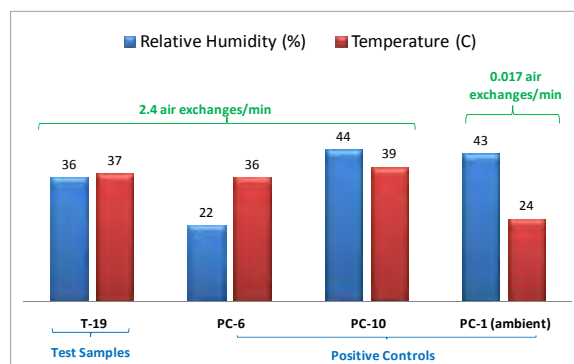
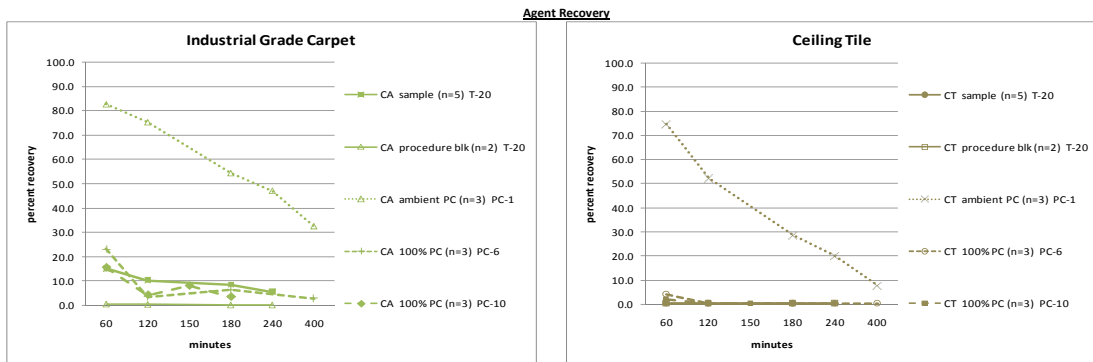


Table D.5 - HD, CA/CT, 100%, 250 ppmv

Environmental Conditions & HD Test Results - Industrial Grade Carpet and Ceiling Tile
100% Output With a Peroxide Level of 250 ppmv



Diffuser Configuration (DC): T-20 = DC 4, PC-6 = DC 3, PC-10 = DC 4, PC1 = NA

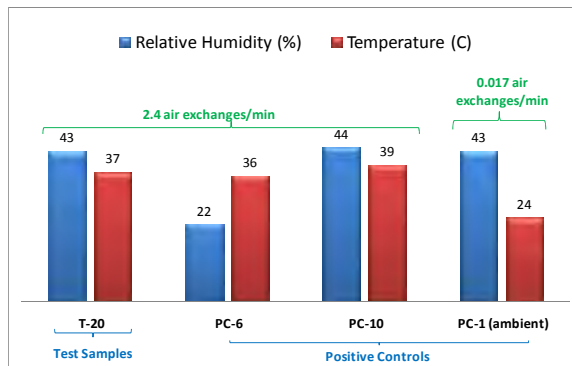
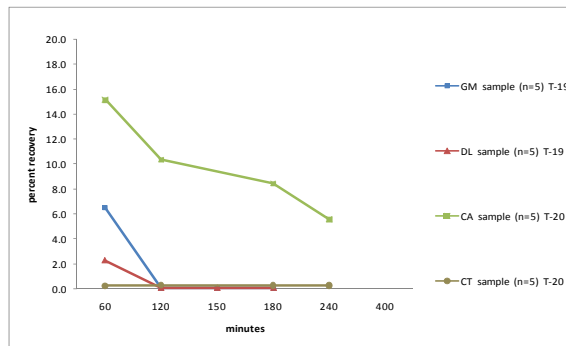


Table D.6 - Temperature, RH and Flow – HD, 100%, 250 ppmv

Temperature, RH and Flow Results for Testing of mVHP® Fumigant for HD
100% Output With a Peroxide Level of 250 ppmv



Diffuser Configuration (DC): T-19 = DC 4, T-20 = DC 4

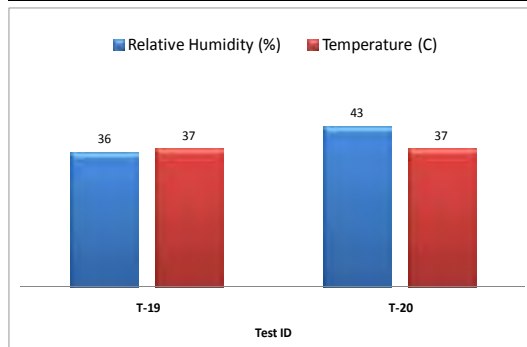
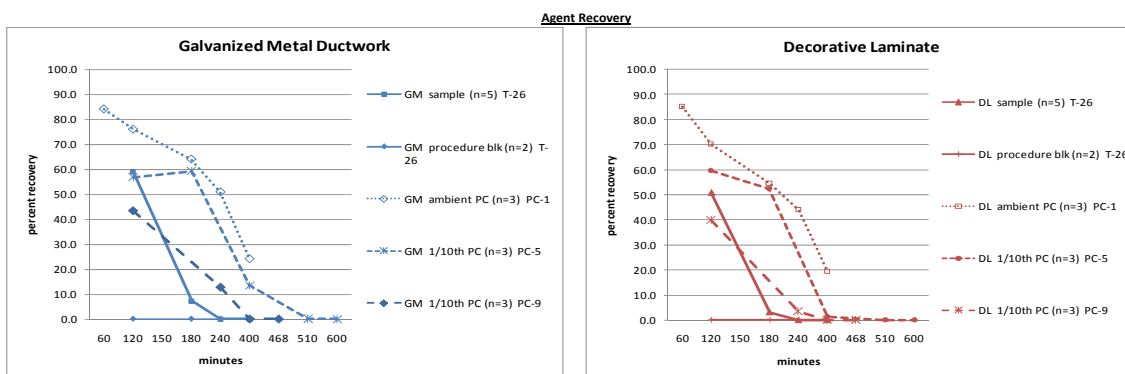


Table D.7 - HD, GM/DL, 10%, 350 ppmv

Environmental Conditions & HD Test Results - Galvanized Metal and Decorative Laminate
10% Output With a Peroxide Level of 350 ppmv



Diffuser Configuration (DC): T-26 = DC 4, PC-5 = DC 1, PC-9 = DC 4, PC1 = NA

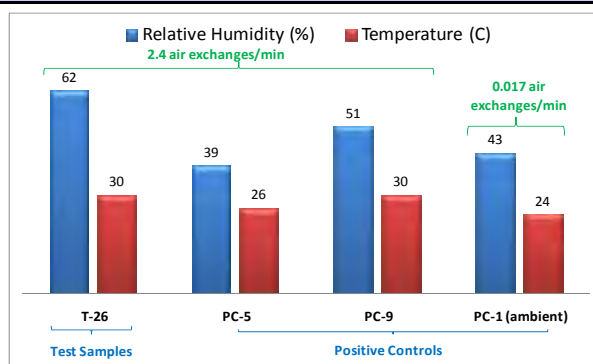
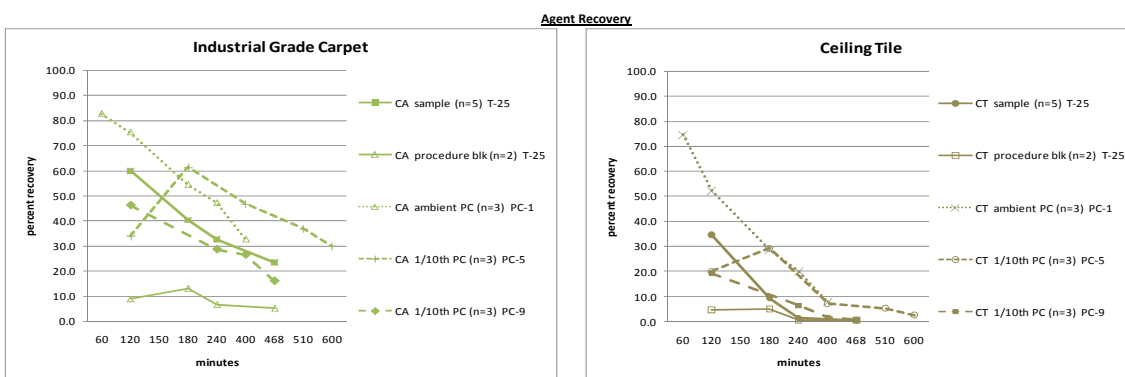


Table D.8 - HD, CA/CT, 10%, 350 ppmv

Environmental Conditions & HD Test Results - Industrial Grade Carpet and Ceiling Tile
10% Output With a Peroxide Level of 350 ppmv



Diffuser Configuration (DC): T-25 = DC 4, PC-5 = DC 1, PC-9 = DC 4, PC1 = NA

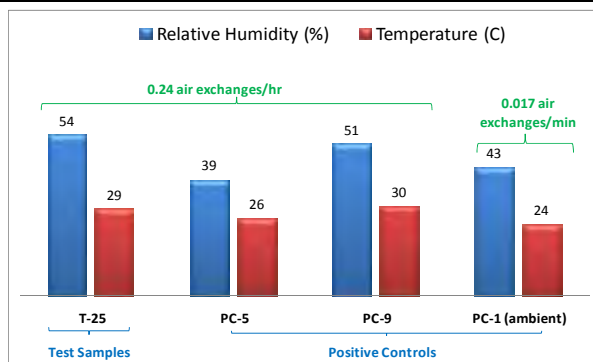
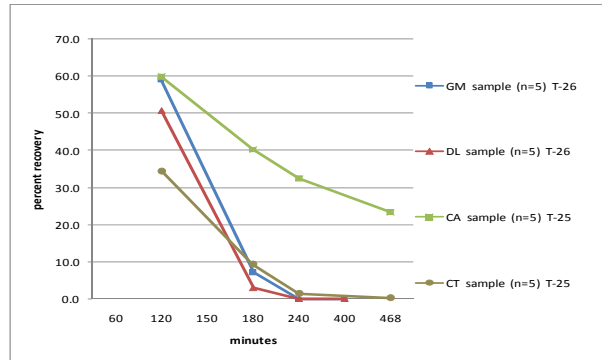


Table D.9 - Temperature, RH and Flow – HD, 10%, 350 ppmv

Temperature, RH and Flow Results for Testing of mVHP® Fumigant for HD
10% Output With a Peroxide Level of 350 ppmv



Diffuser Configuration (DC): T-26 = DC4, T-25 = DC 4

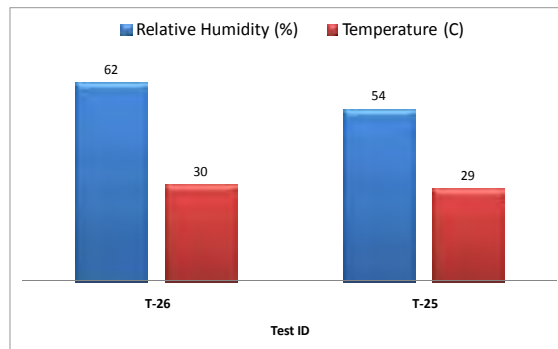
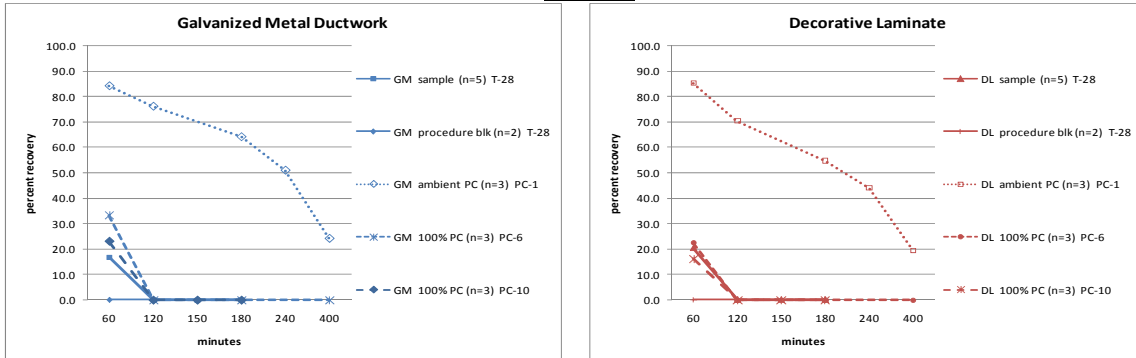


Table D.10 - HD, GM/DL, 100%, 350 ppmv

Environmental Conditions & HD Test Results - Galvanized Metal and Decorative Laminate
100% Output With a Peroxide Level of 350 ppmv

Agent Recovery



Diffuser Configuration (DC): T-28 = DC 4, PC-6 = DC 3, PC-10 = DC 4, PC1 = NA

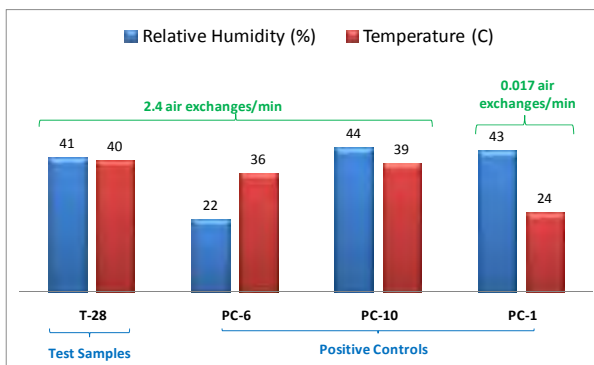
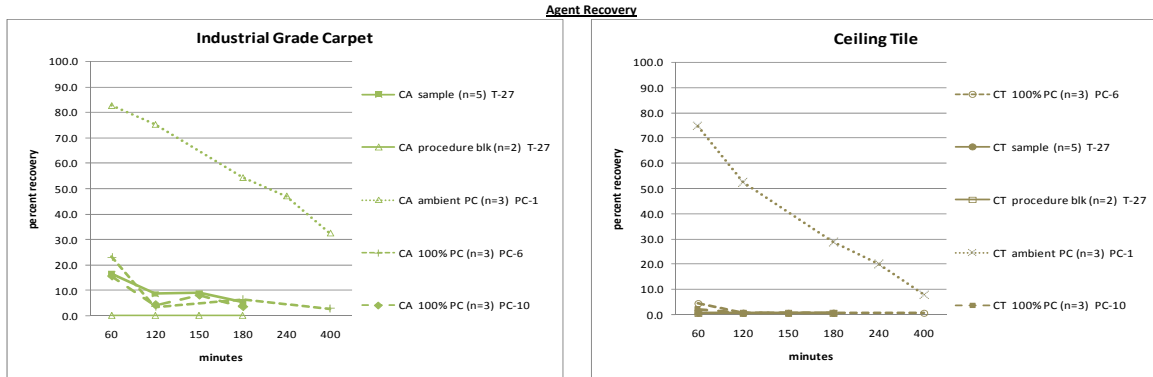


Table D.11 - HD, CA/CT, 100%, 350 ppmv

Environmental Conditions & HD Test Results - Industrial Grade Carpet and Ceiling Tile
100% Output With a Peroxide Level of 350 ppmv



Diffuser Configuration (DC): T-27 = DC 4, PC-6 = DC 3, PC-10 = DC 4, PC1 = NA

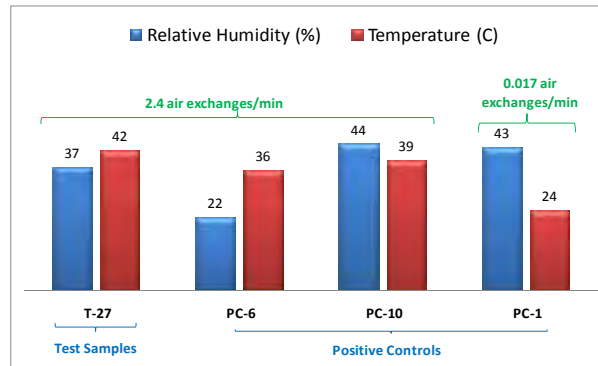
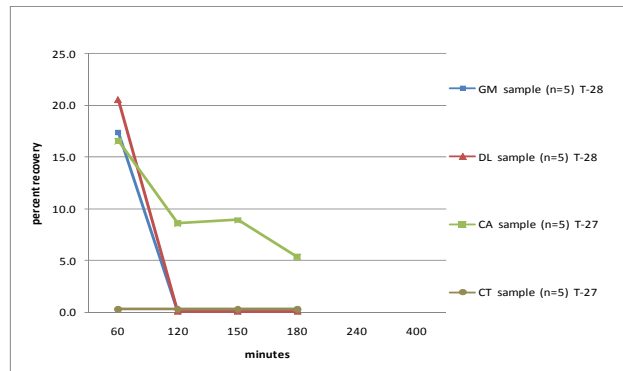


Table D.12 - Temperature, RH and Flow – HD, 100%, 350 ppmv

Temperature, RH and Flow Results for Testing of mVHP® Fumigant for HD
100% Output With a Peroxide Level of 350 ppmv



Diffuser Configuration (DC): T-28 = DC 4, T-27 = DC 4

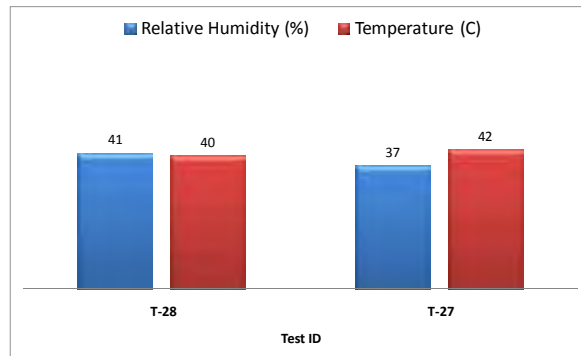
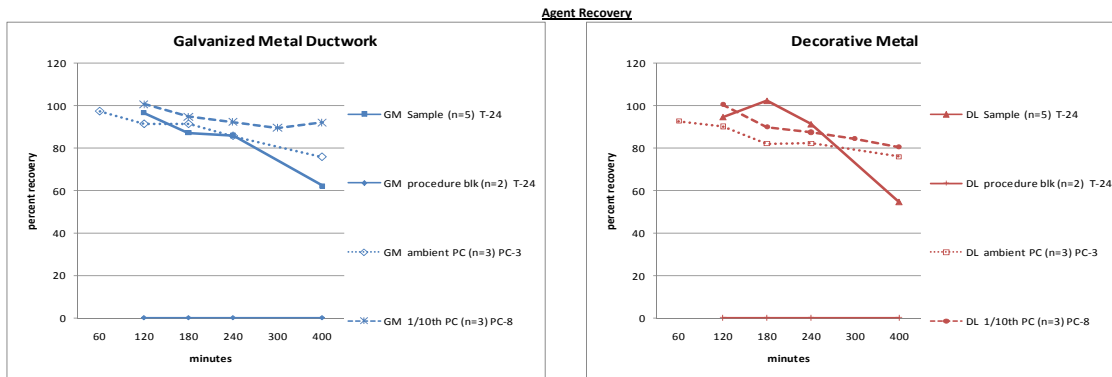


Table D.13 - VX, GM/DL, 10%, 250 ppmv

Environmental Conditions & VX Test Results - Galvanized Metal and Decorative Laminate
10% Output With a Peroxide Level of 250 ppmv



Diffuser Configuration (DC): T-24 = DC 4, PC-8 = DC 4, PC3 = NA

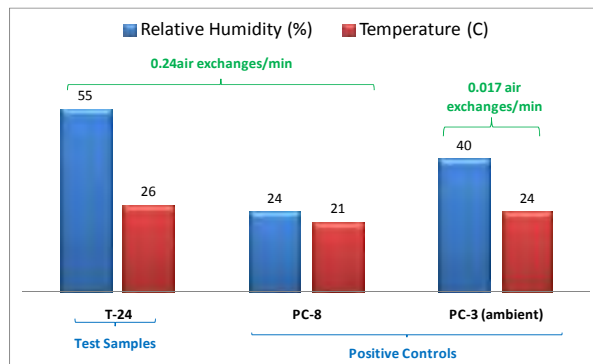
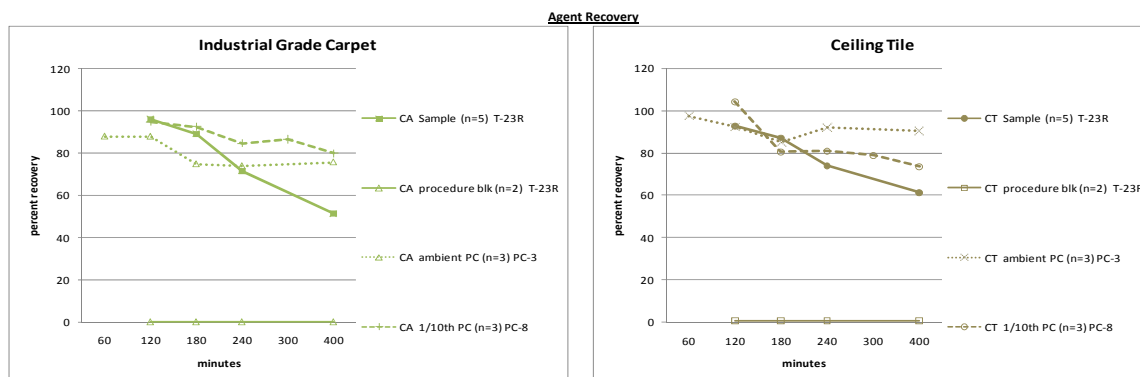


Table D.14 - VX, CA/CT, 10%, 250 ppmv

Environmental Conditions & VX Test Results - Industrial Grade Carpet and Ceiling Tile
10% Output With a Peroxide Level of 250 ppmv



Diffuser Configuration (DC): T-23R = DC 4, PC-8 = DC 4, PC3 = NA

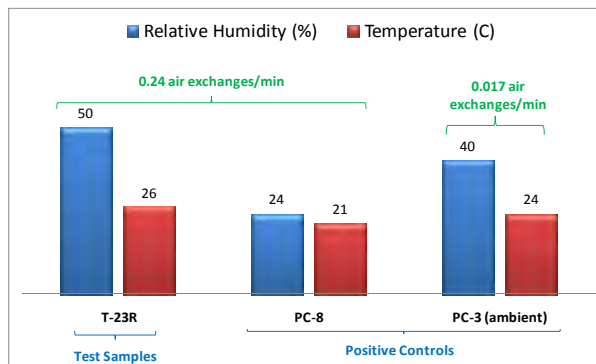
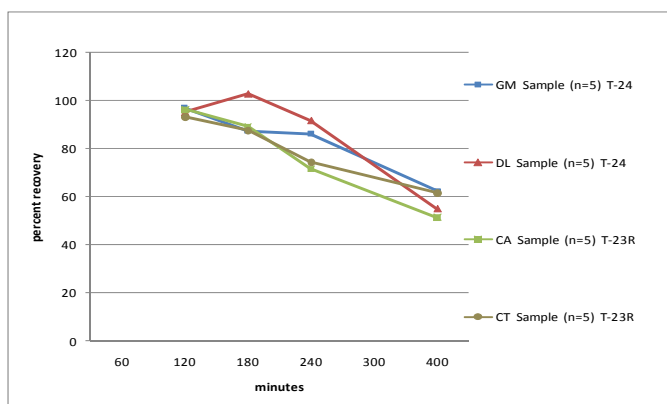


Table D.15 - Temperature, RH and Flow – VX, 10%, 250 ppmv

Temperature, RH and Flow Results for Testing of mVHP® Fumigant for VX
One-tenth Output With a Peroxide Level of 250 ppmv



Diffuser Configuration (DC): T-24 = DC 4, T-23R = DC 4

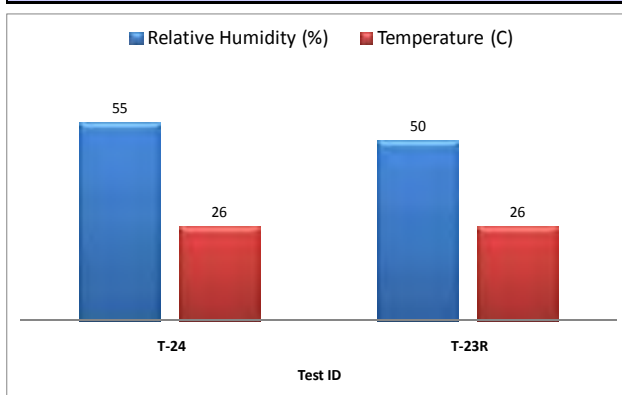
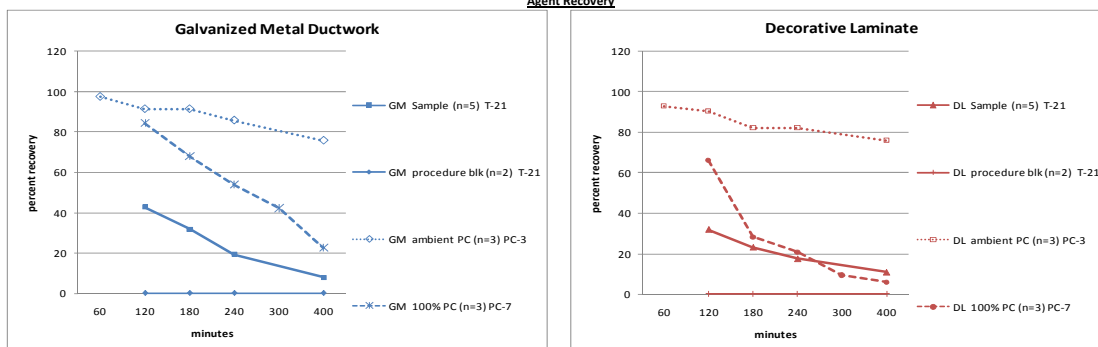


Table D.16 - VX, GM/DL, 100%, 250 ppmv

Environmental Conditions & VX Test Results - Galvanized Metal and Decorative Laminate
100% Output With a Peroxide Level of 250 ppmv

Agent Recovery



Diffuser Configuration (DC): T-21 = DC 4, PC-7 = DC 4, PC3 = NA

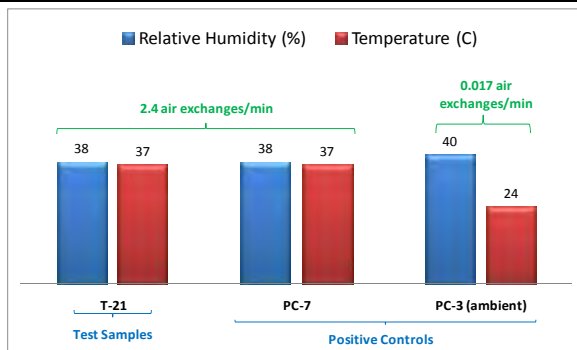
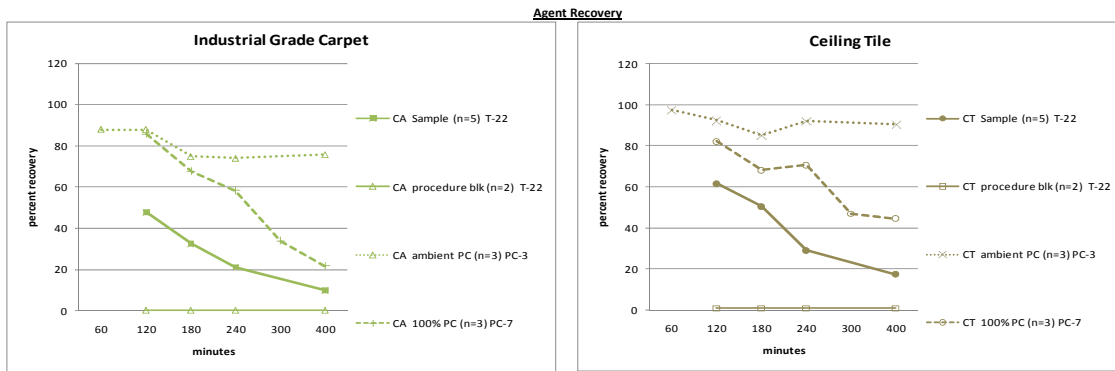


Table D.17 - VX, CA/CT, 100%, 250 ppmv

Environmental Conditions & VX Test Results - Industrial Grade Carpet and Ceiling Tile
100% Output With a Peroxide Level of 250 ppmv



Diffuser Configuration (DC): T-22 = DC 4, PC-7 = DC 4, PC3 = NA

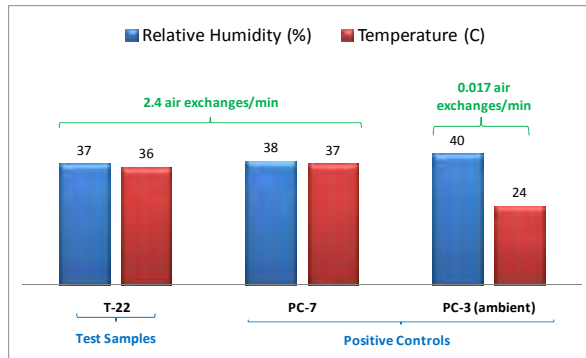
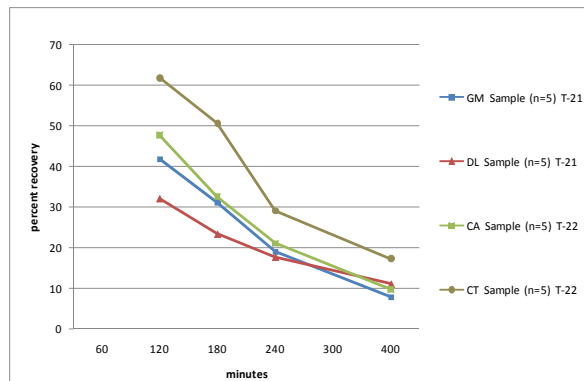
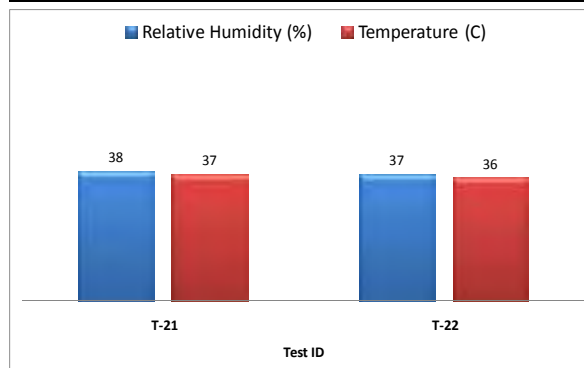


Table D.18 - Temperature, RH and Flow – VX, 100%, 250 ppmv

Temperature, RH and Flow Results for Testing of mVHP® Fumigant for VX
100% Output With a Peroxide Level of 250 ppmv



Diffuser Configuration (DC): T-21 = DC 4, T-22 = DC 4



7.0

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