

Material Demand Studies: Materials Sorption of Vaporized Hydrogen Peroxide



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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 in carrying 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 determination and selection of an appropriate technology depends on many factors including the anticipated impacts on materials and equipment, costs, logistics of application, waste generation, and health and safety.

This document provides specific information to aid in deciding on the appropriateness of a particular decontamination agent: vaporized hydrogen peroxide (VHP®). Past large-scale use of the fumigant resulted in a significant difference between anticipated and actual VHP® generation requirements necessary to achieve the target hydrogen peroxide concentration in the enclosed space. This was believed to be due to the consumption of hydrogen peroxide vapor by materials in the facility. The study undertaken provides results of the demand (or consumption) that the resident materials (walls, carpet, etc.) exhibit for hydrogen peroxide when used to cleanup areas following chemical or biological contamination. This information is useful to help determine fumigant generation requirements and cleanup strategies for facilities.

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. NHSRC has made this publication available to assist the response community prepare for and recover from disasters involving chemical and biological 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

Preface

To address Homeland Security needs for decontamination, the U.S. Environmental Protection Agency (EPA) established an Interagency Agreement with the U.S. Army Edgewood Chemical and Biological Center (ECBC) to take advantage of ECBC's extensive expertise and specialized research facilities for the decontamination of surfaces contaminated with chemical and biological (CB) warfare agents. The EPA's National Homeland Security Research Center (NHSRC) collaborated with ECBC to determine the impact of vaporized hydrogen peroxide (decontaminant) on indoor surfaces in buildings. The vaporized hydrogen peroxide work was completed under EPA IAG DW 939917-01-0. The work was conducted from November 2003 to July 2005 and recorded in ECBC laboratory notebooks 05-0059, 04-0043, and 05-0044.

The use of either trade or manufacturers' names in this report does not constitute an official endorsement of any commercial products. Manufacturer names and model numbers are provided for completeness. This technical report may not be cited for purposes of advertisement.

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Acknowledgments

The authors thank the following individuals for their contributions toward the successful completion of this test program. The authors thank Mr. Dave Stark for completing the independent audits per the EPA program requirements. The authors also thank Dr. David Cullinan for preparing many coupon run baskets, performing coupon measurements and preparing chain-of-custody forms during the time his assigned laboratory was closed. The authors thank Mr. Dave Sorrick for assistance with acquiring materials and equipment fabrication. The authors thank Ms. Diane Simmons for assistance with the issuance of this report. The authors thank Dr. Emily Snyder, Dr. Zhishi Guo and Mr. Leroy Mickelsen for reviewing and commenting on the final report.

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List of Acronyms and Symbols

A	surface area
APG	Aberdeen Proving Ground
ASTM	American Society for Testing and Materials
Ave	average
b	As a subscript in Section 4.5, the baseline tests
CB	chemical and biological
CoC	chain of custody
cfm	cubic feet per minute
CT	concentration multiplied by time
CT_{inlet}	CT of the affluent
$CT_{diff,b}$	difference in CT_{inlet} and CT_{outlet} for the baseline tests
$CT_{diff,k}$	difference in CT_{inlet} and CT_{outlet} for a specific material after baseline correction
CT_{outlet}	CT of the effluent
$d_{H_2O_2}$	density (g/L) of H_2O_2 at 30 °C using $PV = nRT$
doc	documentation
DS	Decontamination Sciences
ECBC	Edgewood Chemical and Biological Center
EOR	end of run
EPA	U.S. Environmental Protection Agency
F_{total}	total volume defined by test limits (0 – 1000 ppmv-h)
h	height
GSA	General Services Administration
H_2O_2	hydrogen peroxide
hr or hrs	hour or hours
IAW	in accordance with
IOP	Internal Operating Procedure
IS	internal standard
ISO 17025	International Standardization Organization Standard 17025 on Laboratory Quality Procedures
l	length
J	mass flux
mb	specific material
MD	material demand
min	minutes
$M_{H_2O_2}$	mass of hydrogen peroxide
NHSRC	U.S. EPA National Homeland Security Research Center

P _{sys}	chamber pressure
ppb	part per billion
ppmv	part per million by volume
QA	quality assurance
QAPP	Quality Assurance Project Plan (QAPP)
QMP	Quality Management Plan
RH	relative humidity
SOP(s)	standing operating procedure(s) (standard may also be used in place of standing with the same meaning)
Std dev	standard deviation
T	temperature
t	time
SOR	start of run
TIC(s)	toxic industrial chemical(s)
TIM(s)	toxic industrial material(s)
TR	technical report
TSA	technical systems audit
U.S.	United States
UV	ultraviolet
Ver	version
VHP®	Steris' registered "vaporized hydrogen peroxide" procedure
w	width

COUPON SPECIFIC CODING

"W"	bare wood
"R"	carpet
"T"	ceiling suspension tile
"G"	latex-painted gypsum wallboard
"S"	painted structural A572 steel
"C"	unpainted concrete cinder block

1.0 Background

The Material Demand effort was designed to determine how building materials impact the ability to maintain a target decontaminant vapor concentration within an enclosed interior space. The building materials may impact the decontaminant vapor concentration by either sorption or decomposition of the decontaminant. Since building interiors may contain large surface areas consisting of different materials, data are needed to determine how these interior surfaces affect the ability to maintain a stable target concentration. Vaporized hydrogen peroxide (VHP®) and chlorine dioxide (ClO₂) were selected since these decontamination technologies

have been used to decontaminate indoor surfaces contaminated by anthrax and/or show potential for use in decontaminating indoor surfaces contaminated by chemical agents. Vaporized hydrogen peroxide (VHP®) results are presented in this report. The representative building interior materials tested were unpainted concrete cinder block, standard stud lumber (wood 2"x 4", fir, type-II), latex-painted ½-inch gypsum wallboard, ceiling suspension tile, painted structural steel and carpet.

Summary of Conclusions

The conclusions reached in this study are summarized below.

- The VHP[®] Material Demand tests showed that the building materials affect the VHP decontamination vapor concentration. The impact varies based on the type of material.
 - The concrete cinder block coupon had the greatest impact on maintaining the VHP[®] concentration due to decomposition of the VHP[®].
 - The cellulose-based materials, wood and ceiling tile, showed adsorption of the VHP[®] with a high material demand value approximately one-third to one-half the value for concrete. VHP[®] desorption resulted in a long aeration time.
 - The wallboard had a moderate effect on the VHP[®] concentration compared to concrete cinder block.
 - The carpet and steel coupons had a low impact on the VHP[®] concentration compared to concrete cinder block.
- The relative humidity sensor measurements were affected by the presence of VHP[®]. The sensor read high in the presence of VHP[®], but responded normally with no apparent visual degradation after the VHP[®] was removed.
- The hydrogen peroxide sensor performance verification using the wet-chemical titration method showed that both the inlet and outlet sensors were not adversely affected by prolonged and repeated exposure to VHP[®]. Neither of the two sensors showed evidence of visual deterioration or change in response during the testing period.
- Hydrogen peroxide concentration sensor calibration using sulfur dioxide in nitrogen gas revealed that the sensors were highly sensitive to changes in pressure. For the custom built exposure chambers, inline sensor calibration is recommended.
- The percentage of VHP[®] decomposition in the chamber was a function of VHP[®] / air flow through the chamber. A VHP[®]/air flow of 3.0 ft³ / min was used in all tests.

3.0

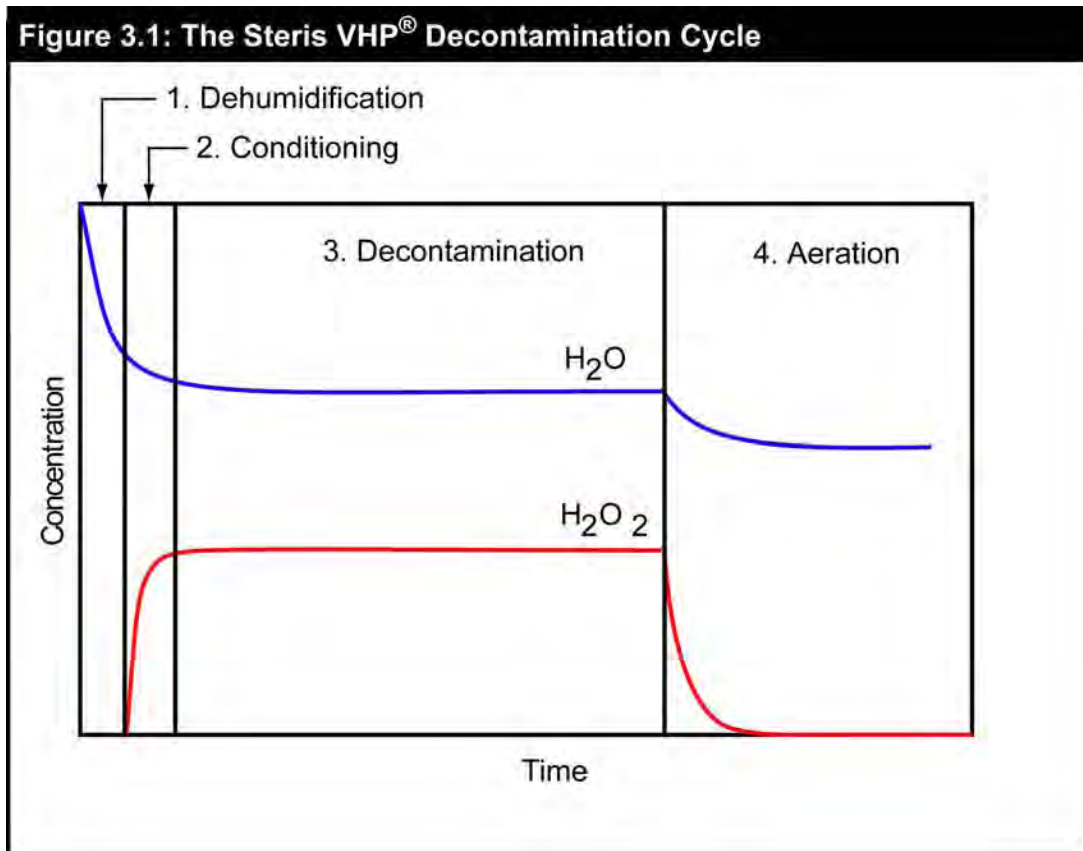
Introduction

To address Homeland Security needs for decontamination, the U.S. Environmental Protection Agency (EPA) established an Interagency Agreement with the U.S. Army Edgewood Chemical and Biological Center (ECBC) to take advantage of ECBC's extensive expertise and specialized research facilities for the decontamination of surfaces contaminated with chemical and biological (CB) warfare agents. The EPA National Homeland Security Research Center (NHSRC) formed a collaboration with ECBC in a mutual leveraging of resources, expanding upon ECBC's ongoing programs in CB decontamination to more completely address the parameters of particular concern for decontamination of indoor surfaces in buildings following a terrorist attack using CB agents, toxic industrial chemicals (TICs) or materials (TIMs). Vaporized hydrogen peroxide (VHP®) and chlorine dioxide (ClO₂) are decontamination technologies that have been used to decontaminate indoor surfaces contaminated with anthrax and show potential for use in decontaminating indoor surfaces contaminated by chemical agents. This program is specifically focused on decontamination of the building environment, for purposes of restoring a public building to a usable state after a terrorist attack. Systematic testing of decontamination technologies generates objective performance data so building and facility managers, first responders, groups responsible for building decontamination, and other technology buyers and users can make informed purchase and application decisions.

Since building interiors contain a large surface composed of different materials, the Material Demand effort was designed to determine how building materials impact the concentration of decontaminant in the vapor phase. The objective of this study was to establish and conduct laboratory test procedures to determine to what degree building materials affect the vaporized decontaminants. The building interior materials used for testing are a subset of the variety of structural, decorative and functional materials common to commercial office buildings regardless of architectural style and age. The building materials encompass a variety of material compositions and porosities. The materials studied included unpainted concrete cinder block, standard stud lumber (wood 2"x 4", fir), latex-painted ½-inch gypsum wallboard, acoustical ceiling suspension tile, primer-painted structural steel and carpet. The focus of this technical report is the evaluation of the building interior materials and VHP®.

The VHP® technology developed by Steris (EPA registration #58779-4) has been in use for more than a decade. The VHP® fumigant was initially used to sterilize pharmaceutical processing equipment and clean rooms.^{1,2} In response to the anthrax attacks of October 2001, Steris adapted its VHP® technology to perform the decontamination of two U.S. government facilities, the General Services Administration (GSA) Building 410 at Anacostia Naval Base, Washington DC, and the U.S. Department of State SA-32 "Sterling, VA" mail center. Through a joint venture with Steris Corporation, the application of vaporized hydrogen peroxide (VHP®) and modified VHP® for chemical- and biological-agent decontamination have been successfully tested in laboratory, large-chamber and field demonstrations including a former office building and a C141 aircraft.³⁻⁵ Vaporized hydrogen peroxide (VHP®) also reacts with and neutralizes VX and HD chemical agents.⁶

Decontamination of an interior space using VHP® is a four-phase process involving preparation of the building interior air (dehumidification), achieving a steady state decontaminant level (conditioning), performing the decontamination, and then aerating for safe reentry (Figure 3.1).³



Dehumidification

Hydrogen peroxide vapor can co-condense with water vapor producing an undesired condensate high in hydrogen peroxide. If ambient conditions are likely to permit condensation – high humidity and/or cold temperatures – this condensation can be prevented by circulating dry, heated air through the interior prior to injection of the hydrogen peroxide vapor. The target humidity level is determined by the concentration of vapor to be injected and the desired steady state concentration for the decontamination. The lower relative humidity permits a higher concentration of hydrogen peroxide without reaching a saturation point. For this study, the maximum relative humidity at start-of-run (prior to introducing decontaminant) was 30%.

Conditioning

During the conditioning phase, the injection of hydrogen peroxide vapor is initiated at a rapid rate to the desired concentration set point without condensation. Once the target concentration is achieved, the injection rate is lowered to maintain the set-point concentrations.

Decontamination

Decontamination is a timed phase dependent on the hydrogen peroxide vapor concentration. In building

and aircraft applications a decontamination timer counts down from the preset decontamination time. If the concentration or temperature values fall below the set point, the timer stops. Stopping the timer ensures that during the decontamination phase, the interior space is exposed to at least the minimum decontamination conditions for the desired exposure time. For this laboratory-scale study, the enclosure VHP® concentration was maintained uninterrupted within the target concentration range.

Aeration

After completion of the decontamination phase, the hydrogen peroxide injection is terminated. Air is introduced into the chamber. The air displaces the hydrogen peroxide. The system is monitored until the hydrogen peroxide concentration falls to a safe level for coupon removal.

4.0

Experimental

The Material Demand testing was conducted in compliance with the Quality Assurance Project and Work Plan (QAPP)⁷ developed under the Quality Management Plans (QMP)^{8,9} and EPA E4 quality system requirements.^{5,10-12}

4.1 Representative Building Material Test Coupons

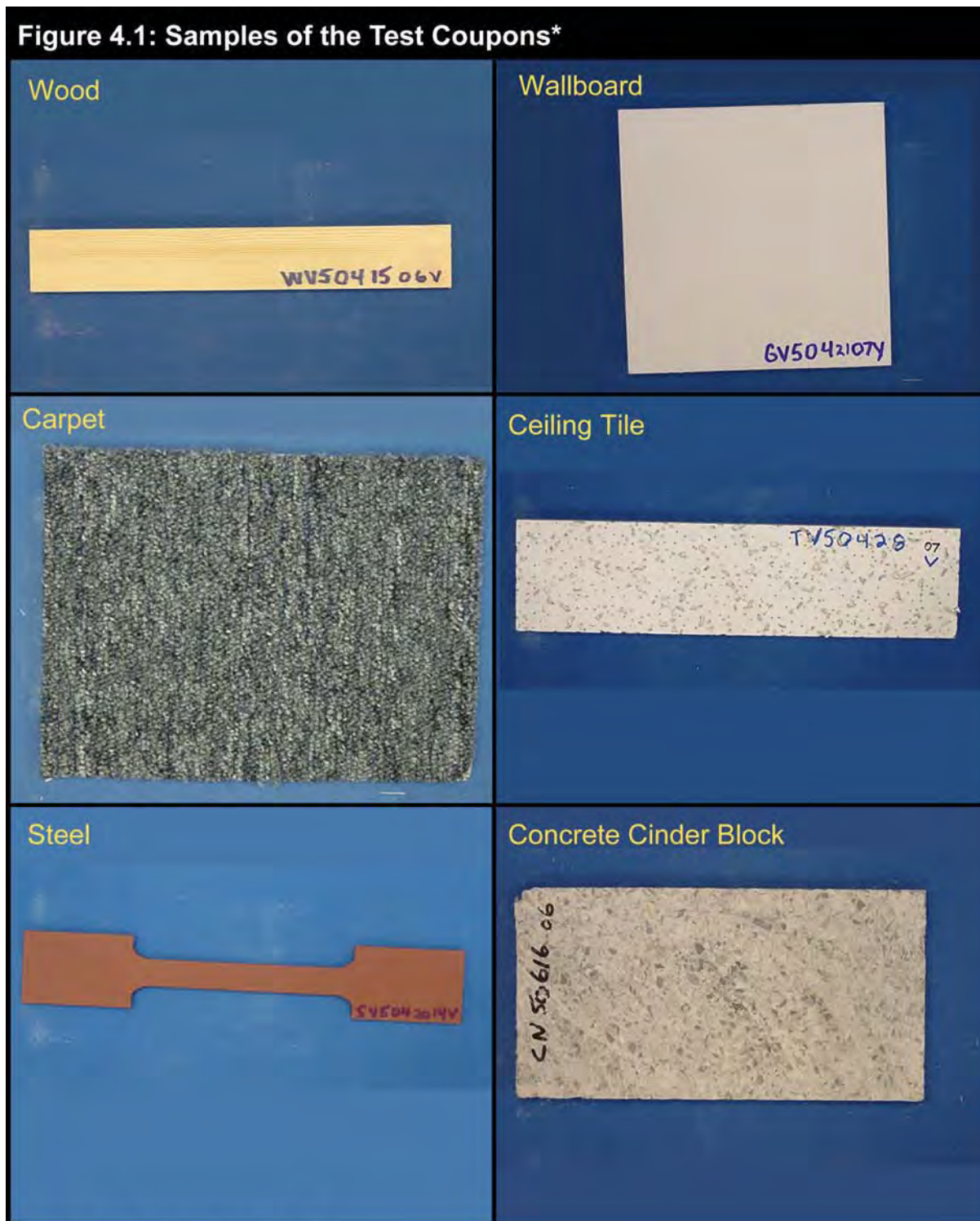
Test coupons were prepared in accordance with the ASTM testing requirements for the Material Compatibility testing.¹³ The coupons were cut from stock material in accordance with the procedure in Appendix B of the QAPP⁷ and reproduced as Appendix A of this report. Coupons were prepared by obtaining a large enough quantity of material that multiple test samples could be obtained with uniform characteristics (e.g., test coupons were all cut from the interior rather than the edge of a large piece of material). The building materials studied, including supplier and coupon dimensions, are provided in Table 4.1 and shown in Figure 4.1.

Chain of Custody (CoC) cards were used to ensure that the test coupons were traceable throughout all

phases of testing. The test coupons were measured and visually inspected prior to testing. Coupons were measured to ensure that the test coupon was within the acceptable tolerances (Appendix A). Coupons were visually inspected for defects and/or damage. Coupon measurements and visual inspection were recorded on the CoC card. Coupons that were not within the allowable size tolerances and/or damaged were discarded. Each coupon was assigned a unique identifier code that matched the coupon with the sample, test parameters, and sampling scheme (Appendix B). The unique identifier code was recorded on the CoC card. The CoC cards followed each sample from Material Demand testing through Material Compatibility testing to disposal.

Table 4.1: Representative Building Interior Materials

Material	Code	Supplier	Length	Width	Thickness
Structural Wood, fir	W	Home Depot	10.0 in	1.5 in	0.5 in
Latex-Painted Gypsum Wallboard	G	Home Depot	6.0 in	6.0 in	0.5 in
Concrete Cinder Block	C	York Supply	4.0 in	8.0 in	1.5 in
Carpet	R	Home Depot	6.0 in	8.0 in	0.0 in
Painted Structural Steel	S	Specialized Metals	12.0 in	2.0 in	0.3 in
			5.3 in	0.8 in	0.3 in
Ceiling Suspension Tile, Acoustical	T	Home Depot	12.0 in	3.0 in	0.6 in



*Coupons are not shown to scale

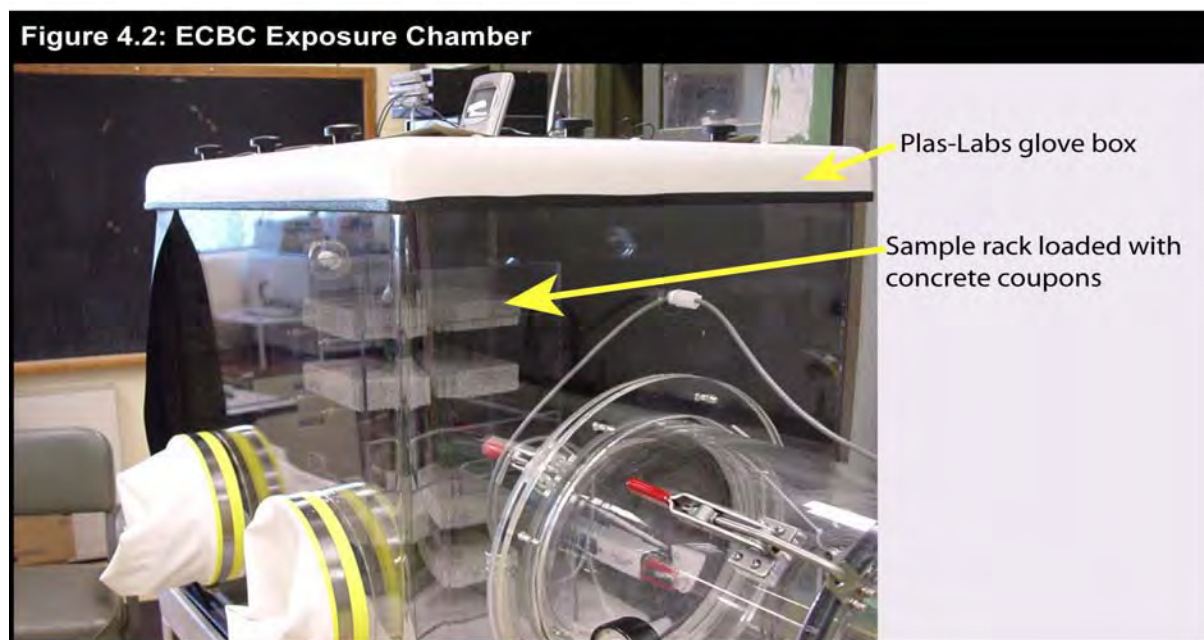
4.2 Hydrogen Peroxide Test Chamber

A Plas-Labs compact glove box (Model 830-ABC) fitted with Hypalon® gloves and glove port plugs was used as the Exposure Chamber (Figure 4.2). The glove box is acrylic with an internal volume of 11.2 cubic feet (28" w x 23" d x 29" h) with an isolated transfer chamber that is 12" long x 11" diameter (I.D.). The chamber was insulated with 0.5-inch thick polyisocyanurate foam insulation (R value 3.3) to help stabilize exposure temperature and minimize possibility of VHP® or water condensation (insulation not shown in Figure 4.2). The chamber insulation blocked exposure of VHP® to light and minimized possible VHP® decomposition. An exposure rack constructed of Lexan® and horizontal stainless steel bars was used to hold the test specimens. The exposure rack was 12" long x 12" wide x 24" tall and had four levels in which to place specimens. Coupons were placed in the glove box in accordance with IOP DS04016 as shown in Appendix B, Figures B.1 and B.2.

The vapor concentration, temperature and relative humidity were recorded each minute during testing. The VHP® concentrations were measured using two Dräger hydrogen peroxide electrochemical sensors (model HC 6809070) coupled with Dräger Polytron 2 transmitters for real time monitoring at the inlet and outlet of the chamber. The sensors were placed in small enclosures attached directly to the inlet and exit ports of the exposure chamber. The inlet detector measured the hydrogen peroxide concentration immediately before entering the enclosure. The hydrogen peroxide

concentration within the chamber was measured by the exit detector immediately after the effluent exits the chamber. The sensors were factory preset to measure from 0 to 4000 ppmv H_2O_2 with sensitivity $\leq \pm 5\%$ of the measured value, but were recalibrated in-line using VHP® concentration values determined by chemical titration of VHP® captured in bubbler solutions. The inlet hydrogen peroxide detector was calibrated to measure from 0 to 800 ppmv H_2O_2 , and the outlet hydrogen peroxide detector was calibrated to measure from 0 to 340 ppmv H_2O_2 IAW IOP DS04001.

Sensor operation was verified during each run using the average value from three iodometric titrations on the VHP® stream entering and exiting the glove box (IOP DS04019). A Vaisala HUMICAP temperature and humidity sensor transmitter (model HMT333) was placed in the center of the chamber. The relative humidity sensor accuracy was $\pm 1\%$ at 0 to 90% RH and $\pm 1.7\%$ at 90 to 100% RH (non-condensing). The temperature sensor operating range was -40°C to 80°C with an accuracy of $\pm 0.20^\circ\text{C}$. The sensor data were collected electronically using a portable data logging system manufactured by Omega Engineering (OMP-MODL). The data were transferred to a PC running the Omega supplied Microsoft Windows based HyperWare software for data plotting, real time trending and initial analysis. An Omega OMP-MLIM-4 expansion module was used to monitor output from the device. Data were collected at a rate of at least one data point per minute.



VHP® was generated using a Steris VHP® M100-S hydrogen peroxide vapor generating system and 35% hydrogen peroxide. Airflow and peroxide solution feed rate were controlled using a Siemens OP-7 interfacing unit. The desired flow rate and peroxide feed rate were entered into the interfacing unit. The VHP concentration ranges for this testing were 250 to 300 ppmv and 125 to 150 ppmv. The bottle containing hydrogen peroxide was not weighed before and after each test so the mass of decontaminant consumed was not recorded. The VHP® decontamination technology requires relatively low humidity conditions to reduce the likelihood of peroxide condensation. The low humidity was maintained by drying the air with a Munters MG90 desiccant dehumidifier before it was fed into the VHP® delivery system. The humidity of the air fed into the VHP® delivery system was < 30% relative humidity at the start of test. The humidity of the air in the test chamber was typically < 15% relative humidity. Exposures were carried out with a minimum 30 °C temperature. Temperature, humidity and hydrogen peroxide concentrations were continuously monitored during the decontamination process. The off gas from the chamber was decomposed and released into a chemical fume hood. VHP® was catalytically decomposed to water and oxygen in a Random Technology catalytic converter containing metal catalysts (platinum and palladium) on an aluminum honeycomb monolith substrate. The chamber was operated IAW SOP RNG-107, IOP DS04015, and IOP DS04016.¹⁴⁻¹⁶ A Dräger Pac III single gas monitor fitted with a Dräger hydrogen peroxide sensor was used to monitor the VHP® outside the chamber in the workspace. The standard measuring range of the VHP® monitor is 0 to 10.0 ppmv H₂O₂ with a display resolution of 0.1 ppmv.

A small recirculation fan was used in the chamber to mimic the air circulation provided by fans in commercial

large room decontamination. Air circulation was observed using a “fog” test of dry ice and warm water rather than a “smoke” test. There was concern that the smoke test might leave residue inside the chamber that could interfere with the coupon studies.

4.3 Material Demand Testing

Each material type was tested independently in three replicate trials at both the target and half-target VHP® concentrations. The number of test materials was dependent on the coupon surface area. The Material Demand test used the appropriate number of coupons so that the total surface area exposed to vapor was essentially the same for each coupon type. The sample surface area was calculated by summing the area for each exposed sample face. For example, the wood surface area is $(2 * l * w) + (2 * l * h) + (2 * w * h)$. The testing was conducted in accordance with the procedures documented in SOP RNG-107 and IOPs DS04015 and DS04016 and as shown in Appendix B, Figure B.1.¹⁴⁻¹⁶ The hydrogen peroxide sensor performance was verified before testing in accordance with IOP DS04015.

Dehumidification

The coupons were exposed to decontaminant in accordance with section 6.0 of the Material Demand QAPP.¹⁷ The coupons were placed in the exposure chamber in accordance with IOP DS04016.¹⁶ The chamber humidity was adjusted below 30% relative humidity using air flow from the dehumidifier prior to the introduction of VHP® into the glove box. The time required to adjust the humidity in the chamber was between 15 to 30 minutes.

Table 4.3 Coupon Exposed Area

Material	Code	Sample Dimensions (cm)			Sample Surface Area* (cm ²)	Coupons per Test	Total Area (cm ²)	Vapor per Sample Area (cm ³ /cm ²)
		length	width	height				
Structural Wood, fir	W	25.4	3.9	1.3	270	18	4863	65.2
Latex-Painted Gypsum	G	15.2	15.2	1.3	539	9	4854	65.3
Concrete	C	10.2	20.3	1.4	495	10	4952	64.0
Carpet	R	15.0	20.0	-----	600	8	4800	66.1
Painted Structural Steel	S	7.8	5.2	0.6	267	18	4798	66.1
		15.0	2.0	0.6				
Ceiling Suspension	T	30.0	8.0	1.4	586	8	4691	67.6

* Sample surface area is calculated for each exposed sample surface

** Volume chamber is 11.2 cubic feet (317,148 cubic cm)

Conditioning

VHP® was introduced into the chamber to reach the target 250 ppmv or half-target 125 ppmv concentration. Once the measured VHP® concentration reached the target concentration, the decontamination phase was started.

Decontamination

The VHP® concentration within the chamber was maintained within the target concentration range of 250- to 300 ppmv or the half-target concentration range of 125- to 150 ppmv. The CT (chamber concentration multiplied by time) values for the target and half-target concentrations were made the same by running the half-target concentration tests for twice the time of the target concentration tests. During Reliability Tests with the VHP® system, the initial residence time of vaporized hydrogen peroxide in the chamber at 0.2 CFM (requested by the EPA) was determined to be longer than the decomposition half-life. The concentration of VHP® at the outlet detector was only 20% of the concentration measured at the inlet detector. After consultation with Steris, the chamber was fitted with larger diameter tubing to allow increased flow through the chamber, and therefore a faster turnover rate. Further characterization/reliability tests (1 to 6 cfm) showed that increasing the flow through the chamber minimized the difference in the VHP® concentrations at the inlet and outlet detectors. The flow rate (3.0 cfm) through the chamber was chosen to optimize for both residence time and decomposition of peroxide. The flow rate was documented in the Quality Assurance Project and Work Plan (QAPP).⁷ The flow rate provided a turnover of approximately sixteen exchanges per hour in the chamber. The flow rate was fixed at 3 cfm for each stage (dehumidification, conditioning, decontamination, and aeration) of the Material Demand tests. The temperature during exposure was kept above the minimum requirement of 30 °C. The hydrogen peroxide sensor performance was verified at least once during each test by comparison to a wet-chemical titration method.

Aeration

Aeration of the chamber was conducted following the decontamination period. The VHP® concentration within the chamber was monitored until end-of-run (EOR). EOR was defined as the reduction of chamber concentration to 10% of the decontamination concentration. For the VHP® studies, EOR was approximately 15 ppmv for half-target or 30 ppmv for target concentration runs. Aeration of the chamber continued until the vapor concentration fell to or below the levels required by the ECBC Risk Reduction Office to assure safe operation for personnel. The procedures for the safe opening of the chamber and coupon removal after fumigant exposure are documented in SOP RNG-

107 and IOP DS04015. Low-level vapor monitors were used for monitoring personnel. A Dräger Pac III single gas monitor fitted with a Dräger hydrogen peroxide sensor was used to monitor the VHP®. The standard measuring range of the VHP® monitor is 0 to 10.0 ppmv H₂O₂ with a display resolution of 0.1 ppmv.

4.4 Data Review and Technical Systems Audits

The approved Material Demand QAPP specified procedures for the review of data and independent technical system audits. All test data were peer reviewed within two weeks of generation. The project quality manager (or designee) was required to audit at least 10% of the data. In addition, the project quality manager (or designee) performed four technical system audits over the course of testing. A technical system audit is a thorough, systematic, on-site qualitative audit of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management and reporting aspects of the system. The QA findings are documented in Section 8.0.

4.5 Material Demand Calculation

The difference in the target chamber CT (CT_{outlet}^* in ppmv-hr) and the inlet CT (CT_{inlet}^* ppmv-hr) required to achieve the target (1000 ppmv-hr) can be attributed to the demand of the material in the chamber for VHP®. This demand is comprised of reversible adsorption (e.g., physisorption) and chemical reaction (e.g., decomposition or chemisorption) on the surfaces within the chamber. A contribution of homogeneous decomposition may also be present; however, efforts were made to minimize the contribution of this mechanism (e.g., rapid turnover rate and shielding from UV). A correction must be made for the hydrogen peroxide remaining in the chamber at the end of the fumigation period. This correction factor, CT_{charge}^* was determined by multiplying the volume of the chamber by the concentration in the chamber at the end of the fumigation period and converting it into terms of ppm-hrs by Equation 3.

The impact of each material on the required CT ($CT_{diff,k}$) can be determined by subtracting the observed difference in CT in the baseline tests ($CT_{diff,b}$) from that observed with a specific material type in the chamber ($CT_{diff,mb}$), shown in Equation 1. It is important to note that while during a fumigation in the field the CT is generally not calculated until the target concentration has been reached, for the purposes of this research the calculated CT begins at the time injection is started in order to account for any sorption occurring prior to reaching the target concentration.

$$CT_{diff,k} = CT_{diff,mb} - CT_{diff,b} = (CT_{outlet} - CT_{inlet} - CT_{charge})_{mb} - (CT_{outlet} - CT_{inlet} - CT_{charge})_b \quad (1)$$

The time and surface area specific material demand over the fumigation period (up to 1000 ppmv-hr) can be calculated according to Equation 2 where $CT_{diff,k}$ is divided by the material surface area (A , in m^2) and the time required to reach the target CT_{outlet} (t , in hr). The units of MD are ppmv-hr per hr per m^2 . The total surface area added to the chamber for each material type is reported in Table 4.3. The total interior surface area of the chamber and material support structures was $3.8 m^2$.

$$MD_k = -\frac{CT_{diff,k}}{tA} \text{ (for materials), } MD_b = -\frac{CT_{diff,b}}{tA} \text{ (for baseline)} \quad (2)$$

The calculation of the material demand via Equation 2 provides for a determination of the relative effect of each material on the chamber VHP[®] concentration.

The Material Demand is also reported in $g/m^2/h$ for the chamber (Table 5.2) and the materials (Table 6.1.1 and Table 6.1.2). The mass of VHP[®] decomposed or sorbed by a specific material, $M_{H_2O_2,k}$ (g), was calculated from the $CT_{diff,mb}$ and $CT_{diff,b}$ by Equation 3.

$$M_{H_2O_2} = \frac{CT_{ppmv-hr} MW_{H_2O_2} P_{sys}}{1000RT_{sys}} F_{total} \quad (3)$$

Where:

$MW_{H_2O_2}$ is the molecular weight of hydrogen peroxide, 34 g/g-mole

P_{sys} is the chamber pressure in atmospheres

T_{sys} is the chamber temperature in K

F_{total} is the flow rate through the chamber, 5097 L/hr

R is the universal gas constant (0.0826 L atm/g-mole K)

The mass flux for the baseline tests, J_b ($g/m^2/h$) was then calculated using Equation 4.

$$J_b = M_{H_2O_2,b} / tA \quad (4)$$

The mass flux for each material, J_k ($g/m^2/h$), was calculated by Equation 5.

$$J_k = (M_{H_2O_2,mb} - M_{H_2O_2,b}) / tA \quad (5)$$

The baseline correction was required since $CT_{diff,mb}$ included homogeneous decomposition and the material demand of the interior of the exposure chamber and the material

The average and standard deviation of each specific test were calculated using Microsoft Office Excel (2003) SP2 software. The determination of statistical outliers was performed according to the Grubb's test, also known as the extreme studentized deviate (ESD) method. No data was discarded as an outlier within a data set (i.e., set of triplicate experiments at each concentration for each material).

5.0

Evaluation of Empty Chamber

5.1 “Fog” Test Results and Discussion

A “Fog” test was conducted to observe the chamber air circulation pattern created by the chamber recirculation fan. The test was conducted with an air flow of 3.0 cfm through the chamber. The small recirculation fan was used in the chamber to mimic the air circulation provided by fans in commercial large room decontamination. The fan was placed on the bottom of the chamber in the back right corner. The fan blew toward the opposite corner of the chamber. The “Fog” test was used to verify that the coupons placed on the exposure rack had contact with decontaminant vapor during testing. A container of dry ice and warm water was placed in the chamber. The fog produced could be sustained for several minutes. Air was introduced into the chamber on the lower right side of the chamber and the flow observed. Figure 5.1 shows

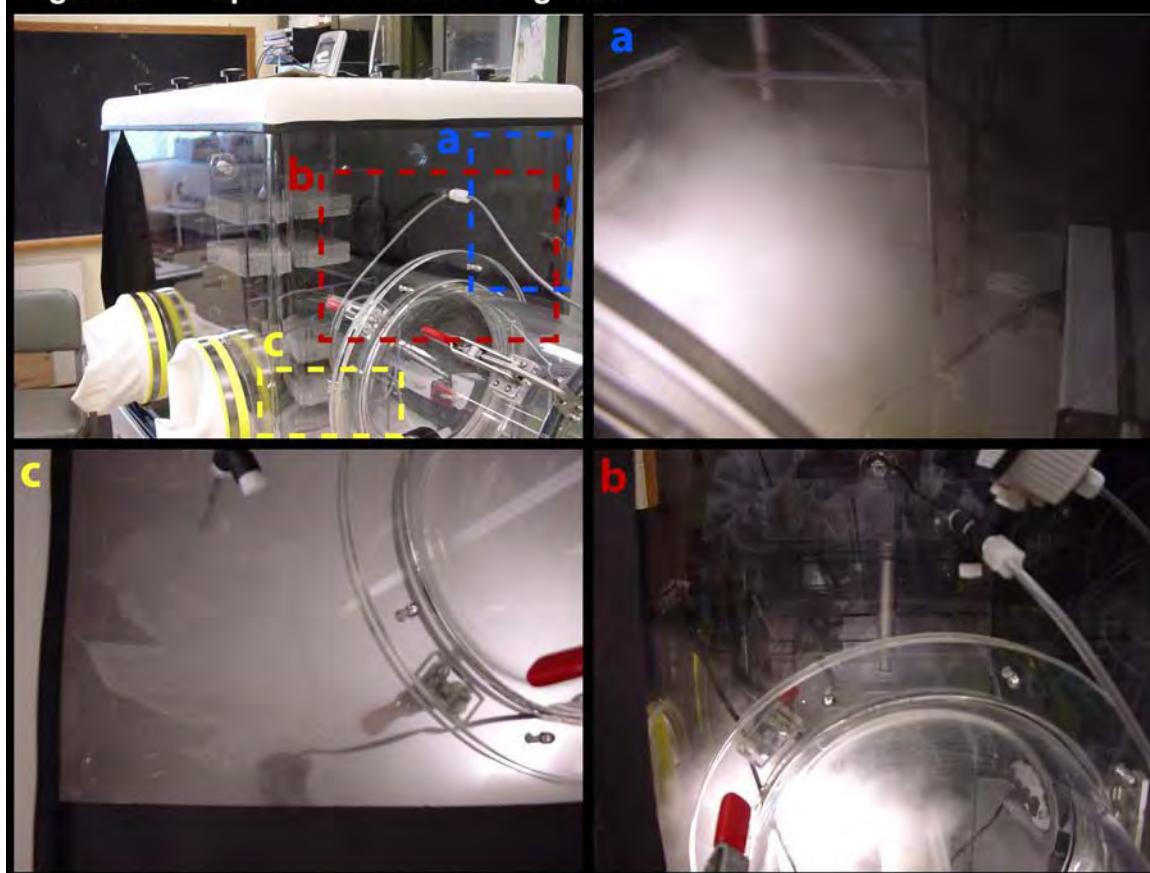
the photographs taken of the fog test within the exposure chamber. The density of the fog was hard to photograph; however, the fog developed an even density and did not stratify.

5.2 Baseline Tests and Results

Three baseline tests were conducted at the target and half-target concentrations for the determination of VHP[®] loss due to spontaneous decomposition and/or adsorption and decomposition from the chamber interior surfaces. The results of those tests are presented in Table 5.2. No coupons were used in the baseline tests. The sample rack was in the chamber during the baseline tests. The air flow rate during the baseline tests was 3.0 cfm.

The hydrogen peroxide sensor performance was verified during testing using a wet-chemical titration procedure.

Figure 5.1: Exposure Chamber Fog Test



For the first three test runs, the titration was performed at the beginning-, middle- and end-of-run. After the first three tests, the titration was performed during the first half of the run. Three replicate samples were collected and titrated. The average agreement between the hydrogen peroxide inlet sensor reading and titration results for the nine titration tests performed for the target and half-target VHP® runs was 4.2% and 4.8%, respectively. The average agreement between the hydrogen peroxide outlet sensor reading and titration

results for the nine titration tests performed for the target and half-target VHP® runs was 3.5% and 3.8%, respectively.

Zero time on all graphs signifies the time where the VHP® concentration within the enclosure first reached the minimum value of the concentration range; either 250- or 125 ppmv (Figure 5.2.1). Based on the four step VHP® process, zero time is the start of the decontamination phase.

Figure 5.2.1: Illustration of the Determination of Zero Time

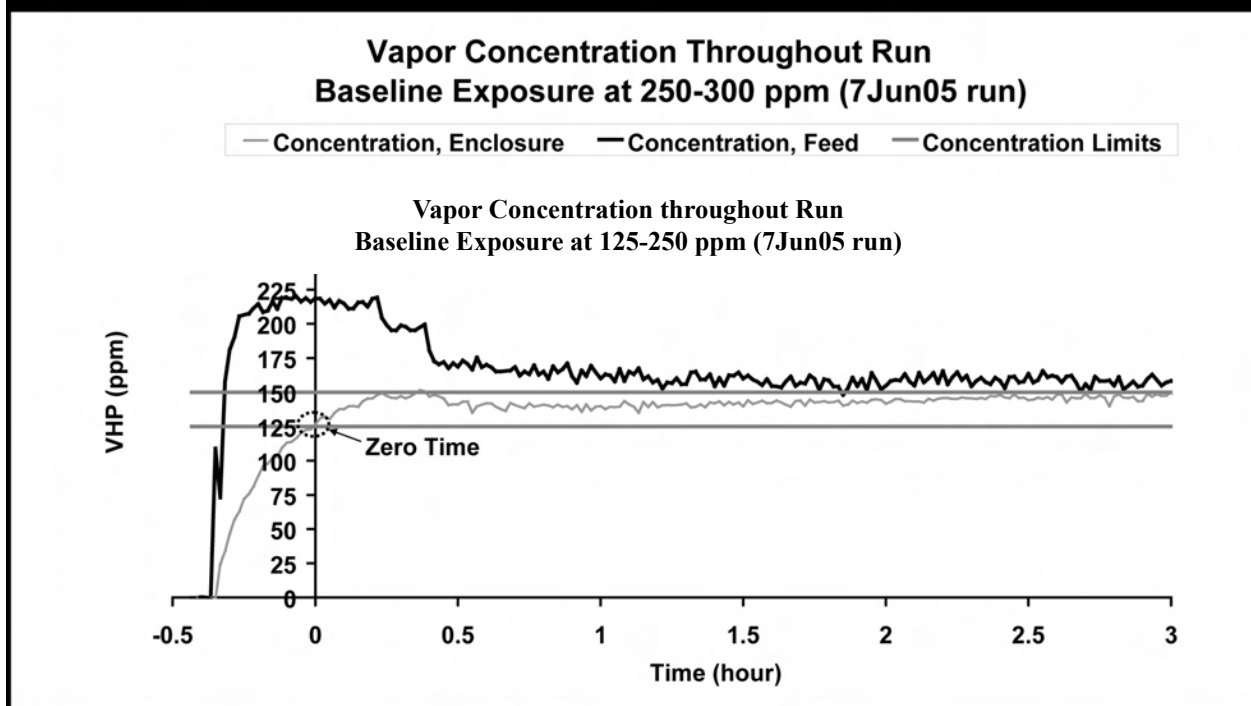


Table 5.2 Baseline Material Demand Test Results

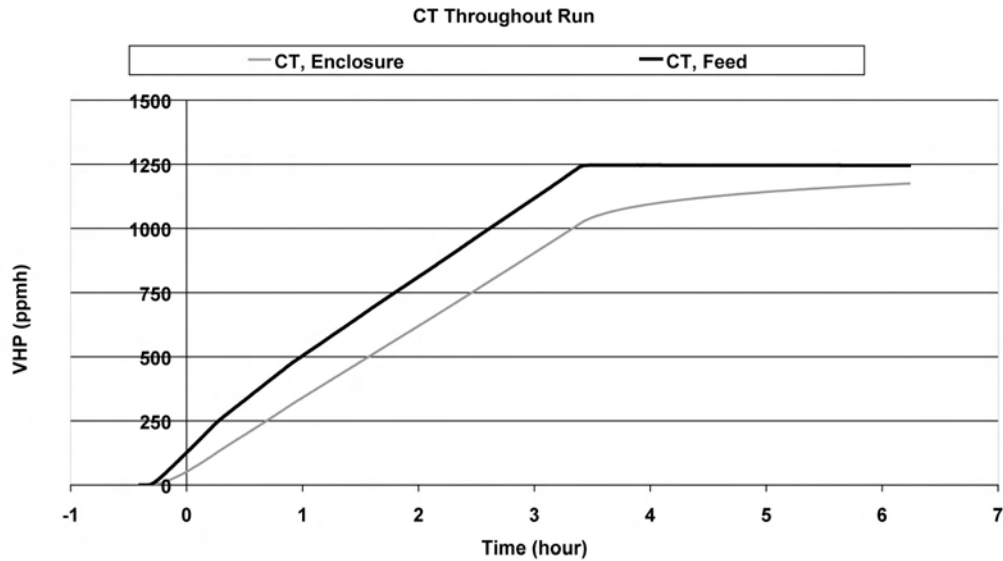
Test	Average Chamber	Average Feed	Time to reach target CT (hr)	DCT (ppmv-hr)	MD (ppmv-hr/hr/m ²)	J _b (g/hr/m ²)
Baseline (125-150 ppmv)	32.5 ± 1.3	151.2 ± 3.2	7.47 ± 0.16	128.3 ± 4.6	4.18 ± 0.16	0.0272 ± 0.0014
Baseline (250-300 ppmv)	32.8 ± 0.1	326.4 ± 6.7	3.73 ± 0.08	199.2 ± 9.8	13.90 ± 0.66	0.0896 ± 0.0046

Representative VHP® 125- and 250 ppmv baseline test CT graphs are shown in Figure 5.2.2. The CT within the chamber and the CT of the feed air are shown in gray and black, respectively. The feed concentration was adjusted as needed to maintain the chamber

concentration within the target concentration range (Figure 5.2.3).

Figure 5.2.2: Baseline VHP Exposure Tests

a) CT of Baseline Exposure at 250-300 ppm (23May05 run)



b) CT of Baseline Exposure at 125-150 ppm (7Jun05 run)

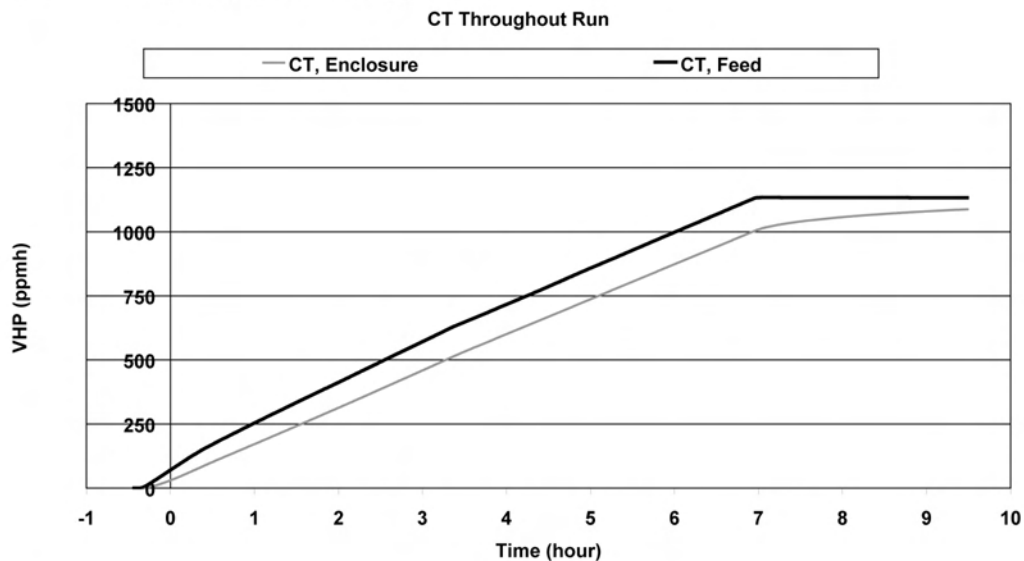
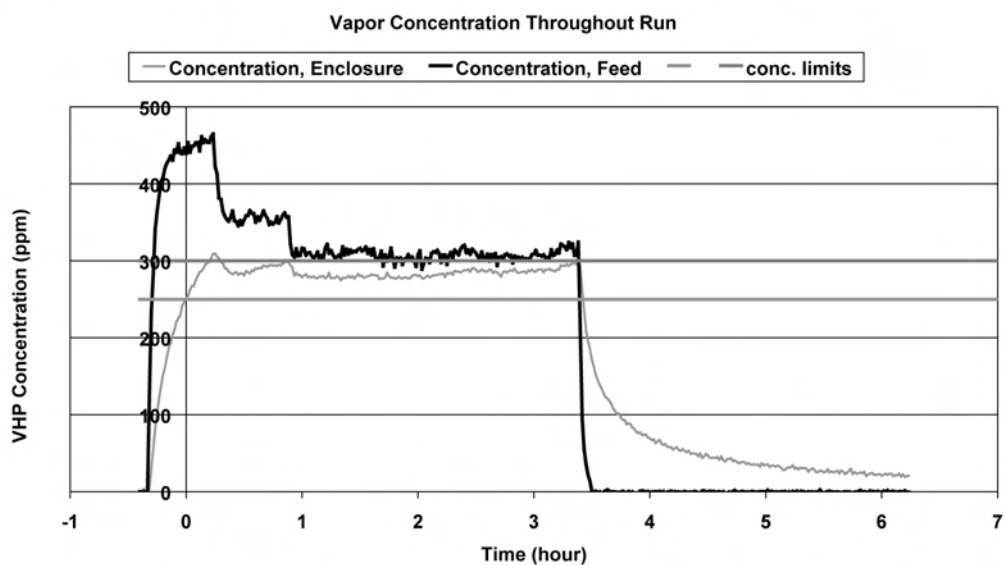
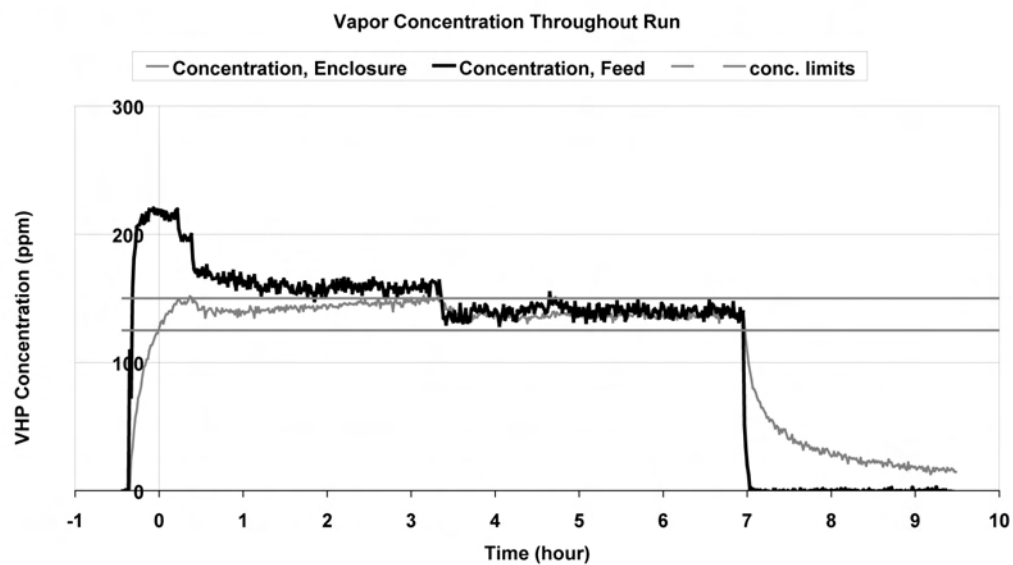


Figure 5.2.3: Concentration Profile for Baseline VHP Exposure Tests

a) Concentration Profile for Baseline Exposure at 250-300 ppm (23May05 run)



b) Concentration Profile for Baseline Exposure at 125-150 ppm (7Jun05 run)



5.3 Baseline Test Discussion

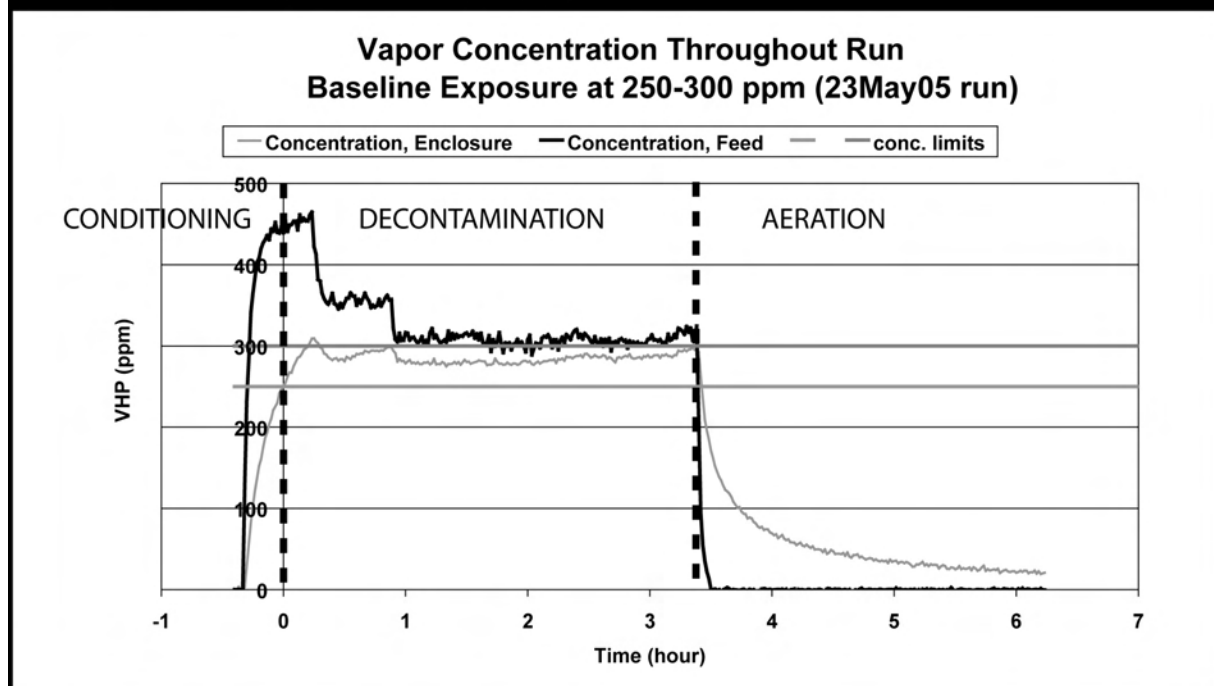
The baseline test results showed minimal hydrogen peroxide loss due to spontaneous decomposition and/or adsorption and decomposition from the chamber interior surfaces at 3 cfm. The hydrogen peroxide sensor performance showed good agreement with the wet-chemical titration results.

The dehumidification step was conducted prior to the start of data collection. Data collection began with the introduction of fumigant into the chamber during the conditioning phase. Once the VHP® concentration reached the target concentration, the feed rate was reduced and the decontamination phase began. In the field, the cumulative CT calculation would not begin until this point. However, in this study the CT was calculated at the start of hydrogen peroxide injection into the chamber, resulting in an exposure less than what would be experienced in the field. An attempt to correct this discrepancy by extrapolating the data out to a true CT of 1000 ppmv-h, showed that the difference was not statistically significant. Because the difference was insignificant the results reported in this document are based on starting the CT calculation at the start of injection. The feed concentration was reduced to zero once the target CT had been reached by immediately stopping the liquid peroxide injection into the VHP® generator. Once the liquid peroxide injection was terminated, the decontamination phase ended and the

aeration phase began (Figure 5.3.1). The immediate termination of liquid hydrogen peroxide injection resulted in the sharp flattening of the feed CT curve (Figure 5.2.2). The enclosure CT did not immediately flatten. The enclosure CT continued to rise at a slower rate after reaching 1000 ppmv-h due to flow rate and chamber volume. The enclosure hydrogen peroxide concentration decreased as the VHP® was diluted with the input air.

Data for CT curves were collected until the VHP® concentrations within the chambers dropped to $\leq 10\%$ of the decontamination concentration. The difference between the feed CT and enclosure CT curves is due to loss of VHP® within the enclosure. Loss of VHP® during the baseline test could result from spontaneous VHP® decomposition, VHP® decomposition on chamber surfaces and/or surface sorption. To minimize potential loss due to condensation, the chamber, sensor enclosures and tubing were wrapped with insulation and maintained above 30 °C. Similarly, potential losses due to light exposure can also be neglected. The chamber, sensor enclosures and tubing were either opaque or wrapped with opaque insulation.

Figure 5.3.1: VHP Process using a Baseline Test



Evaluation of Building Materials

6.1 Results

The VHP® Material Demand chamber exposure tests were conducted from February through June 2005. The exposure chamber temperature profile was maintained within a small range of 30 – 35 °C throughout testing (Figure 6.1.1).

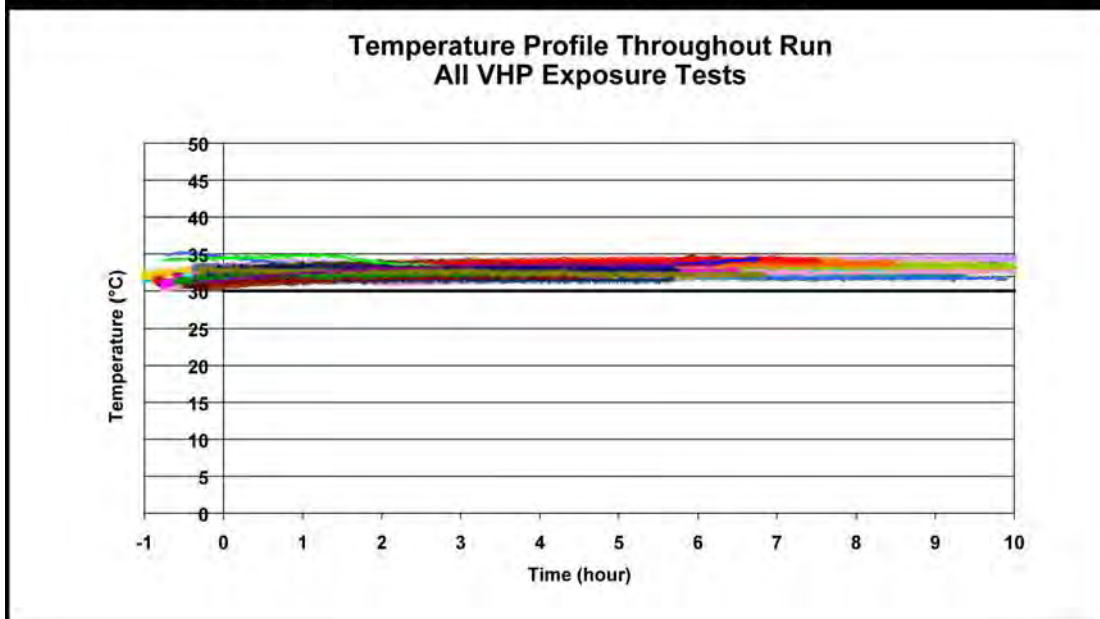
The Vaisala HUMICAP humidity sensor (model HMT333) read slightly high in the presence of VHP® at approximately 300 ppmv, but when removed from air containing VHP® the sensors responded normally with no change in response. Since the relative humidity constraint was for the condition of the chamber prior to the introduction of the fumigant, the condition was satisfactorily met with the sensors. Additional evaluation of the relative humidity sensors is provided in Section 8.1.

The hydrogen peroxide sensor performance was also verified on each material exposure run using the wet-chemical titration procedure. The titration was performed during the first half of each run. Three replicate samples were collected and titrated. The average agreement between the hydrogen peroxide inlet sensor reading and titration results for the eighteen titration tests performed for the target and half-target VHP runs was 6.5% and

3.2%, respectively. The average agreement between the hydrogen peroxide outlet sensor reading and titration results for the eighteen titration tests performed for the target and half-target VHP runs was 5.4% and 4.2%, respectively. The hydrogen peroxide sensor performance verification procedure conducted during each run showed that both sensors were not adversely affected by prolonged and repeated exposure to VHP®. Neither of the two sensors showed visual evidence of deterioration or change in response during the testing period.

Representative CT graphs for each of the test materials are shown in Figures 6.1.3 through 6.1.8. Each graph consists of two CT curves. The enclosure CT (gray line) reflects the hydrogen peroxide CT within the chamber. The enclosure CT determined the test run duration. The concentration of the enclosure was maintained during the decontamination phase within either the target or half-target concentration range. The feed CT (black line) shows the hydrogen peroxide CT from the generator. During the decontamination phase, the generator feed concentration was adjusted to maintain the chamber within the target or half-target concentration range.

Figure 6.1.1: Chamber Temperature Profile Throughout Testing



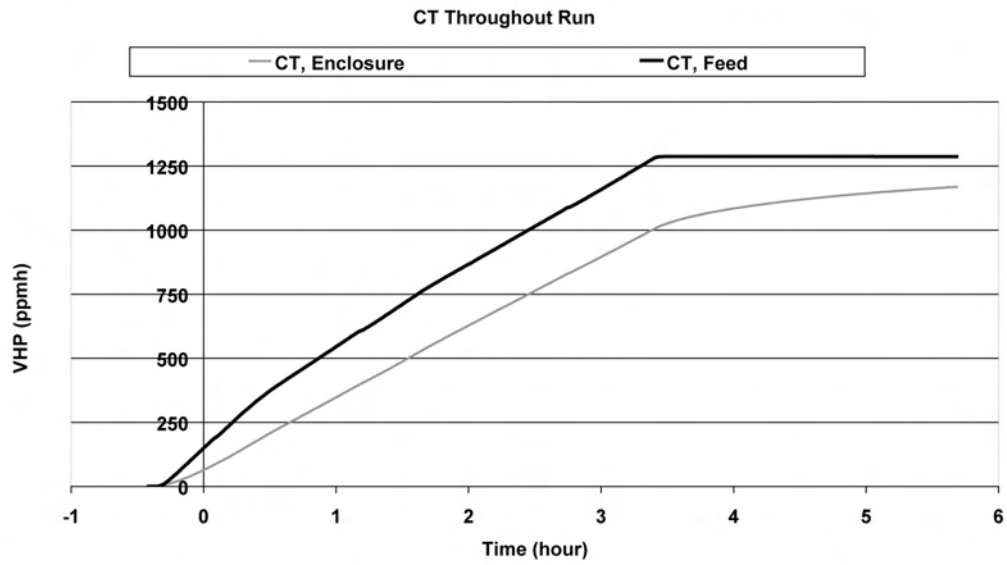
The Material Demand contributions attributable to each of the building materials are shown in Tables 6.1.1 and 6.1.2. The materials are listed in order of their ability to decrease the VHP® concentration in the 125-150 ppmv exposures: carpet, painted structural steel, latex painted wallboard, ceiling tile, wood, and concrete cinder block. The order is similar for the 250-300 ppmv test exposures with the exception of ceiling tile and wood, which are reversed for these exposures. The values shown are the average of three replicate exposures. Concrete cinder block had, by far, the greatest effect of the materials studied under both concentrations.

Representative concentration profile graphs for each of the test materials are shown in Figures 6.1.9 through 6.1.14. Each graph consists of two concentration profiles. The enclosure concentration (gray line) reflects the hydrogen peroxide concentration within the chamber

during the duration of the test. The concentration of the enclosure was maintained during the decontamination phase within either the target or half-target concentration range. The feed concentration (black line) shows the hydrogen peroxide concentration exiting the generator. During the decontamination phase, the generator feed concentration was adjusted to maintain the chamber within the target or half-target concentration range. Figure 6.1.12a shows small dips in the measured concentration. The cause of the occasional dip was a small air bubble that was drawn into the vaporizer feed tube from the hydrogen peroxide solution.

Figure 6.1.3: Representative VHP Test Results for Carpet

a) CT of Carpet VHP Exposure at 250-300 ppm (2May05 run)



b) CT of Carpet VHP Exposure at 125-150 ppm (20May05 run)

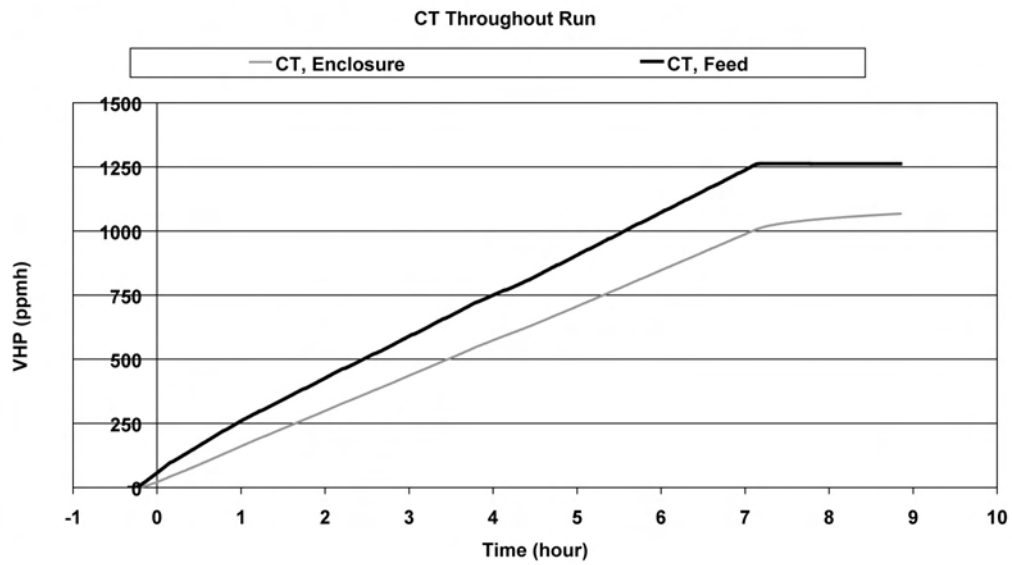
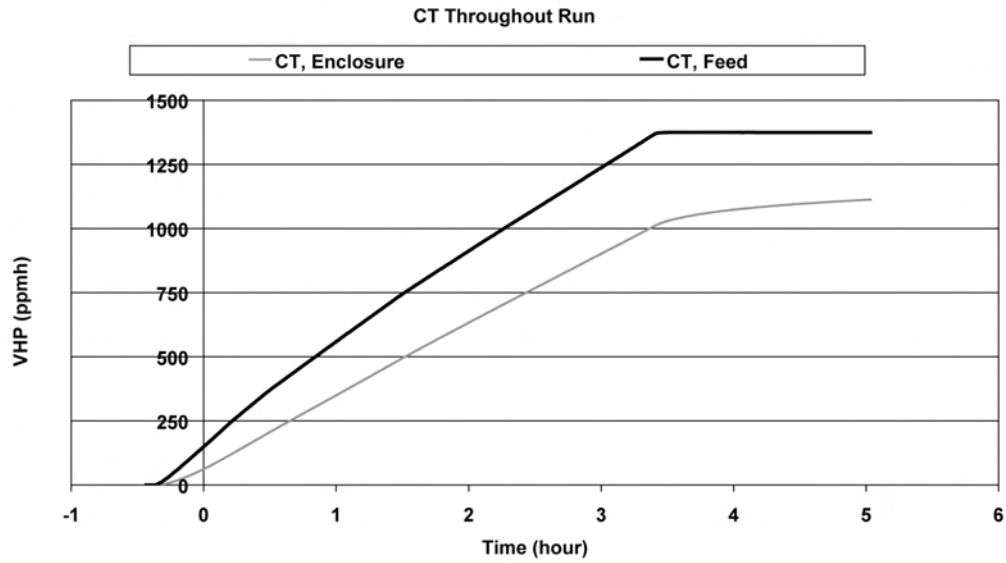


Figure 6.1.4: Representative VHP Test Results for Steel

a) CT of Steel VHP Exposure at 250-300 ppm (18May05 run)



b) CT of Steel VHP Exposure at 125-150 ppm (6Jun05 run)

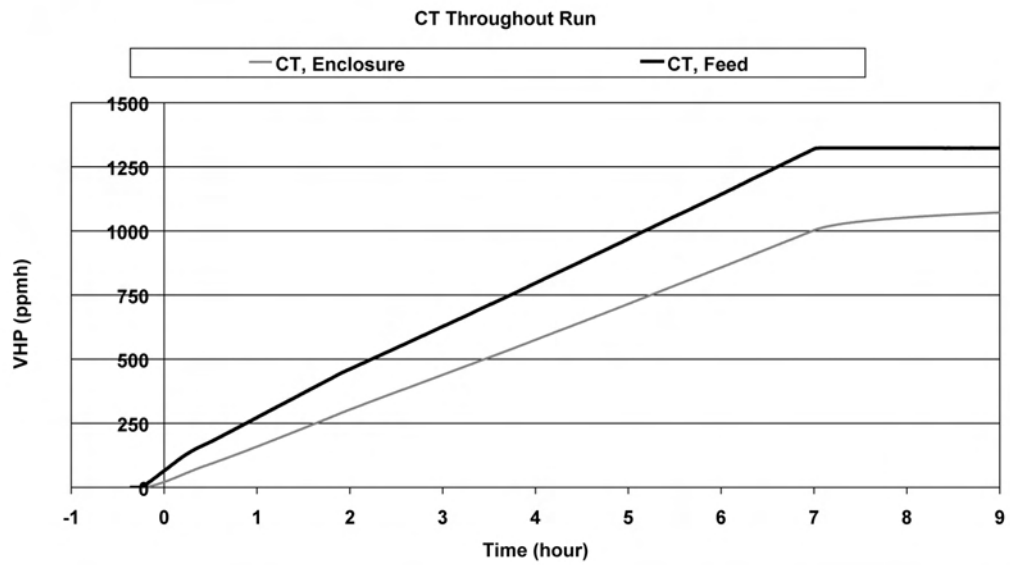
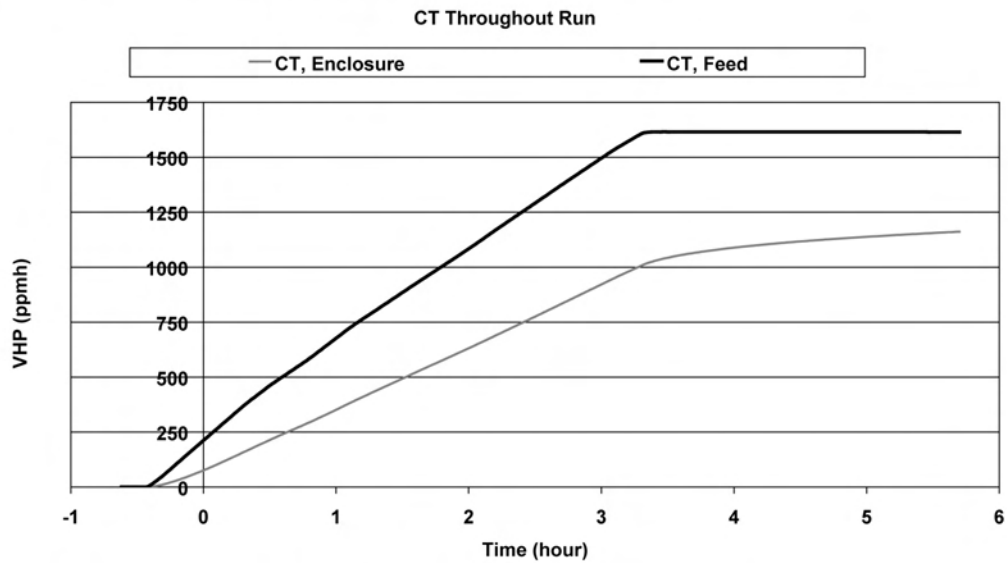


Figure 6.1.5: Representative VHP Test Results for Painted Gypsum Wallboard

a) CT of Gypsum Wallboard Exposure at 250-300 ppm (6May05 run)



b) CT of Gypsum Wallboard VHP Exposure at 125-150 ppm (31May05 run)

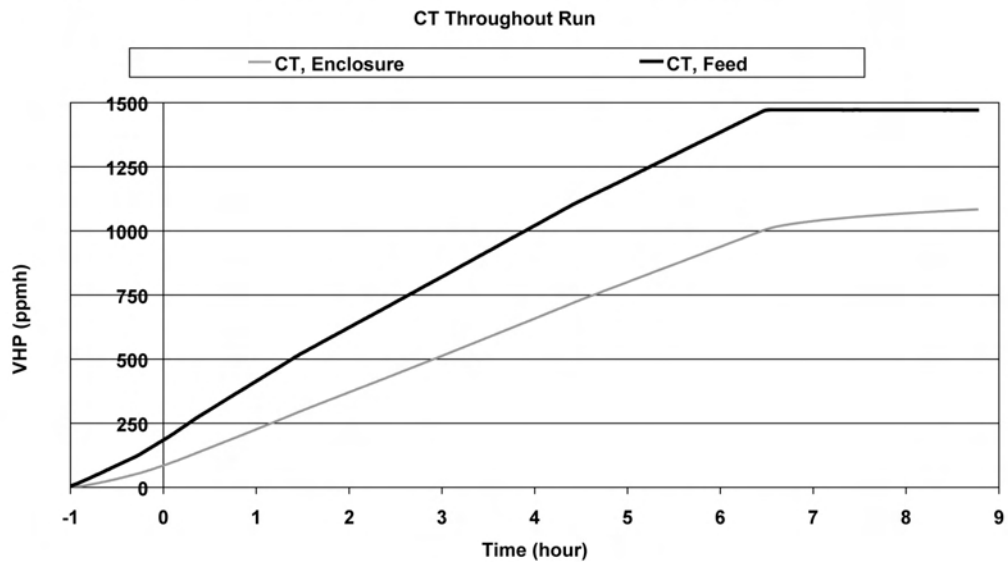
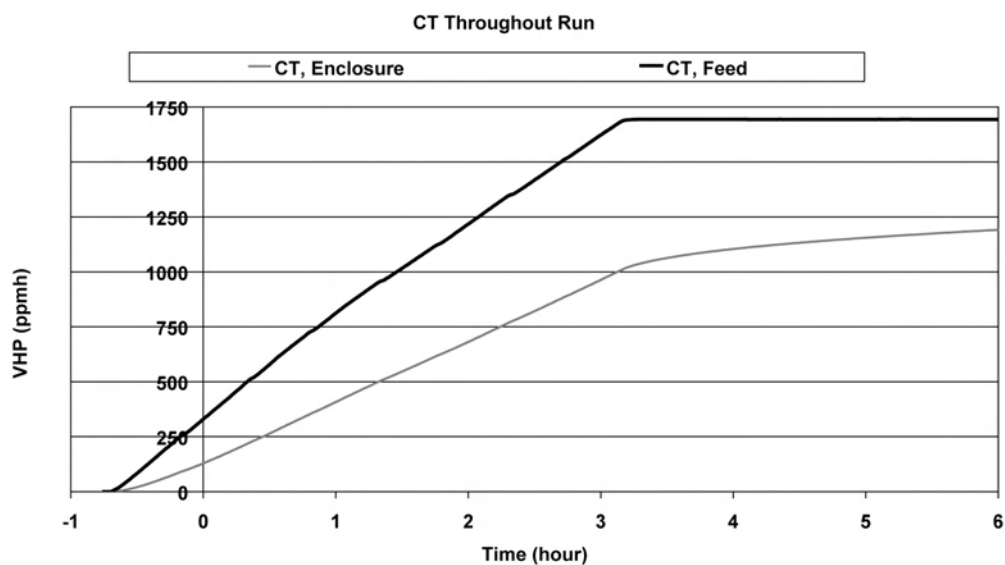


Figure 6.1.6: Representative VHP Test Results for Acoustical Ceiling Tile

a) CT of Acoustical Ceiling Tile VHP Exposure at 250-300 ppm (28Apr05 run)



b) CT of Acoustical Ceiling Tile VHP Exposure at 125-150 ppm (1Jun05 run)

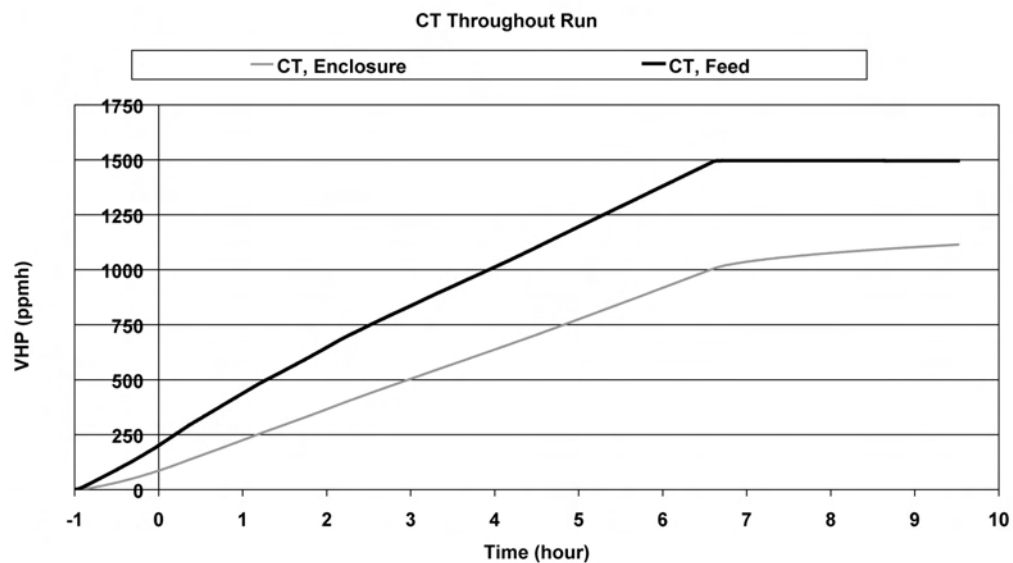
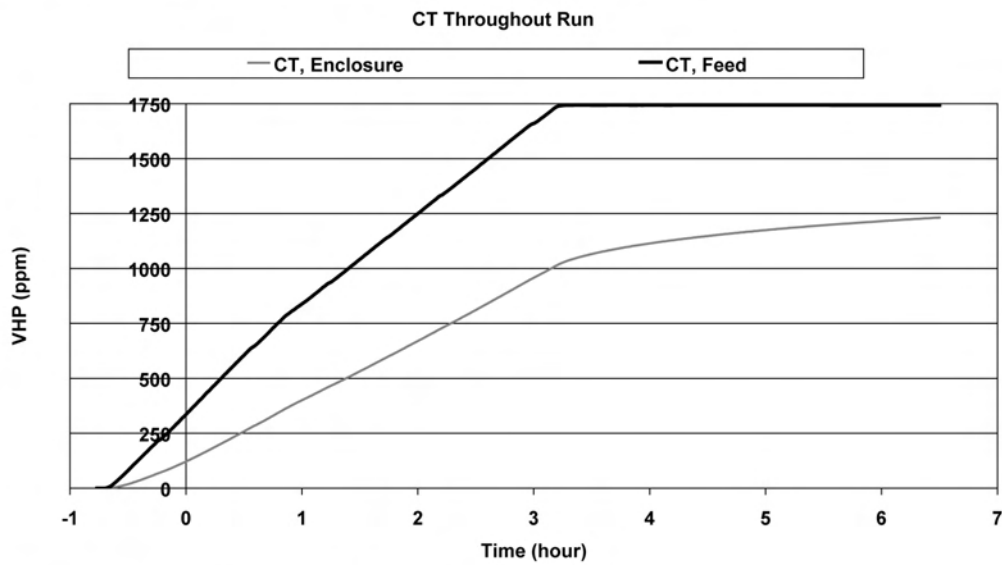


Figure 6.1.7: Representative VHP Test Results for Wood

a) CT of Wood VHP Exposure at 250-300 ppm (26Apr05 run)



b) CT of Wood VHP Exposure at 125-150 ppm (20May05 run)

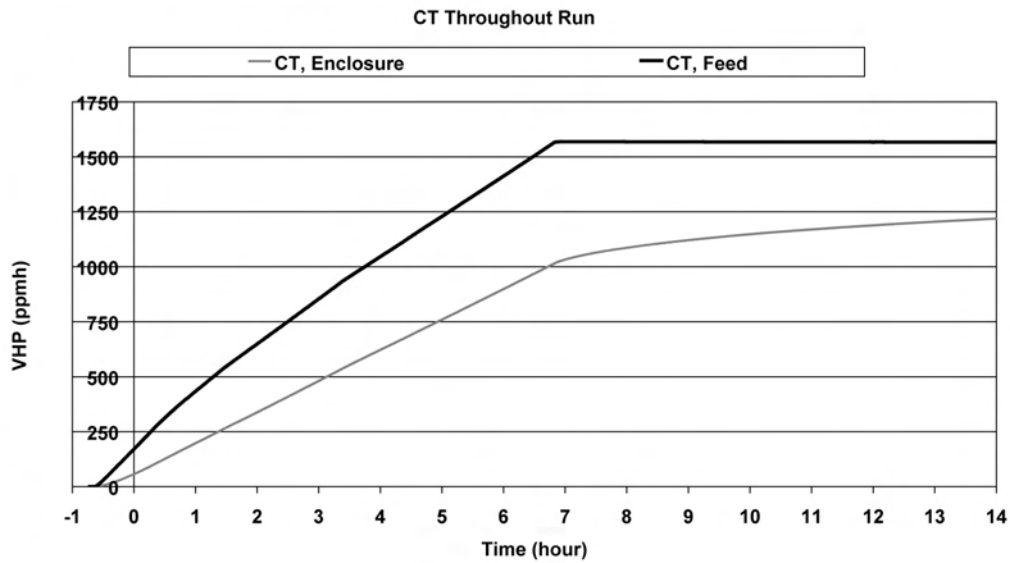
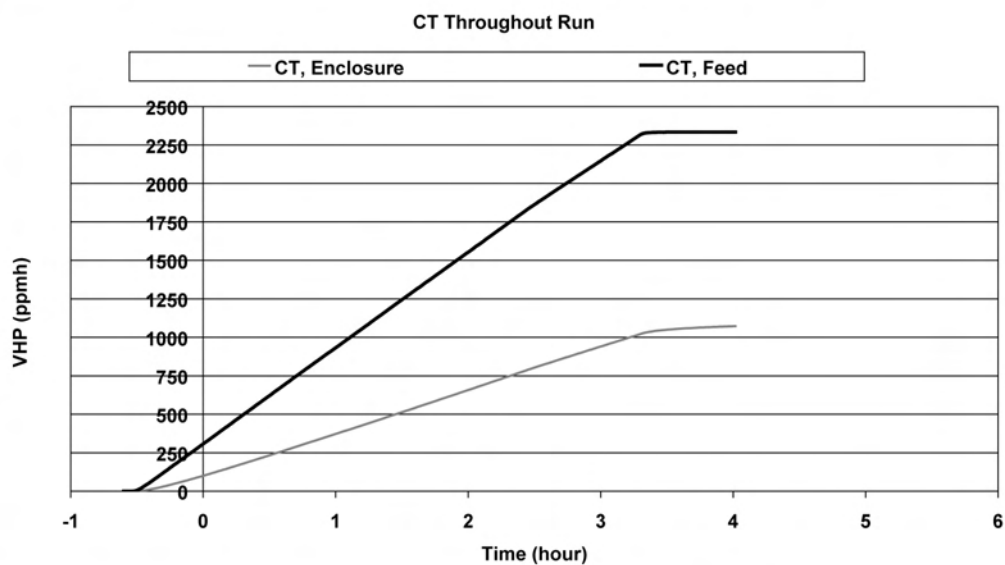


Figure 6.1.8: Representative VHP Test Results for Concrete Cinder Block

a) CT of Concrete Cinder Block VHP Exposure at 250-300 ppm (4May05 run)



b) CT of Concrete Cinder Block VHP Exposure at 125-150 ppm (2Jun05 run)

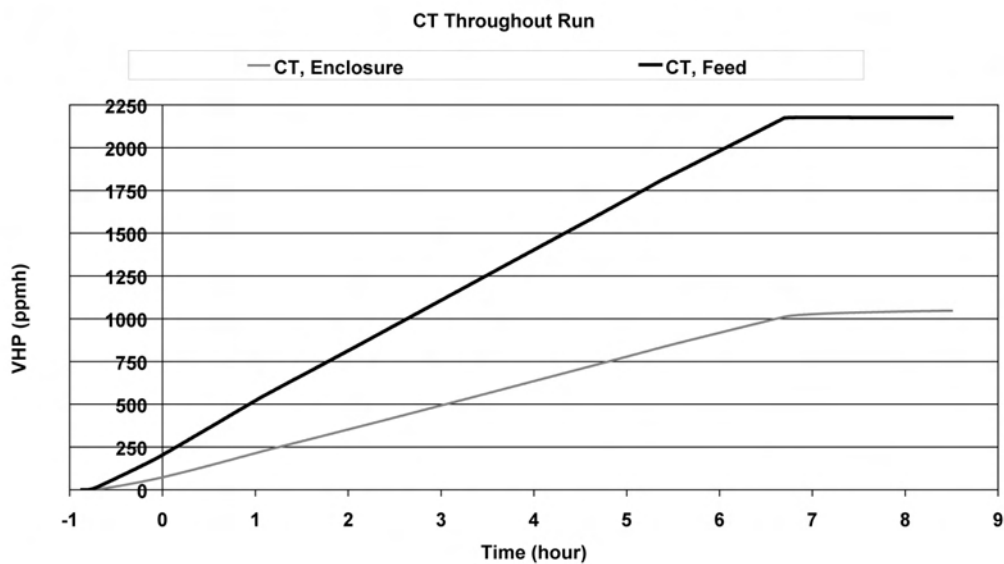


Table 6.1.1 Material Demand Results for Building Materials (125-150 ppmv)

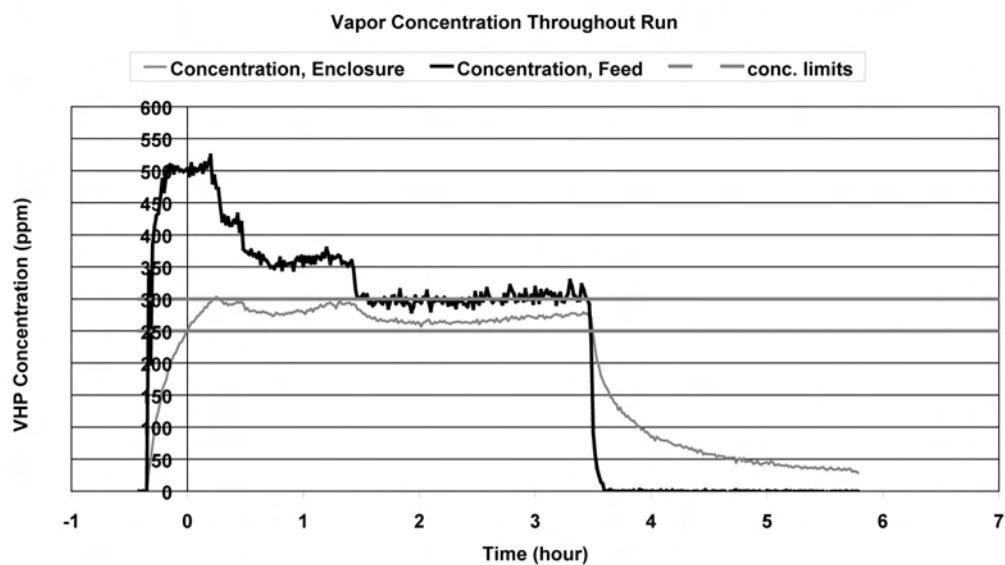
Test	Average Chamber Temperature (°C)	Average Feed Concentration (ppm)	Time to reach target CT (hr)	DCT (ppmv-hr)	MD (ppmv-hr/hr/m ²)	J (g/hr/m ²)
Carpet	33.0 ± 0.7	163.4 ± 3.2	7.29 ± 0.14	62.4 ± 6.0	17.83 ± 1.40	0.111 ± 0.016
Steel	33.2 ± 0.5	174.4 ± 6.4	7.36 ± 0.13	154.8 ± 31.3	43.89 ± 9.35	0.274 ± 0.054
Wallboard	33.1 ± 0.1	197.5 ± 2.6	7.42 ± 0.07	336.0 ± 14.5	93.33 ± 4.06	0.591 ± 0.024
Ceiling Tile	33.1 ± 1.1	198.7 ± 4.6	7.51 ± 0.22	363.2 ± 14.5	103.07 ± 2.41	0.653 ± 0.041
Wood	32.8 ± 0.3	206.1 ± 7.1	7.41 ± 0.04	398.9 ± 49.8	110.69 ± 13.97	0.709 ± 0.083
Concrete	33.5 ± 0.2	299.2 ± 9.8	7.43 ± 0.03	1095.6 ± 82.7	297.58 ± 21.13	1.870 ± 0.123

Table 6.1.2 Material Demand Results for Building Materials (250-300 ppmv)

Test	Average Chamber Temperature (°C)	Average Feed Concentration (ppm)	Time to reach target CT (hr)	DCT (ppmv-hr)	MD (ppmv-hr/hr/m ²)	J (g/hr/m ²)
Carpet	31.9 ± 0.7	344.0 ± 5.1	3.79 ± 0.03	87.6 ± 20.8	48.15 ± 11.34	0.338 ± 0.073
Steel	33.1 ± 0.6	358.1 ± 3.3	3.76 ± 0.03	130.8 ± 21.2	72.44 ± 11.33	0.455 ± 0.050
Wallboard	32.2 ± 0.4	429.1 ± 3.9	3.73 ± 0.01	383.7 ± 18.7	212.03 ± 9.86	1.403 ± 0.075
Ceiling Tile	33.2 ± 0.8	436.4 ± 3.8	3.82 ± 0.02	451.3 ± 7.5	251.69 ± 4.87	1.591 ± 0.062
Wood	32.3 ± 0.2	439.9 ± 7.9	3.83 ± 0.04	466.9 ± 48.1	250.64 ± 23.28	1.650 ± 0.162
Concrete	32.2 ± 0.3	594.5 ± 10.2	3.77 ± 0.06	1022.9 ± 46.1	548.32 ± 21.07	3.612 ± 0.110

Figure 6.1.9: Representative Concentration Profile Results for Carpet

a) Concentration Profile for Carpet, VHP Exposure at 250-300 ppm (29Apr05 run)



b) Concentration Profile for of Carpet, VHP Exposure at 125-150 ppm (20May05 run)

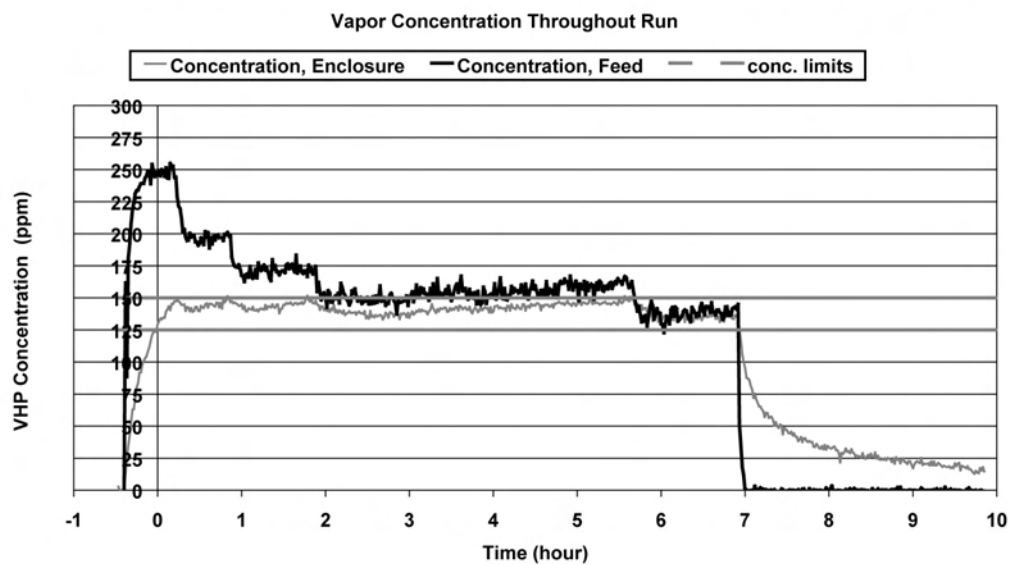
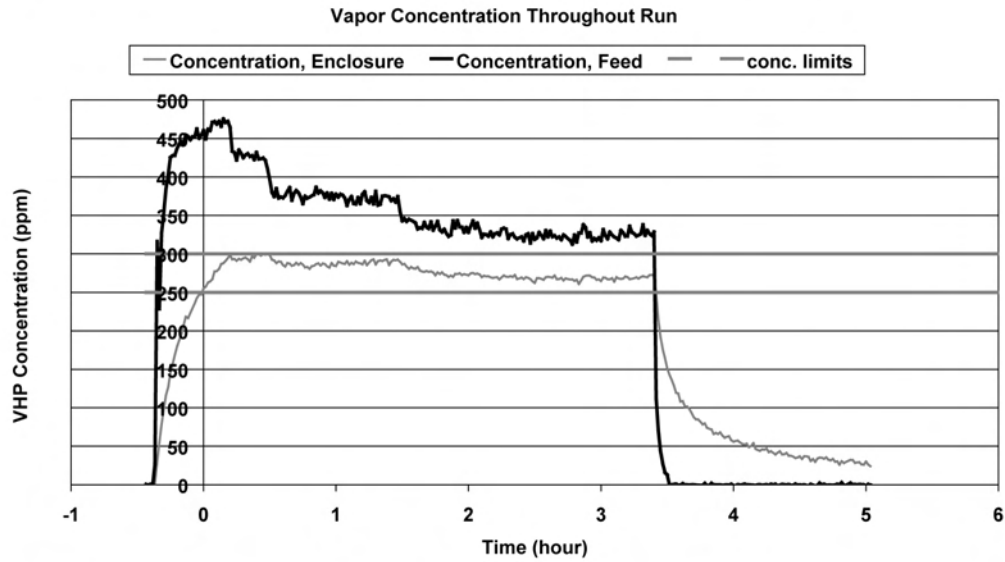


Figure 6.1.10: Representative Concentration Profile Results for Steel

a) Concentration Profile for Steel, VHP Exposure at 250-300 ppm (18May05 run)



b) Concentration Profile for Steel, VHP Exposure at 125-150 ppm (6Jun05 run)

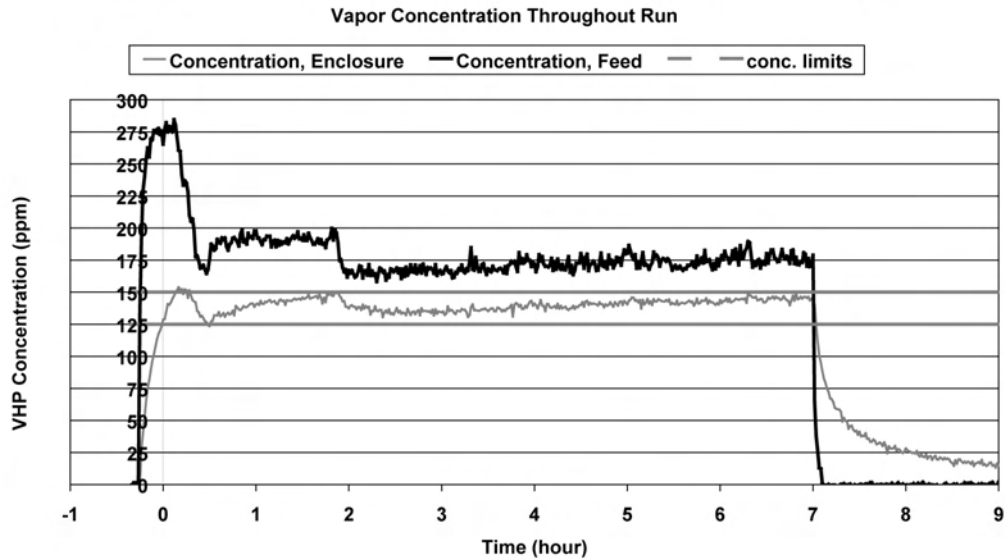
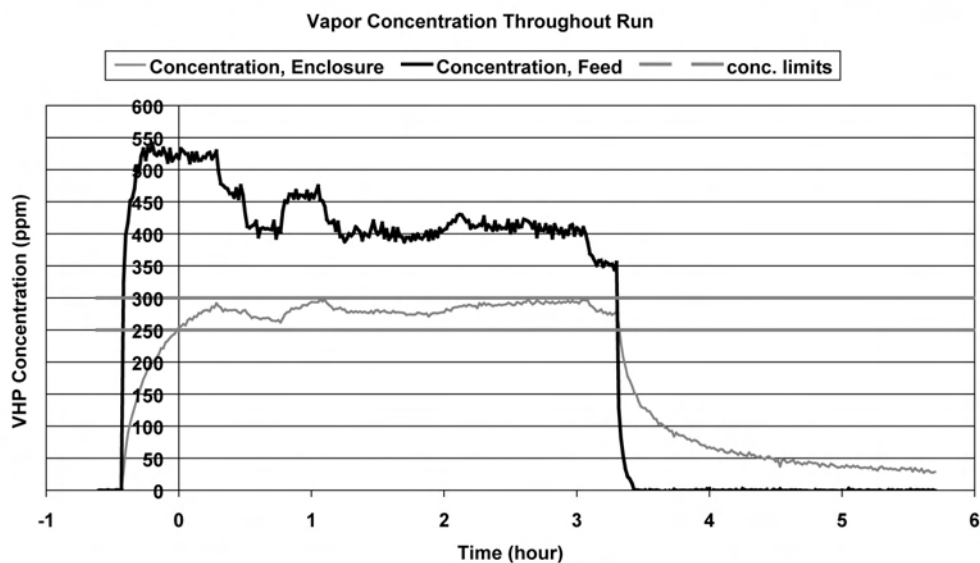


Figure 6.1.11: Representative Concentration Profile Results for Gypsum Wallboard

a) Concentration Profile for Gypsum Wallboard, Exposure at 250-300 ppm (6May05 run)



b) Concentration Profile for Gypsum Wallboard, VHP Exposure at 125-150 ppm (31May05 run)

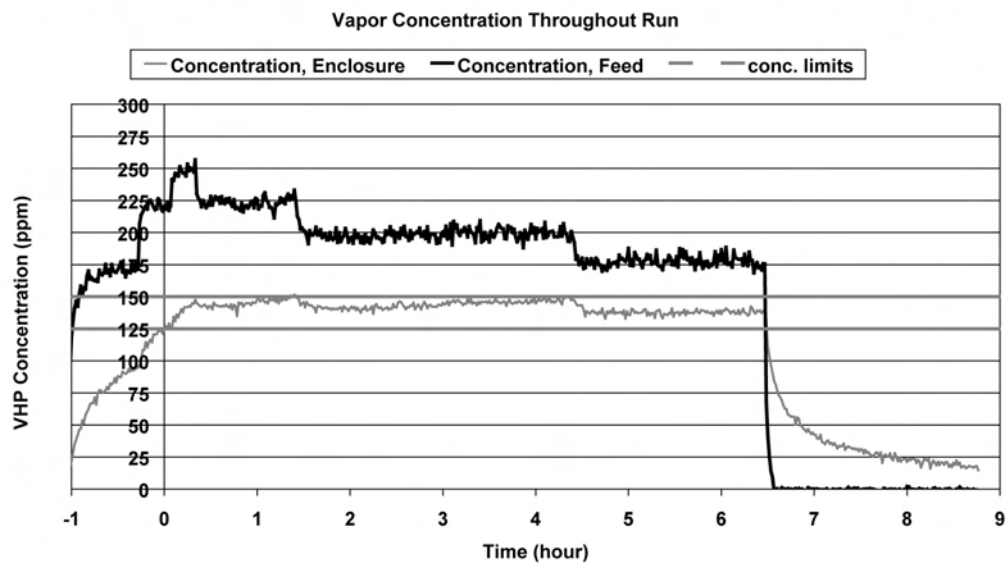
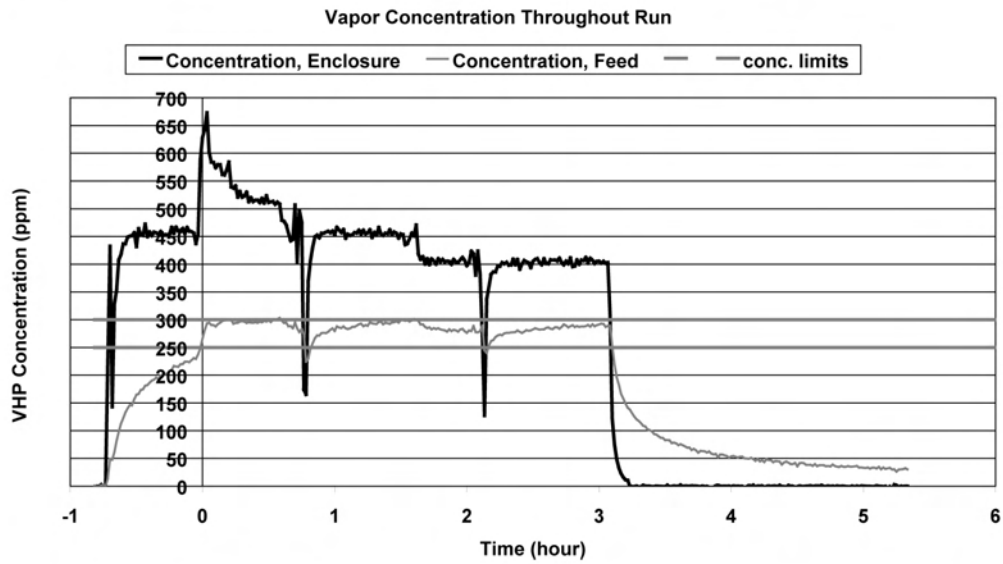


Figure 6.1.12: Representative Concentration Profile Results for Acoustical Ceiling Tile

a) Concentration Profile for Acoustical Ceiling Tile, VHP Exposure at 250-300 ppm (16Mar05 run)



b) Concentration Profile for Acoustical Ceiling Tile, VHP Exposure at 125-150 ppm (1Jun05 run)

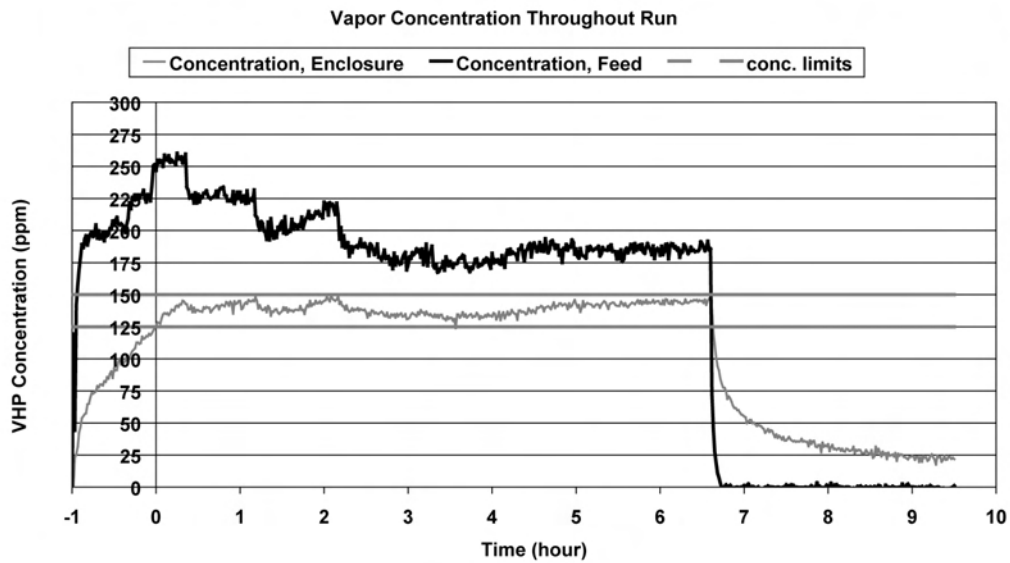
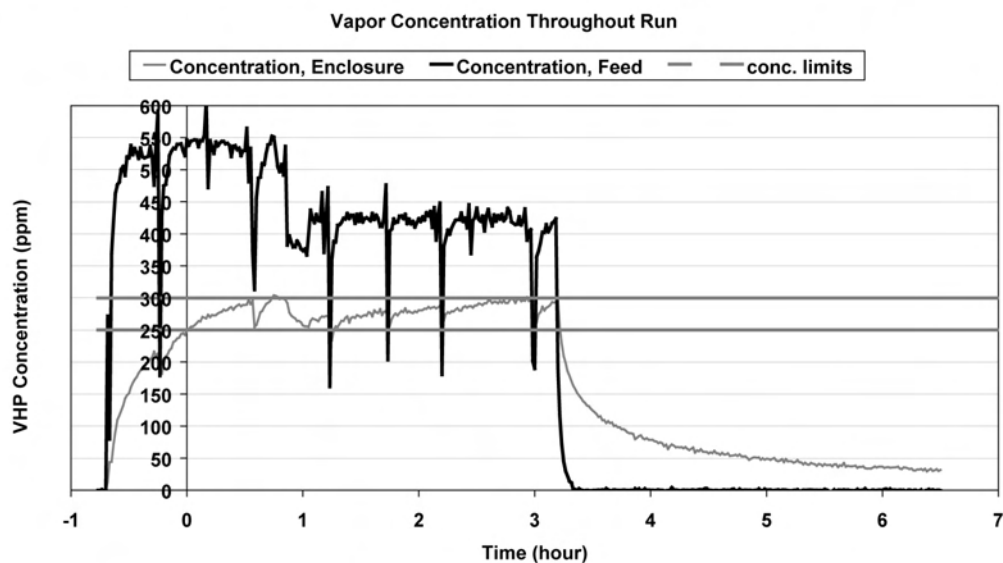


Figure 6.1.13: Representative Concentration Profile Results for Wood

a) Concentration Profile for Wood, VHP Exposure at 250-300 ppm (26Apr05 run)



b) Concentration Profile for Wood, VHP Exposure at 125-150 ppm (10May05 run)

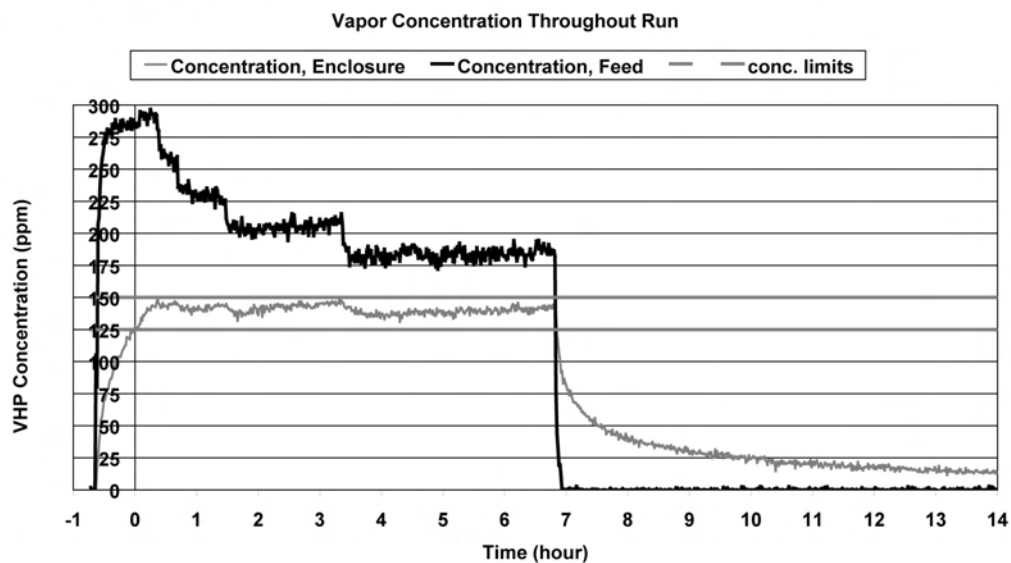
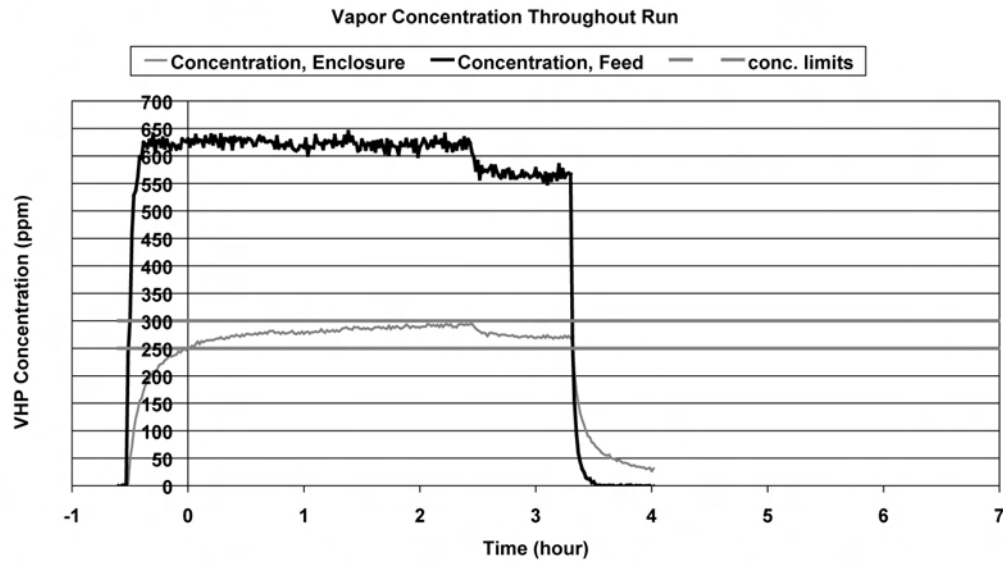
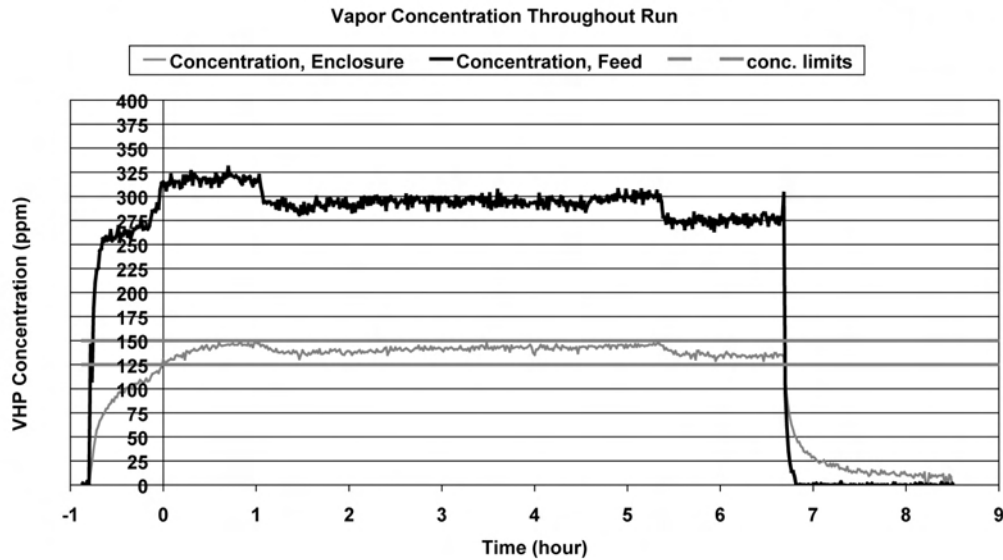


Figure 6.1.14: Representative Concentration Profile Results for Concrete Cinder Block

a) Concentration Profile for Concrete Cinder Block, VHP Exposure at 250-300 ppm (4May05 run)



b) Concentration Profile for of Concrete Cinder Block, VHP Exposure at 125-150 ppm (2Jun05 run)



6.2 Discussion

Carpet and the two materials that were painted (steel and wallboard) had the lowest VHP® material demand. The sorptive building materials, such as ceiling tile, wood, and concrete cinder block had greater VHP® material demand. Concrete cinder block showed the greatest material demand of the materials studied.

Using the baseline as the reference point, some of the materials were observed to adsorb VHP® and others to decompose VHP®. The aeration time for the 250-ppmv VHP® run is shown in Figure 6.2.1a. Figure 6.2.1b shows a zoomed in view of the aeration cycle at the 30-ppmv end of run with bars highlighting the aeration span covered by the three replicate runs for each material and the empty chamber.

The concrete cinder block required the highest increase in generator output to maintain the target concentration within the enclosure, hence the highest material demand. The concrete cinder block also had the shortest aeration time indicating that the majority of the excess VHP® introduced into the chamber was decomposed by the concrete cinder block surface.

The two cellulose-based materials wood and acoustical ceiling tile required a high increase in the generator output to maintain the target concentration within the enclosure. The wood and acoustical ceiling tile tests

also had the longest aeration time indicating that these materials adsorbed VHP® during the decontamination phase and off-gassed VHP® during the aeration phase.

The wallboard test results had a similar material demand value and shorter aeration time compared to ceiling tile and wood. Based on this comparison, the VHP® was most likely adsorbed and decomposed by the painted wallboard surface.

The carpet test results indicated a low material demand value and an aeration time similar to the baseline study. Based on this comparison, the VHP® was not adversely affected by the carpet.

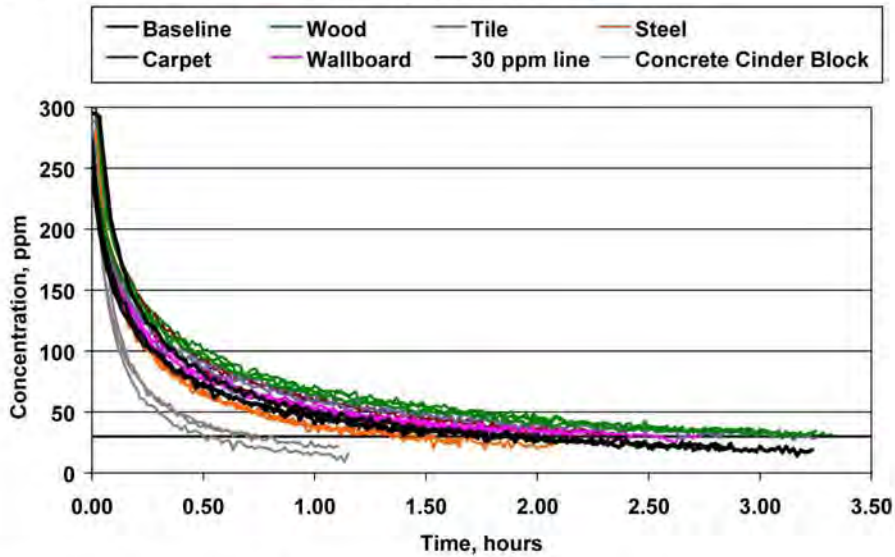
The steel test results indicated a low material demand value compared to wallboard, ceiling tile, wood and concrete cinder block. The steel samples also had a short aeration time comparable to the baseline tests. The steel may be adsorbing or decomposing the VHP®.

Based on these results, building materials may impact the ability to maintain the target concentration by adsorption and/or decomposition of the VHP®. In addition, some materials may continue to offgas VHP® after decontamination is completed resulting in longer cycle times.

Figure 6.2.1: Aeration Time for Building Materials Exposed to 250 ppm VHP

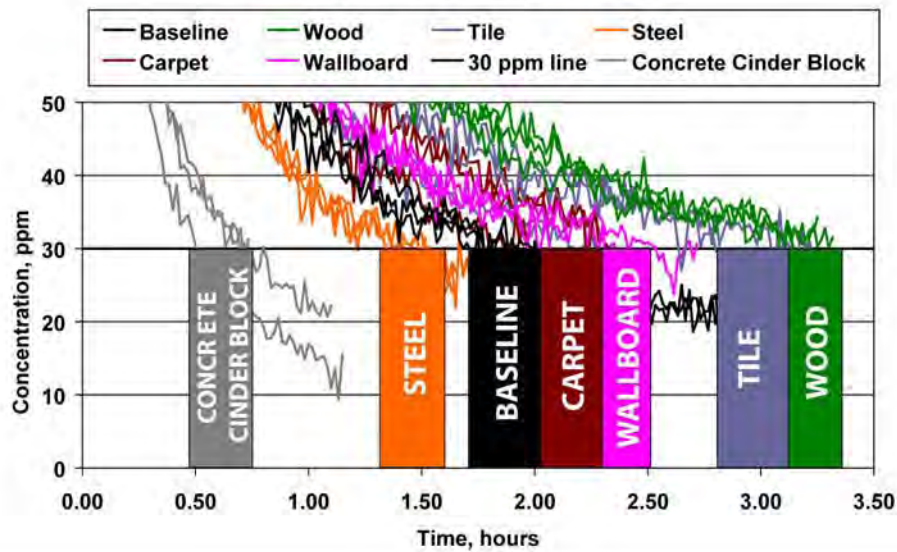
a) Aeration Time - Full View

Aeration Cycle, 250 ppm Test



b) Aeration Time - Zoom View with Approximate Timespan

Aeration Cycle, 250 ppm Test (Zoom View)



6.3 Consequences for Building Decontamination

This study provides information that may be used to support the determination of the VHP[®] concentration and aeration time requirements for the decontamination of an interior space containing the building materials studied. Based on these results, building materials may impact the ability to maintain the target concentration by adsorption and/or decomposition of the VHP[®]. In addition, some materials may continue to offgas VHP[®] after decontamination is completed, resulting in longer cycle times.

The material demand values reported can be used to estimate the total hydrogen peroxide capacity required to maintain the desired peroxide concentration. As an example of how such an estimate might be generated, consider two 900 m² (~10,000 ft²) spaces: one a warehouse environment and the other an office space. For this example, the warehouse has a concrete floor, cinderblock walls, and a steel roof. The office building has a carpeted floor, painted wallboard walls, and a dropped ceiling. In both buildings, the walls are 3 meters from the floor to the ceiling/roof and the building is 30 meters on each side. The buildings will be fumigated at a concentration of 300 ppmv of hydrogen peroxide for 3.33 hours resulting in a CT of 1000 ppmv-hrs.

Tables 6.3.1 and 6.3.2 give the estimate of required hydrogen peroxide production capacity by multiplying the surface area of each material present in the building by the flux, J, from Table 6.1.2. The requirement calculated represents the excess capacity that the hydrogen peroxide generator must supply in addition to the capacity needed to maintain a concentration of 300

ppmv, or 0.42 g/m³. To illustrate how this information could be used in practice, consider the STERIS VHP[®] 100M, which in an open loop configuration has maximum peroxide and air flow rates of 504 g/hr (24 g/min of 35% H₂O₂) and 75 m³/hr, respectively, for a feed concentration of 6.7 g/m³. Of that capacity, 6.3 g/m³ would be available to overcome the material demand in the contaminated space. Dividing the material demand by this excess generation capacity results in the air exchange rate required to maintain the air concentration of peroxide and overcome the material demand.

From Tables 6.3.1 and 6.3.2, it is clear that the materials within a particular building can have a significant impact on the generation capacity required for decontamination. The concrete/cinderblock building requires about twice as much vapor generation capacity as the office space, mainly due to the high material demand of the concrete. As a result, the warehouse would require at a minimum use of eleven STERIS VHP[®] 100M units, while the office building would require at least five. These requirements could be lowered if the air circulation, temperature and relative humidity within the buildings were such that the hydrogen peroxide vapor generators could be configured to output even higher concentrations.

Table 6.3.1 Material Demand of Warehouse Surfaces

Surface	Surface area (m ²)	J (g/hr/m ²)	HP required (g/hr)	Air exchange (m ³ /hr)
Concrete floor	929	3.61	3354	532
Cinderblock walls	360	3.61	1300	206
Steel ceiling	929	0.455	423	67
Total			5076	805

Table 6.3.2 Material Demand of Office Surfaces

Surface	Surface area (m ²)	J (g/hr/m ²)	HP required (g/hr)	Air Exchange (m ³ /hr)
Carpet	929	0.338	314	50
Painted Wallboard	360	1.403	505	80
Ceiling tile	929	1.650	1532	243
Total			2351	373

7.0

Quality Assurance Findings

Four technical systems audits were completed. Overall there were no follow-up corrective actions from testing. During the first audit two on-the-spot corrections were made. The first correction occurred during the chamber shutdown procedure. The operator skipped a step in the procedure. The auditor caught the mistake and the operator backed up and completed the shutdown. The second mistake was misnumbering of one coupon. The operator identified the error, re-measured the coupon and matched the coupon to the correct CoC card. The sample was re-numbered. The corrections during the first audit were not unexpected since the procedures and equipment were new and the audit was done during the first test.

8.0

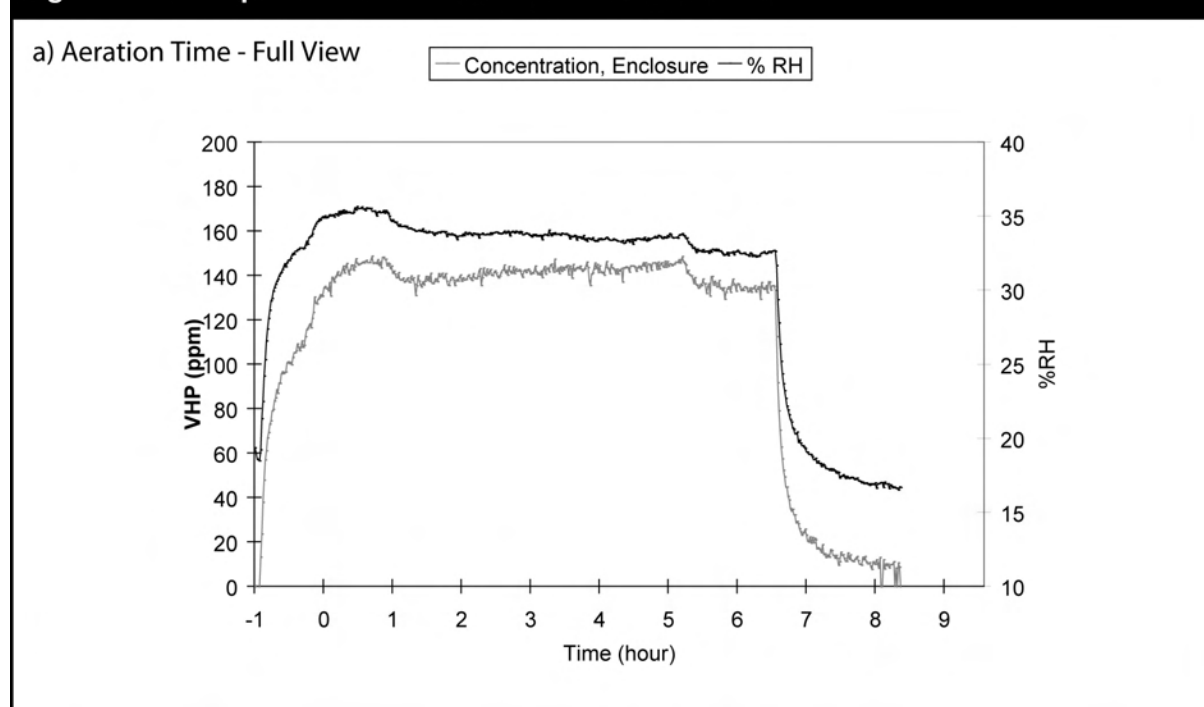
Challenges and Lessons Learned

8.1 VHP® Relative Humidity Sensors

The Vaisala HUMICAP humidity sensor (model HMT333) read slightly high in the presence of VHP® at approximately 300 ppmv, but when removed from air containing VHP® the sensors responded normally with no change in response. The relative response of the sensor to VHP® and humidified air was checked by separately injecting distilled water or 35% hydrogen peroxide at identical rates into the VHP® generator and observing the resultant %RH in the chamber after equilibration. The injection rate was similar to that used during most of the exposure runs. When distilled water or 35% hydrogen peroxide was injected, the sensor read 32.2 %RH and 34.3 %RH, respectively, indicating that the sensor reads the %RH high by approximately 2 %RH units in the presence of VHP®. Even though the VHP® effect on %RH was small, VHP® did not have

any impact on the maximum 30% RH SOR requirement since the sensor was not exposed to VHP® at the SOR. The %RH probe response was verified after completing approximately 3/4 of the runs by exposure to humidified air above saturated salt slurries. Slurries of potassium acetate, potassium carbonate and sodium chloride were used to yield standard humidities of 23, 44, and 76 % relative humidity, respectively. The variations between the standard % relative humidities and the sensor %RH readings (23.4, 42.6, and 77.9 %RH, respectively) were < 3.5%. During the test runs the %RH generally mimicked the VHP® concentration in the enclosure, showing that the change in %RH was related generally to the changes in VHP® concentration (Figure 8.1).

Figure 8.1: Comparison of VHP Concentration and %RH



8.2 Calibration of Dräger Hydrogen Peroxide Electrochemical Sensors

Initial calibration attempts for the hydrogen peroxide sensors using sulfur dioxide in nitrogen gas revealed that the sensors were highly sensitive to changes in pressure. The calibration procedure with sulfur dioxide required that the sensors be removed from their test enclosures and placed in a low volume calibration adaptor through which the calibration gas could be passed. This procedure allowed for conservation of the calibration gas and also for the quick equilibration of the calibration gas concentration. The calibration procedure was quick and straightforward, but when the sensors were placed in their test enclosures, the inlet sensor read higher than the enclosure sensor, due to the inlet enclosure experiencing slightly more pressure than the exit enclosure.

Switching the sensors revealed that the variation was due to their placement and not the specific sensor. The sensors could be calibrated with the sulfur dioxide gas procedure, but the verification agreement with the peroxide concentration from the iodometric titration was typically in error by approximately 15% due to the slight variation in enclosure pressures. Attempts to refine the sulfur dioxide calibration procedure and successfully validate were exhausted.

The hydrogen peroxide sensors were recalibrated in place using VHP[®] concentration values determined by chemical titration of VHP[®] captured in bubbler solutions. The inlet hydrogen peroxide detector was calibrated to measure from 0 to 800 ppmv H₂O₂, and the outlet hydrogen peroxide detector was calibrated to measure from 0 to 340 ppmv H₂O₂ IAW IOP DS04001. The outlet sensor was expected to experience concentrations no greater than 300 ppmv and was therefore calibrated slightly higher at 340 ppmv. The inlet sensor was calibrated from 0 to 800 ppmv H₂O₂ to accommodate anticipated higher VHP[®] concentrations in the feed air. Verification of both sensors was conducted during each run using the average value from three iodometric titrations on the VHP[®] stream entering and exiting the glove box (IOP DS04019).

9.0

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Appendix A:

Detailed Coupon Preparation and Inspection Procedures

Appendix A: Detailed Coupon Preparation and Inspection Procedures

COUPON PREPARATION PROCEDURE

The coupon preparation, unless otherwise noted, will be conducted at the Edgewood Chemical Biological Center Experimental Fabrication Shop.

Mechanically Graded Lumber (Bare Wood)

- The machined ends of the stock will be discarded by removing $> \frac{1}{4}$ in. of the machined end. Coupons will be cut from stock using a table saw equipped with an 80 tooth crosscut blade.

Latex-Painted Gypsum Wallboard

- Stock Item Description: $\frac{1}{2}$ in. 4 ft. x 8 ft. Drywall
- Supplier/Source: Home Depot, Edgewood Maryland
- Coupon Dimensions: 6 in. x 6 in. x $\frac{1}{2}$ in.
- Preparation of Coupon:
 - The ASTM method requires that the samples be taken from the interior of material rather than from the edge (machined edge). The machined ends of the stock will be discarded by cutting away > 4 inches from each side.
 - Coupons will be cut from stock using a table saw equipped with an 80 tooth crosscut blade.
 - The 6 in. x 6 in. coupons will be painted with 1-mil of Glidden PVA Primer and followed by 1-2-mils of Glidden latex topcoat. The primed coupons will be allowed to stand for > 24 hours prior to the application of the topcoat.
 - All six sides of the 6 in. x 6 in. coupon will be painted.

Concrete Cinder Block

- Stock Item Description: 8 in. x 16 in. x 1.5 in. concrete block cap
- Supplier/Source: York Supply, Aberdeen Maryland
- Original Coupon Dimensions: 4 in. x 8 in. x 1.5 in.
- Modified Coupon Dimensions: 4 in. x 8 in. x 0.5 in.
- Preparation of Coupon:
 - Coupons will be cut from stock using a water-jet.
 - Four coupons will be cut from each stock piece.
 - Original dimensions too large for material testing
 - Each coupon cut into three sections.
 - Two sections measured at modified coupon dimensions
 - Third section discarded

Appendix A:

Detailed Coupon Preparation and Inspection Procedures

Carpet

- Stock Item Description: 12 ft. Powerhouse 20 Tradewind
- Supplier/Source: Home Depot, Edgewood, Maryland
- Coupon Dimensions: 6 in. x 8 in.
- Preparation of Coupon:
 - Coupons will be cut from the stock using a utility knife.
 - The longer direction (8 in.) will be cut parallel to the machine edge.
 - The machined edge will be discarded by removing > ½ in.

Painted Structural Steel

- Stock Item Description: A572 Grade 50, 4 ft. x 8 ft. x ¼ in.
- Supplier/Source: Specialized Metals
- Coupon Dimensions: 1/4 inches x 12 inches total, dog bone shaped with 2 inches wide at ends, ¾" wide inch center
- Preparation of Coupon:
 - Coupons will be cut from stock using a water-jet.
 - A visual observation will be conducted on each coupon to determine if size and shape have deviated from dimension. If deviation has occurred, the coupon will be discarded.
 - Coupons will be cleaned and degreased following procedures outlined in TTC-490.
 - Coupons will be prepared for painting per TT-P-645 with red oxide primer.

The Edgewood Chemical Biological Center Experimental Fabrication Shop prepared the materials IAW the standards used for the preparation and painting of steel. TTC-490 is a Federal Standard providing cleaning methods and pretreatment for iron surfaces for application of organic coatings. The pretreatment is the application of a zinc phosphate corrosion inhibitor. TT-P-645 is a Federal Standard for the application of alkyd paint. These standards were not obtained through this program but were purchased by the Shop for their work.

Ceiling Suspension Tile

- Stock Item Description: Armstrong 954, Classic Fine Textured, 24 in.x 24 in.x 9/16 in.
- Supplier/Source: Home Depot, Edgewood, Maryland
- Coupon Dimensions: 12 in. x 3 in. x 9/16 in.
- Preparation of Coupon:
 - Coupons will be cut from stock using a table saw equipped with an 80-tooth crosscut blade.
 - Sixteen samples will be removed from each stock item.

Appendix A:

Detailed Coupon Preparation and Inspection Procedures

COUPON INSPECTION PROCEDURE

All coupons will be inspected prior to testing to ensure that the material being used is in suitable condition. Coupons will be rejected if there are cracks, breaks, dents or defects beyond what are typical for the type of material. In addition, coupons will be measured to verify the coupon dimensions. Coupons deviating from the dimension ranges listed below will be discarded.

Mechanically Graded Lumber (Bare Wood)	10 in. $\pm$ 1/16 in. x 1.5 in. $\pm$ 1/16 in. x 0.5 in. $\pm$ 1/32 in.
Latex-Painted Gypsum Wallboard	6 in. $\pm$ 1/16 in. x 6 in. $\pm$ 1/16 in. x 0.5 in. $\pm$ 1/16 in.
Concrete cinder Block	4 in. $\pm$ 1/2 in. x 8 in. $\pm$ 1/2 in. x 0.5 in. $\pm$ 1/8 in.
Carpet	6 in. $\pm$ 1/8 in. x 8 in. $\pm$ 1/8 in.
Painted Structural Steel	1/4 in. $\pm$ 1/128 in. x 12 in. $\pm$ 1/16 in. with 2 in. $\pm$ 1/16 in. wide at ends, 3/4 in. $\pm$ 1/16 in. wide inch center Lot SS: same but with 0.27 in. $\pm$ 0.02 in. thickness
Ceiling Suspension Tile	12 in. $\pm$ 1/8 in. x 3 in. $\pm$ 1/16 in. x 9/16 in. $\pm$ 1/16 in.

Appendix B:

Coupon Identifier Code

Appendix B: Coupon Identifier Code

All coupons will be marked with an ID number that will consist of a nine character alphanumeric code. A description of the identifier pattern and an example code are shown below.

Code Pattern

<u>Character</u>	<u>Explanation</u>
1	Material W = wood G = gypsum S = A572 steel T = acoustic ceiling tile C = concrete cinder block R = carpet B = circuit breakers
2	Fumigant: V = VHP N = no fumigant
3	Test start date
4,5	year for example: 4 = 2004
6,7	month for example: 06 = June
	day for example: 10 = the 10 th of a month
8,9	Chamber position (see IOP DS04016 figure 1)

Example

GV4101104

Gypsum Wallboard with test start date of October 11th, 2004.
Chamber position number 4.

Appendix B:

Coupon Identifier Code (Cont.)

Figure B.1: IOP DS04016 Figure 1, "Coupon Placement in Chambers"

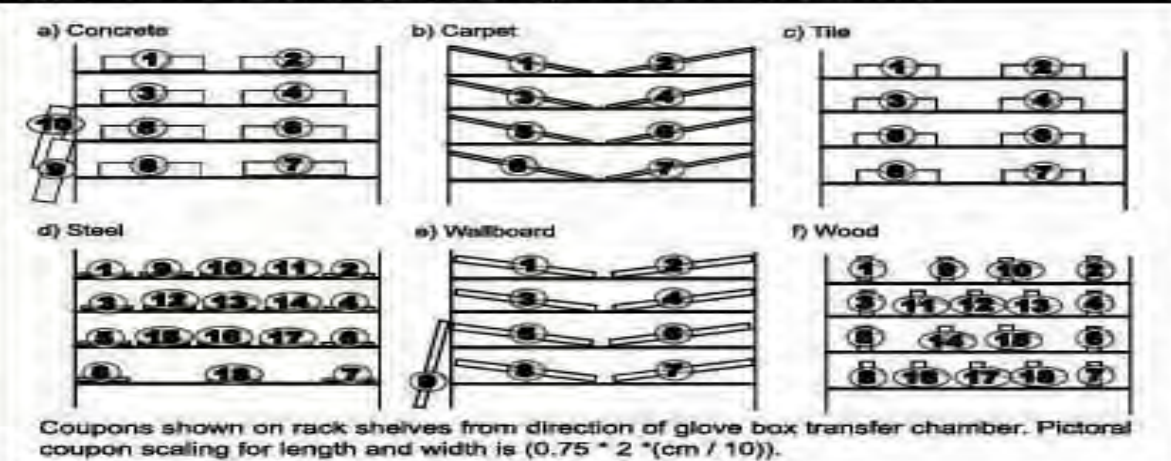
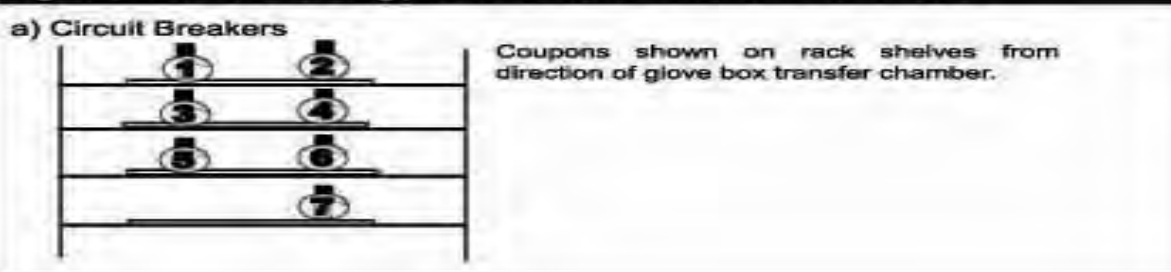


Figure B.2: IOP DS04016 Fig., "Circuit Breaker Placement in Chambers"



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