

Material Demand Studies: Interaction of Chlorine Dioxide Gas With Building Materials

REPORT



Office of Research and Development National Homeland Security Research Center

Material Demand Studies: Interaction of Chlorine Dioxide Gas With Building Materials

Philip W. Bartram Joseph T. Lynn Louis P. Reiff Mark D. Brickhouse Teri A. Lalain EDGEWOOD CHEMICAL AND BIOLOGICAL CENTER RESEARCH AND TECHNOLOGY DIRECTORATE Aberdeen Proving Ground, MD

Shawn Ryan Blair Martin UNITED STATES ENVIRONMENTAL PROTECTION AGENCY Research Triangle Park, NC 27711

David Stark SCIENCE APPLICATIONS INTERNATIONAL CORPORATION Abingdon, MD 21009

Disclaimer

EPA through its Office of Research and Development partially funded and collaborated in the research described herein under Interagency Agreement (IAG) DW 939917-01-0 with the U.S. Army Edgewood Chemical and Biological Center (ECBC). The work performed in association with this report was conducted from November 2003 through July 2006. The report has been subject to an administrative review but does not necessarily reflect the views of the Agency. No official endorsement should be inferred. EPA does not endorse the purchase or sale of any commercial products or services.

Executive Summary

1.0 BACKGROUND

The material demand effort was initiated 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 composed of concrete cinder block, wood, steel, carpet, ceiling suspension tile, and painted wallboard, 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. Chlorine dioxide results are presented in this report. The representative building interior materials tested were unpainted concrete cinder block, standard stud lumber (fir, type-II), latex-painted ¹/₂-inch gypsum wallboard, ceiling suspension tile, painted structural steel, and carpet. The collaborative effort was funded by the U.S. Environmental Protection Agency (EPA) National Homeland Security Research Center (NHSRC).

2.0 TEST PROTOCOL

The tests were monitored under an approved Quality Assurance Project Plan (QAPP). The Deposition Velocity QAPP specified procedures for the review of data and independent technical system audits. All test data were peer reviewed within two weeks of data 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.

3.0 SUMMARY OF CONCLUSIONS

The chlorine dioxide material demand tests showed that the feed concentration and time required to reach the target concentration (1000 and 2000 parts per million volume [ppmv]) were a function of building material. The chlorine dioxide demand for the building materials over the 0–12000 concentration time (CT) range was (from highest to lowest) ceiling tile > wood ≥ gypsum wallboard > carpet > concrete = steel = baseline for the 1000 ppmv tests and ceiling tile > gypsum wallboard > carpet > wood > concrete = steel = baseline for the 2000 ppmv tests. Concrete and steel were not statistically different from the baseline in unpaired Student's t Tests at $\alpha = 0.05$.

Preface

Data were recorded in ECBC laboratory notebook 04-0055, entitled EPA Material Compatibility Study.

Reproduction of this document either in whole or in part is prohibited except with permission of the Director, U.S. Army Edgewood Chemical Biological Center, ATTN: AMSRD-ECB-RT-OM, Aberdeen Proving Ground, MD 21010-5424, and the U.S. Environmental Protection Agency (MD-E343-06), Office of Research and Development, National Homeland Security Research Center, Research Triangle Park, NC 27711. However, the Defense Technical Information Center is authorized to reproduce the document for U.S. government purposes.

Acknowledgments

The authors thank the following individuals for their contributions toward the successful completion of this test program: Mr. Brian MacIver and Mr. Dave Sorrick for assistance with acquiring materials and equipment fabrication; Ms. Diane Simmons for assistance with the issuance of this report; and 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.

Contents

| 1. | INTRODUCTION | 1 | | | | | |
|-----|---|-----|--|--|--|--|--|
| 2. | OBJECTIVE | 1 | | | | | |
| 3. | EXPERIMENTAL PROCEDURE | | | | | | |
| | 3.1 Representative Building Material Test Coupons | | | | | | |
| | 3.2 Chlorine Dioxide Test Glove Box, Auxiliary Equipment, | | | | | | |
| | and Operation | | | | | | |
| | 3.3 Calibration of INTERSCAN Detectors | 5 | | | | | |
| | 3.4 Calculation of Material Surface Area | 6 | | | | | |
| | 3.5 Humidity and Temperature Control | 7 | | | | | |
| | 3.6 Additional Requirements for Humidity Conditioning | 7 | | | | | |
| | 3.7 Fumigation Cycle | 7 | | | | | |
| | 3.7.1 Control of Chlorine Dioxide Concentration in Exposure Chamber | 7 | | | | | |
| | 3.7.2 Aeration Cycle | 7 | | | | | |
| 4. | DATA REVIEW AND TECHNICAL SYSTEM AUDIT | 8 | | | | | |
| 5. | MATERIAL DEMAND CALCULATIONS AND DESCRIPTIVE STATISTICAL ANALYSIS | 8 | | | | | |
| | 5.1 Material Demand Calculations | 8 | | | | | |
| | 5.2 Descriptive Statistical Analysis | .10 | | | | | |
| 6. | RESULTS | .10 | | | | | |
| | 6.1 Evaluation of Empty Glove Box | .10 | | | | | |
| | 6.1.1 "Fog" Test Results and Discussion | .10 | | | | | |
| | 6.1.2 Material Demand of the Baseline Chamber | .11 | | | | | |
| | 6.2 Material Demand of the Selected Building Materials | .12 | | | | | |
| | 6.2.1 Carpet | .13 | | | | | |
| | 6.2.2 Painted Steel | .13 | | | | | |
| | 6.2.3 Gypsum Wallboard | .14 | | | | | |
| | 6.2.4 Ceiling Tile | .14 | | | | | |
| | 6.2.5 Wood | .15 | | | | | |
| | 6.2.6 Concrete | .15 | | | | | |
| | 6.3 Total CT Demand for Baseline and Materials | .16 | | | | | |
| | 6.4 Discussion | .17 | | | | | |
| 7. | OPERATIONAL PROBLEMS ENCOUNTERED | .17 | | | | | |
| 8. | QUALITY ASSURANCE FINDINGS | .18 | | | | | |
| 9. | CONCLUSIONS | .19 | | | | | |
| 10. | LITERATURE CITED | .20 | | | | | |

Appendices

| A. | DETAILED COUPON PREPARATION AND INSPECTION PROCEDURES | .A-1 |
|----|--|------|
| B. | COUPON IDENTIFIER CODE | .B-1 |
| C. | TYPICAL TIME/CONCENTRATION AND TIME/CT PROFILES FOR CHLORINE DIOXIDE TESTS | .C-1 |
| D. | EXCEL DATA WORKSHEET FOR COMPARISON OF INTERSCAN DETECTORS AND TITRATION CHECKS | .D-1 |
| E. | RESULTS OF CHLORINE DIOXIDE MATERIAL DEMAND TESTS | .E-1 |

Figures

| 1. | Representative Building Material Test Coupons | 2 |
|----|---|-----|
| 2. | Glove Box Used for Chlorine Dioxide Material Demand Tests | 3 |
| 3. | Photograph of Chlorine Dioxide Material Demand Test Equipment | 5 |
| 4. | Typical Calibration Curve for INTERSCAN Detector | 6 |
| 5. | Illustration of Time Zero (Baseline Test on 18 Jan 06) | 8 |
| 6. | Illustration of the Calculation of the Material Demand | 8 |
| 7. | Exposure Chamber Fog Test | .11 |
| 8. | Photographs of the Interior of an INTERSCAN Detector | .18 |
| 9. | Comparison of Chamber Fans Exposed to Chlorine Dioxide and VHP® | .19 |

Tables

| 1. | Representative Building Materials | 2 |
|----|--|----|
| 2. | Exposed Surface Area of Coupons | 6 |
| 3. | Baseline Material Demand Test Results | |
| 4. | Baseline-Corrected Material Demand Test Results for Each Material Type (Target Chamber Concentration = 1000 ppmv ClO ₂) | 12 |
| 5. | Baseline-Corrected Material Demand Test Results for Each Material Type (Target Chamber Concentration = 2000 ppmv ClO ₂) | 13 |
| 6. | Total Feed and Effluent CT for Each Material Test | 16 |

List of Acronyms and Symbols

| ACGIH® | American Conference of Government Industrial Hygientists, Inc. |
|-----------------------------|--|
| APG | Aberdeen Proving Ground |
| ASTM | American Society for Testing and Materials |
| atm | atmosphere |
| bp | boiling point |
| °C | celsius |
| CAS | Chemical Abstract Services |
| CB | chemical and biological |
| ClO ₂ | chlorine dioxide |
| cm | centimeters |
| CoC | chain-of-custody |
| CT or CT _{ppmy-hr} | concentration · time (units of ppmv-hr) |
| CTinlet | CT of stream entering the glove box |
| CToutlet | CT of stream exiting the glove box |
| CTmass | CT in mass concentration units (g \cdot hr/m ³) |
| decon | decontamination |
| ECBC | Edgewood Chemical and Biological Center |
| EOR | end of run |
| EPA | U.S. Environmental Protection Agency |
| ESD | extreme studentized deviate |
| ft | feet |
| g | grams |
| hr | hour or hours |
| IAW | in accordance with |
| in. | inches |
| IOP | Internal Operating Procedure |
| J | flux $(g \cdot m^{-2} \cdot hr^{-1})$ |
| 1 | length |
| L | liters |
| K | Kelvin |
| m | meters |
| mg | milligram |
| MD | material demand |
| min | minutes |
| mL | milliliters |
| mp | melting point |
| MW _{CIO2} | molecular weight of ClO ₂ |
| NHSRC | National Homeland Security Research Center |
| NIOSH | National Institute for Occupational Safety and Health |
| OSHA | Occupational Safety and Health Administration |
| ppm | part per million |
| ppmv | parts-per-million (volume) |
| P _{sys} | chamber pressure in units of atmosphere |
| PEL | permissible exposure limit |
| Q | inlet and outlet flow rate |
| QA | quality assurance |

| QAPP | Quality Assurance Project Plan (QAPP) |
|-----------------------|--|
| QMP | Quality Management Plan |
| R | universal gas constant |
| REL | recommended exposure limits |
| RH | relative humidity |
| SA or A | surface area |
| SOP | standing operating procedures ("standard" may also be used in place of "standing" with the same meaning) |
| SD | standard deviation |
| STEL | short-term exposure limits |
| Т | temperature |
| t | time |
| TWA | time-weighted average |
| TICs | toxic industrial chemicals |
| TIMs | toxic industrial materials |
| TR | technical report |
| T _{sys} | chamber temperature |
| U.S. | United States |
| UV | ultraviolet |
| VHP® | Steris' registered "vaporized hydrogen peroxide" procedure |
| Vi | affluent flow rate in m ³ · min ⁻¹ |
| Vo | effluent flow rate in $m^3 \cdot min^{-1}$ |
| W | width |
| ΔCT_{b} | $CT_{inlet} - CT_{outlet}$ for baseline study |
| ΔCT_{mb} | $CT_{inlet} - CT_{outlet}$ for material study |
| ΔCT_k | difference between target CT and input CT after baseline subtraction |
| $\Delta CT_{k(mass)}$ | difference between inlet and outlet ClO_2 in units of $\mathbf{g} \cdot \mathbf{hr}^1 \cdot \mathbf{m}^3$ |
| $\Delta_{\rm mass}$ | difference in chlorine dioxide between feed and effluent in grams |
| | |

Coupon Specific Coding

| "W" | bare wood |
|-----|--|
| "R" | carpet |
| "T" | ceiling suspension tile |
| "G" | latex-painted gypsum wallboard |
| "S" | painted structural A572 steel |
| "С" | unpainted concrete cinder block |
| "V" | exposed to chlorine dioxide fumigation |
| "N" | no fumigant exposure |

Material Demand Studies: Interaction of Chlorine Dioxide Gas With Building Materials

1. INTRODUCTION

In 2004, the U.S. Environmental Protection Agency (EPA) established a collaborative Interagency Agreement with the U.S. Army Edgewood Chemical and Biological Center (ECBC) to conduct research and specialized testing in building decontamination. ECBC has been the major government agency for chemical and biological (CB) decontamination research and product development since World War I. The EPA National Homeland Security Research Center (NHSRC) and Decontamination Sciences Team, Research & Technology Directorate collaborated to study the effects of hydrogen peroxide vapor and chlorine dioxide gas on interior building materials and to determine the rate of adsorption (and/or decomposition) of these decontaminants by the materials. Laboratory tests confirmed that chlorine dioxide at a concentration of > 600 parts-per-million volume (ppmv) and \geq 75 % relative humidity (RH) was very effective in killing (~ 7 log reduction in 12 hour [hr]) Bacillus anthracis var. ames, and Bacillus anthracis var. vollum.¹ The hydrogen peroxide fumigant was initially used to sterilize pharmaceutical processing equipment and clean rooms.^{2,3} Chlorine dioxide (ClO₂) and vaporized hydrogen peroxide (VHP®) technologies have since been used to decontaminate (fumigate) the interior of buildings contaminated with anthrax. In 2001, chlorine dioxide was successfully employed to decontaminate the anthrax-contaminated Hart Senate Office Building in Washington, D.C. In 2003, VHP® was used to disinfect the U.S. Department of State SA-32, Sterling Mail Facility, in Virginia, and the General Services Administration (GSA) Building 410, Anacostia Naval Base, in Washington, D.C.

Gaseous reactive compounds provide several advantages over standard liquid decontaminants for the decomposition of chemical and biological warfare agents deposited in the interiors of building. The most significant advantages are the ease of dispersal of reactive molecules throughout a defined space and access to non-line-of-sight areas. However, building interiors may contain large surface areas composed of complex materials such as concrete, wood, steel, carpet, ceiling tile, and painted wallboard that may affect or be affected by the fumigant. The NHSRC and Decontamination Science Team collaboration was initiated to determine how building materials impact the concentration of the decontaminant in the vapor phase and how the materials are impacted by the fumigant. 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.

In this study, the material demand for chlorine dioxide was determined in tests designed to simulate decontamination of a building. The term "material demand" includes adsorption and decomposition of the fumigant that will affect its concentration within the fumigation volume. Data from these tests could be used to predict the concentration of chlorine dioxide in the feed stream that would be required to maintain the target concentration inside a facility. Material demand and compatibility data will be used by facility managers, first responders, groups responsible for building decontamination, and other technology buyers and users for purposes of restoring a public building to a usable state after a terrorist contamination incident.

2. OBJECTIVE

The objective of this study was to determine the material demand, expressed as mass flux, of selected building materials (concrete, painted steel, wood, gypsum wallboard, ceiling tile, and carpet) for chlorine dioxide at 1000 ppmv and 2000 ppmv during tests similar to a building decontamination process.

3. EXPERIMENTAL PROCEDURE

The material demand testing was conducted in compliance with the Quality Assurance Project Plan (QAPP)⁴ developed under the Quality Management Plans (QMP)^{5, 6} and EPA Quality Assurance (QA) Category 4 requirements.^{7, 8, 9, 10}

3.1 Representative Building Material Test Coupons

Test coupons were prepared in accordance with the American Society for Testing and Materials (ASTM) requirements for the material compatibility testing¹¹ and under the QAPP,⁴ entitled "Effects of Vaporized Decontamination Systems on Selected Building Interior Materials." The coupons were cut from stock material in accordance with the procedure in Appendix B of the QAPP⁴ and reproduced in 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 1 and shown in Figure 1. Complete information on the materials can be found in Appendix A and the QAPP.⁴

Table 1. Representative Building Materials (Dimensions from Selected Sampling of Specimens)

| Material | Supplier | Length (cm) | Width (cm) | Thickness (cm) | | | |
|--|--------------------|-----------------|--------------------|----------------|--|--|--|
| Structural Wood (fir, type II) | Home Depot | 25.4 | 3.7 ± 0.05 | 1.2 ± 0.02 | | | |
| Concrete Block | York Supply | 19.4 ± 0.2 | 9.8 ± 0.5 | 4.1 ± 0.1 | | | |
| Painted Steel | Specialized Metals | 30.4 ± 0.04 | 5.1 ± 0.02^{a} | 0.6 | | | |
| Latex-Painted Gypsum Wallboard | Home Depot | 15.2 | 15.2 | 1.3 | | | |
| Carpet | Home Depot | 20.4 ± 0.1 | 15.2 ± 0.07 | 0.6 | | | |
| Ceiling Suspension Tile, Acoustical | Home Depot | 30.5 | 7.6 | 1.4 | | | |

^a The width was measured at the end of the "dog bone" shaped specimen. The width in the center of the dog bone was 1.9 ± 0.02 cm. Two lots of painted steel were used. Both lots had similar length and width; however, the thickness of one lot was 0.6 ± 0.003 cm and the thickness of the second lot was 0.7 ± 0.01 cm. The standard deviation when not shown was either zero or rounded to zero.

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 they were 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 were damaged were discarded. Each coupon was assigned a unique identifier code that matched the coupon with the sample, test parameters, and sampling scheme as detailed in Appendix B (e.g., Some codes are displayed in Figure 1.). 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.

Figure 1. Representative Building Material Test Coupons



Note: Coupons are not shown to scale . Coupon codes were not required for this study; however, they were used for traceability to determine loss of physical integrity in subsequent tests.

3.2 Chlorine Dioxide Test Glove Box, Auxiliary Equipment, and Operation

A Plas-Labs (PLASLABS, Inc., 401 East North Street, Lansing, MI 48906) compact glove box (Model 830-ABC) fitted with Hypalon® gloves and glove port plugs was used as the exposure chamber (Figure 2). The glove box was acrylic with an internal volume of 317 L (71.1 cm x 58.4 cm x 73.7 cm) or 11.2 cubic feet (28 in. x 23 in. x 29 in.) with an isolated transfer chamber having a 30.5 cm long x 27.9 cm inside diameter. The glove box was sealed with black cardboard and plastic to prevent ultraviolet light (UV) from decomposing the chlorine dioxide; the laboratory window was also blocked with black plastic. An exposure rack constructed of Lexan® and horizontal stainless steel bars was used to hold the test specimens. The exposure rack was 30.5 cm long x 30.5 cm wide x 61 cm tall with four levels. Coupons were placed in the glove box in accordance with Internal Operating Procedure (IOP) DS0401612 and shown for each material type in Appendix B. A photograph of the coupon placement in the exposure chamber for the concrete cinder block material is provided in Figure 2.

The CIO₂ feed and effluent concentrations were monitored by INTERSCAN RM Series detectors (Interscan Corporation, P.O. Box 2496, Chatsworth, CA 91313-2496). The inlet detector measured the chlorine dioxide concentration by sampling the inlet stream prior to entering the Plas-Labs glove box. The chlorine dioxide concentration within the glove box was measured by sampling the effluent stream exiting the glove box. The sampling rate for each detector was 200 mL/min. The sensors were factory preset to measure from 0 to 4000 ppmv CIO₂ with sensitivity $\leq \pm 5\%$ of the measured value. The inlet and outlet ClO₂ detectors were calibrated over a range of 0 to 4000 ppmv in accordance with IOP DS04017.¹³

The INTERSCAN detectors were checked during the tests by sampling the affluent and effluent streams by bubblers and analyzing by the classical iodometric titration method. In this method, iodine produced from the reaction of iodide with ClO_2 was titrated with sodium thiosulfate. The endpoint was determined by color change. Detailed procedures for sampling and determination of ClO_2 were documented in IOP DS04017¹³ and DS04002.¹⁴

RH and temperature were monitored using either a General Eastern Humiscan industrial sensor or a HOBO[®] U12 Temp/RH Data Logger (Onset Computer Corporation, 470 MacArthur Blvd., Bourne, MA 02532). The Humiscan sensor was preset to measure 0 to 100% RH (noncondensing). The accuracy of the sensor was $\pm 1\%$ at 0.5 to 90% RH and $\pm 2\%$ at 90 to 100% RH (noncondensing). The sensor operating temperature range was -40 °C to 80 °C. The Humiscan was preset to measure from -40 °C to 80 °C. The accuracy of the temperature sensor was ± 0.20 °C. The HOBO[®] accuracy was ± 0.35 °C from 0 to 50 °C and $\pm 2.5\%$ from 10 to 90 % RH. Initially, a Vaisala temperature – humidity sensor was evaluated; however, the sensor was severely affected by the chlorine dioxide and, therefore, not used further.

The sensor data were collected electronically using a portable data logging system manufactured by Omega Engineering (OMP-MODL). The system had four channels of input. The collected 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.



Figure 2. Glove Box Used for Chlorine Dioxide Material Demand Tests

Note: Glove box and exposure chamber are used interchangeably in this report.

Chlorine dioxide $(ClO_2, CAS 10049-04-4, mp -59.0 \,^{\circ}C, bp 11.0 \,^{\circ}C)$ is a strong oxidizer and must be generated at the point of use due to its instability. ClO_2 was generated using a chlorine dioxide bench-scale generator (Model "Micro") manufactured by CDG Technology, Inc. (140 Webster Street, Bethlehem, PA 18015). This system produced ClO_2 by the reaction of chlorine over dry sodium chlorite; the stoichiometry of the reaction is provided in Equation 1.

 $Cl_2 + 2NaClO_2 \rightarrow 2ClO_2 + 2NaCl$ Equation 1

Two molecules of chlorine dioxide are produced from one molecule of chlorine; the volumetric concentrations of ClO₂ = $2C_{C12} / (1 + C_{C12})$ are expressed in decimals.¹⁵ For example, the certified mixture of 4.0% chlorine in nitrogen (40,000 ppmv; 0.04 decimal concentration) used in these studies will theoretically produce 7.7% ClO₂ in nitrogen. CDG Technology claims this reaction produces no by-products, just pure chlorine dioxide gas (in nitrogen), free of chlorite ion, chlorate ion, and molecular chlorine. The results from the iodometry titrations did not agreed with the hypothetical chlorine dioxide concentration, based on flow, during the evaluation of the system. Titrations using the Occupational Safety and Health Administration (OSHA) method ID-126SGX (available at www.OSHA.gov) indicated that a small amount of chlorine was present in the feed stream. Therefore, during all tests a bubbler containing 200 mL of 25-35 % sodium chlorite was placed in the stream after the chlorine dioxide generator to eliminate any free chlorine.

The OSHA-permissible exposure limit (PEL) for ClO_2 in air is 0.1 ppm (0.3 mg/m³) as an 8-hour time-weighted average (TWA). NIOSH and ACGIH[®] short-term exposure limits

(STEL) are 0.3 ppm (0.83 mg/m³) for periods not to exceed 15 minutes and four exposures per day with each exposure separated by an interval of ≥ 60 minutes.¹⁵

The desired ClO_2 concentration was determined by manual adjustment of the chlorine–nitrogen flow rate to the flow rate of dilution air. Dilution air was provided by house air conditioned by a Miller-Nelson model HCS-401 series Flow-Temperature-Humidity Control System (Miller-Nelson Research, Inc, 8 Harris Court Building C-6, Monterey, CA 93940). The chlorine dioxide flow was controlled by a certified flow meter (Gilmont Instruments, Inc.). Material demand tests were conducted at a minimum of 25 °C and 75% RH. The total flow rate through the glove box was ~ 5.3 L/min (0.321 m³/hr).

A small recirculation fan was used in the glove box to mimic the air circulation provided by fans in commercial large-room decontamination. Prior to testing, air circulation patterns were 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 a residue inside the glove box that could interfere with the material demand studies.

The effluent from the glove box and sensors was scrubbed in sodium hydroxide and sodium thiosulfate solution and released inside a hazardous fume hood. A Scott Instruments Mini-SA portable gas detection instrument was used to monitor for CIO_2 vapor in the work area. The standard measuring range of the CIO_2 monitor was 0.00 to 2.00 ppmv CIO_2 .

A photograph of the chlorine dioxide bench-scale generator, INTERSCAN detectors, and exposure glove box is shown in (Figure 3).

Figure 3. Photograph of Chlorine Dioxide Material Demand Test Equipment



3.3 Calibration of INTERSCAN Detectors

The correlation of response to concentration was determined by plotting INTERSCAN voltage output to concentration determined by iodometry titration over a range of 0 to 4000 ppmv. The correlation was determined at or near 200, 1000, 2000, and 3500 ppmv. Feed and effluent INTERSCAN detectors were calibrated in accordance with IOP DS04017.13. The frequency of calibration was approximately one time per month. Some baseline drift required that the detectors be zeroed before the start of each test. A representative calibration curve is shown in Figure 4. The data points (diamonds) in Figure 4 were fitted using a polynomial equation.





Note: Calibration of eff uent INTERSCAN detector on 22 February 06.

3.4 Calculation of Material Surface Area

The total surface area of each material exposed to chlorine dioxide was approximately 5000 cm². The number of material coupons in the glove box per test was dependent on the coupon surface area. The sample surface area was calculated by summing the area for each exposed sample

face. For example, the wood surface area was 4863 cm² [(2 * 1 * w) + (2 * 1* h) + (2 * w * h)] using data from Table 2. The coupon surface area, total surface area per test, and the ratio of chamber volume to material surface area are provided in Table 2. The interior surface area of the chamber and coupon support was 38,766 cm².

 Table 2. Exposed Surface Area of Coupons

| Material | Coupon Surface Area (cm ²) ^a | Coupons per Test | Total Area (cm²) | Chamber Volume per Sample Surface Area (cm³/cm²) |
|-------------------------------------|--|---------------------|---------------------|--|
| Structural Wood (fir, type II) | 270 | 18 | 4863 | 65.2 |
| Concrete Block | 495 | 10 | 4952 | 64.0 |
| Painted Steel | 267 | 18 | 4798 | 66.1 |
| Latex-Painted Gypsum Wallboard | 539 | 9 | 4854 | 65.3 |
| Carpet | 600 | 8 | 4800 | 66.1 |
| Ceiling Suspension Tile, Acoustical | 586 | 8 | 4691 | 67.6 |

^a The coupon surface area was rounded to the nearest whole number for this table.

3.5 Humidity and Temperature Control

The coupons were exposed to chlorine dioxide in accordance with Section 6.0, entitled "Test Procedures for Deposition Velocity Testing," of the Deposition Velocity QAPP.⁴ The coupons were placed in the exposure glove box in accordance with IOP DS04016.12 The glove box was maintained above 75% RH using conditioned air supplied by a Miller-Nelson generator prior to the introduction of chlorine dioxide into the glove box. The temperature of the glove box was maintained above 25 °C by varying the Miller-Nelson temperature control. Additional controls were required when the room temperature fell to the point that the temperature inside the glove box could not be maintained by the Miller-Nelson generator. A heating mantel positioned beneath the glove box was sufficient for small corrections in temperature. However, a plastic tent had to be erected around the glove box and a small hot air blower used to heat the enclosure for some tests when the lab temperature was outside of the range in which the Miller-Nelson could be used to compensate.

3.6 Additional Requirements for Humidity Conditioning

Typically, materials were conditioned as required at >75% RH for 2–3 days in a plastic chamber to reduce the time required for equilibrium to occur in the glove box during a test. The materials were inspected after the humidification process; no precipitation occurred on the materials.

3.7 Fumigation Cycle

After the chamber with the coupons in place reached the desired temperature and RH, the fumigation cycle was started. An example of the overall fumigation cycle is shown in Figure 5. The three phases of the cycle are initial (or ramp-up), steady-state, and aeration. Time zero (0,0) on all graphs corresponds to the start of chlorine flow to the chlorine dioxide generator and the start of data logging by the HyperwareTM software. The combination of the initial and steady-state phases until a CT of 12,000 ppmv \cdot hr was reached can be defined as the decontamination cycle; therefore, the difference between the fumigation and decontamination cycles is the inclusion of the aeration phase in the former. The total flow through the glove box was 5.31 L/min (25 °C) for all phases. This flow rate was equivalent to ~ 1 turnover or air exchange per hour.

3.7.1 Control of Chlorine Dioxide Concentration in Exposure Chamber

The feed concentration was set between 3500 and 4000 ppmv during the ramp-up cycle of the test (T=0, effluent = 0 to the target concentration) in order to shorten the time required to reach the target concentration within the chamber. Once the target chamber concentration was achieved, the chlorine dioxide concentration within the glove box was maintained within the target concentration range of either 1000–1250 ppmv or 2000–2500 ppmv manually by adjusting the feed until the target CT of 12,000 ppmv · hr was obtained (~ 10.0 and 5.5 hr, respectively). This latter phase is the steady-state region, where the inlet and chamber chlorine dioxide concentrations are approximately constant.

The chlorine dioxide stream was controlled manually by adjusting a flow meter during the tests. The operation required constant monitoring and correction. Therefore, several artifacts, recognized as uncharacteristic spikes, appear in the affluent concentration profiles. Examples of those artifacts are found in Figures C3a, C8a, C15a, C29a, C31a, C32a, C33a, C35a, C36a, C37a, C38a, C39a, C41a, and C42a (Appendix C). In Figure C20a, an artifact spike was created by accidentally turning the INTERSCAN detector off and on. In Figures C10a and C26a, the effluent concentration profiles show spikes that exceed the target limits. The spikes were due to momentary problems with the INTERSCAN detector. The problems were self-correcting and did not invalidate the tests.

An electronic switch was evaluated as a control for the chlorine dioxide stream. The switch was either on or off when the effluent concentration exceeded limits set within the target concentration zone. The switch worked well; however, because the chlorine dioxide flow was not continuous, agreement between the feed INTERSCAN reading and the titration could not be done.

3.7.2 Aeration Cycle

After the target CT of 12,000 ppmv \cdot hr (end of test) was reached, the feed was stopped and aeration of the glove box with air (~ 5.35 L/min, > 25 °C and > 75 % RH) continued until the ClO₂ concentration fell to a safe level (nondetect). Chlorine dioxide concentration within the glove box was monitored for > 20 hours. The concentration (~0 ppmv at t \geq 20 hr) was lower than the criterion of 10% of the target, which defined the end of the run. The concentration was then considered safe by the Risk Reduction Office to open the glove box to remove the specimens. The procedures for safely opening the glove box and coupon removal after fumigant exposure were documented in Standard Operating Procedure (SOP) RNG-108¹⁶ and IOP DS04014.¹⁷



4. DATA REVIEW AND TECHNICAL SYSTEMS AUDITS

The approved Deposition Velocity QAPP specified procedures for the review of data and independent technical system audits.⁴ Test data (Excel worksheets created for each test, which contained material information, affluent and effluent concentrations and CT, exposure chamber temperature and humidity, detector and titration comparisons) were peer reviewed within two weeks of data 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 results of the audits are discussed in Section 8.0, "Quality Assurance Findings."

5. MATERIAL DEMAND CALCULATIONS AND DESCRIPTIVE STATISTICAL ANALYSIS

This section of the report provides details on the calculation of the material demand and the statistical tools used in the analysis of the data. The focus of this section is on only the ramp-up and steady-state phases of the fumigation cycle. Thus, the material demand for each building material type is calculated as an average over the fumigation cycle duration required to reach the target 12,000 ppmv · hr.

5.1 Material Demand Calculations

The difference between the target chamber outlet CT (CT_{outlet} , in ppmv · hr) and the inlet CT (CTinlet, ppmv · hr) required to achieve the target (12,000 ppmv · hr) at the target chamber concentration (1000 ppmv or 2000 ppmv) can be attributed to the demand of the material in the chamber for ClO₂. This demand is composed of reversible adsorption (e.g., physisorption) and chemical reaction (e.g., decomposition or chemisorption) on the materials within the chamber. A contribution of homogeneous decomposition (gas-phase decomposition) may also be present; however, efforts were made to minimize the contribution of this mechanism (e.g., turnover rate and shielding from UV light). The mass balance for the chamber can be expressed as:

$$W_i - W_o = W_c + W_{md}$$
 Equation 2

where,

 W_i = the total amount of ClO_2 that entered the chamber via the inlet flow;

 W_0 = the total amount of ClO₂ that was removed from the chamber by the exit flow;

 $W_c =$ the total amount of ClO₂ remaining in the chamber; and

 W_{md} = the total amount of ClO₂ adsorbed and/or consumed by the material (and/or chamber).

The difference between the inlet and outlet chamber concentrations in the empty chamber (ΔCT_{μ}) and with materials in the chamber (ΔCT_{mb}) can be determined by subtracting CT_{inlet} from CT_{outlet} for each experiment. These values represent the CT added over the target CT at the time the target CT was achieved. An illustration of this calculation is shown in Figure 6. These differences do not correct for the amount of ClO, left in the chamber (W) at the point in time that the target CT is achieved; i.e., the difference in CT is not due entirely to the loss (i.e., material demand) of ClO₂. In order to correct for this, the theoretical aeration curve starting at the chamber concentration at the time the target CT was achieved can be integrated to determine CT. This value represents the concentration and time product $(ppmv \cdot hr)$ removed via aeration at the conclusion of the test and not lost due to material demand. Since it is the theoretical aeration curve, it is normalized for all experiments since the experimental conditions remained consistent (e.g., size of the chamber, air exchange rate). Accounting for the loss to aeration in each experiment, the difference between the inlet and outlet can be expressed as $\Delta CT_{ha} = \Delta CT_{h} - CT_{a}$ and $\Delta CT_{mb-a} (= \Delta CT_{mb} - CT_{a})$ for the baseline and materials, respectively. CT_{a} is calculated specific to each experiment (i.e., using the exact concentrations in the chamber at the time the target CT was achieved). However, due to the insignificant differences in these concentrations, a single

 CT_a could have also been used for each concentration (e.g., 1000 and 2000 ppmv) without adding any significant error to the calculation. For the demand of each material, CT_a does not practically factor into the calculation due to essentially

canceling out. However, for the baseline acrylic chamber, the demand determined using this approach is specific to the size of the chamber and the experimental parameters.





The impact of each material type on achieving the target fumigant concentration in the chamber can be determined by subtracting the observed difference in CT in the baseline tests corrected for the loss to aeration in that test (ΔCT_{b-a}) from that observed with a specific material type in the chamber (ΔCT_{mb-a}) . This is shown in Equation 3, where ΔCT_k is the difference between the target CT and input CT required to achieve the target CT at the target chamber concentration attributed directly to the impact of the material.

$$\Delta CT_{k} = \Delta CT_{mb-a} - \Delta CT_{b-a} = (CT_{outlet} - CT_{inlet} - CT_{a})_{mb} - (CT_{outlet} - CT_{inlet} - CT_{a})_{b}$$

Equation 3

It should be noted that ΔCT_{mb} and ΔCT_{b} are the differences in the outlet and inlet CT values without subtracting the loss to aeration.

The surface area specific material demand for each material (MD_k) over the fumigation period (up to 12,0000 ppmv \cdot hr) can be calculated according to Equation 4, where ΔCT_k is divided by the material surface area (A, in m²) and the time (t, in hr) required to reach the target CT_{outlet} . A similar expression can be used for the material demand of the baseline chamber (MD_b). The units of MD are ppmv \cdot hr per hr per m². The total surface area added to the chamber for each material type is reported in Table 2. The total interior surface area of the chamber and material support structures is 3.8766 m².

$$MD_{k} = \frac{\Delta CT_{k}}{tA} \text{ (for materials),}$$
$$MD_{b} = \frac{\Delta CT_{b-a}}{tA} \text{ (for baseline)} \text{ Equation 4}$$

The material demand can also (and more traditionally) be expressed as a time-average mass flux to the material surface. This can more traditionally be determined using Equation 2 and converting the volume concentration units (ppmv) to mass concentration (e.g., g/m^3). For the purpose of this report, CT was converted from volume units (ppmv \cdot hr) to mass concentration units ($g \cdot$ hr/m³) according to Equation 5:

$$CT_{mass} = \frac{CT_{ppmv-hr}MW_{CIO_2}P_{sys}}{1000RT_{sys}}$$
 Equation 5

where,

- CT_{mass} = the cumulative mass concentration of ClO_2 over a defined time period (g · hr/m³);
- CT_{ppmv} = the cumulative volume concentration of ClO_2 over a defined time period (ppmv · hr);

 MW_{CIO_2} = molecular weight of ClO_2 (67.5 g/mole);

P_{sys} = chamber pressure (in units of atmosphere [atm]);

- R = universal gas constant (0.0826 L atm/mole K); and
- T_{svs} = chamber temperature (in units of K).

The mass flux (J) for each material can then be calculated according to Equation 6:

$$J = \frac{\Delta CT_{k(mass)}Q}{tA}$$
 Equation 6

where,

J is in units of $g \cdot hr^{-1} \cdot m^{-2}$;

Q = the inlet and outlet flow rate (equal for all experiments) = $0.319 \text{ m}^3/\text{hr}$ at 25 °C;

t = time (in hr) to reach target CT of 12,000 ppmv \cdot hr; and

A = exposed coupon surface area (in m^2).

A similar expression can be used to determine the mass flux to the empty chamber surfaces (i.e., baseline) by replacing $\Delta CT_{k(mass)}$ with $\Delta CT_{b-a(mass)}$. J_k is denoted as the mass flux to the materials, and J_b is the mass flux to the chamber surfaces.

 $\begin{array}{l} \Delta CT_{k(mass)} \text{ is determined by first converting } \Delta CT_{mb-a} \text{ and } \\ \Delta CT_{b-a} \text{ from ppmv} \cdot \text{hr to mass concentration units according to Equation 4 and then subtracting these values to obtain the background (baseline) corrected mass concentration and time product difference between inlet and outlet ClO₂ in units of g \cdot \text{hr}^1 \cdot \text{m}^{-3}. \end{array}$

This time-averaged material demand assumes that the adsorption and consumption of ClO_2 by a material is relatively constant over the time period defined as t. For materials showing a high initial adsorption amount and limited reaction of ClO_2 on the material, this assumption will become less valid with increasing time. In this stated case, the material demand will occur over an initial period and the material will have little to no further demand with increasing time. Since the inlet concentration of ClO_2 was adjusted to decrease the time needed to reach the target chamber concentration (1000 or 2000 ppmv), an analysis of the change in material demand with time over this period is not possible.

5.2 Descriptive Statistical Analysis

The average and standard deviation (SD) were calculated for three replicates for each material and ClO₂ concentration. Data were processed in Microsoft® Office Excel 2003 SP2 and rounded to the nearest tenth. The error propagation was determined for all arithmetic calculations.¹⁸ The determination of statistical outliers was performed according to the Grubb's test, also known as the extreme studentized deviate (ESD) method. No data were discarded as an outlier with a data set (i.e., a set of triplicate experiments at each concentration for each material). Statistical comparisons between the data sets were then performed using the Student's t Test calculator available from graphpad.com. All statistical probabilities (p_{values}) were determined for an unpaired test at a confidence interval $\alpha = 0.05$. The p_{values} represent the probability (ranging from zero to one) that the difference between sample means is unlikely to be a coincidence; i.e., how much evidence exists that the null hypothesis is not true. However, the p_{value} is not the

probability that the null hypothesis is true. A two-tail p_{value} was used for this testing; this approach is used to determine the chance that randomly selected samples could have means at least as far apart as observed if the null hypothesis were true. The null hypothesis for this work is that there is no difference in the means of the test groups; i.e., the means are likely from the same population. The magnitude of the p_{value} is used to indicate whether the two means might be from the same population; the traditional criteria of rejecting the hypothesis if the p_{value} was less than 0.05 was used for this analysis. A small p_{value} (e.g., below 0.05) is evidence against the null hypothesis; in other words, a small p_{value} is an indication that the difference of the means of the two populations is statistically significant.

A large p_{value} may suggest that the null hypothesis is true; however, other factors may also contribute and the evidence should, therefore, be automatically taken to indicate the truth of the hypothesis. The 95% confidence level ($\alpha = 0.05$) used in this study can provide additional evidence against the null hypothesis in the case of large p_{values} . To further support the acceptance of the null hypothesis, the 95% confidence interval should lie entirely within the range of indifference. A confidence interval of 95% means that there is a 95% chance that the calculated interval included the true difference between the population means.

6.0 RESULTS

6.1 Evaluation of Empty Glove Box

The empty glove box, defined as not having the coupons to be tested in place, was evaluated for mixing and for establishing a baseline effect on ClO_2 . The mixing evaluation was done to ensure that all the coupons experienced the same concentration of ClO_2 and that the chamber could indeed be considered a well-stirred chambered. The baseline material demand studies were done in order to be able to isolate the impact of the building materials from that of the chamber in subsequent testing (Section 6.2).

6.1.1 "Fog" Test Results and Discussion

A "fog" test was conducted to observe the glove box air circulation pattern created by the glove box recirculation fan. The small recirculation fan was used in the glove box to mimic the air circulation provided by fans in commercial large-room decontamination. The fan was placed on the bottom of the glove box in the back right corner and blew toward the opposite corner of the glove box. The "fog" test was used to verify that the coupons placed on the exposure rack would have decontaminant vapor contact during testing. A container of dry ice and warm water was placed in the glove box. The fog produced could be sustained for several minutes. Air was introduced into the glove box on the lower right side and the flow observed. Figure 7 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.

Figure 7. Exposure Chamber Fog Test



Note: Figures (a), (b), (c) are close-up photographs of different areas (a, b, c) of the chamber, as illustrated in the upper left corner photo. The close-up photos are intended to provide an indication of how appropriate mixing was assessed during the fog test.

6.1.2 Material Demand of the Baseline Chamber Three baseline tests were conducted for each chlorine dioxide concentration of 1000 and 2000 ppmv. The sample rack without material coupons was in the glove box during the baseline tests. For the first three tests, the titration verification of INTERSCAN readings was performed at the beginning, middle, and end of run. Afterwards, the titration check was performed only at target CT/2. Three replicate samples were collected from the feed and effluent streams and assayed in accordance with IOP DS04002.14 The concentration of ClO₂ was initially calculated in mg/mL and then converted to ppmv. The data were recorded in an Excel worksheet specific to that test for peer review and validation (reference section 4). The maximum acceptance criterion for the agreement between detector and titration was \pm 15%. An example of the detector and titration results is provided in Table D1, Appendix D.

The overall effect of the baseline chamber (without materials included) on maintaining the desired ClO_2 concentration can be determined from the data presented in Table 3. The data in the table includes (in order from left to right) the average chamber temperature, average concentration of ClO_2 in the feed to the chamber, the time to achieve a chamber CT of 12,000 ppmv \cdot hr, the difference between the total inlet and

chamber cumulative CT values corrected for the amount remaining in the chamber at the target CT, the material demand calculated according to Equation 4, and the mass flux calculated according to Equation 6. The average values for these columns described above for the three runs at each condition (1000 ppmv and 2000 ppmv target chamber concentrations) are presented together with the corresponding standard deviations (\pm SD). The target CT value for all experiments was 12,000 ppmv · hr. There was no statistical difference between the input CT required to reach the target CT value at chamber concentrations of 1000 ppmv compared to 2000 ppmv at a confidence interval of $\alpha = 0.05$ (p_{value} = 0.0537). On average, 6% more ClO₂ was required to be added to the chamber than the amount required for the target CT. The average material demand (MD_{μ}) of the baseline chamber and mass flux to the chamber surfaces (J_{h}) over the time required to achieve the 12,000 ppmv · hr can be determined as outlined in Section 5.1 (Equations 4 and 6, respectively). The difference in CT between the target and required inlet for each experimental fumigation test can be found in Table E1 of Appendix E. Concentration profiles (Figures C1a – C6a) and CT profiles (Figures C1b - C6b) are provided in Appendix C.

| Table | 3. | Baseline | Material | Demand | Test Results |
|-------|----|----------|----------|--------|--------------|
|-------|----|----------|----------|--------|--------------|

| Tests | Average Chamber Temperature (°C) | Average Feed Concentration (ppmv) | Time (t, in hr) to reach target CT | ∆CT _{ь-a} (ppmv-hr) | MD _b (ppmv-hr/hr m²) | Ј _ь (g/hr m²) |
|--|---|--|---|---------------------------------|---------------------------------------|--|
| Baseline (1000 ppmv) Baseline (2000 ppmv) | 29.2 ± 0.2 27.2 ± 1.2 | $\begin{array}{c} 1278.7 \pm 50.6 \\ 2327.4 \pm 246.0 \end{array}$ | 10.99 ± 0.43 6.22 ± 0.27 | 896.2 ± 208.3 764.5 ± 370.8 | 21.0 ± 5.0 31.7 ± 15.4 | $\begin{array}{c} 0.02 \pm 0.004 \\ 0.03 \pm 0.01 \end{array}$ |

6.2 Material Demand of the Selected Building Materials

Three replicate tests were conducted at each of the two target concentrations (1000 ppmv and 2000 ppmv) for each of the six building material types investigated. The concentrationtime and CT-time plots for each of the experiments are included in Appendix C. The baseline-corrected average differences between the inlet and outlet $CT (\Delta CT_{\mu})$ for each material type are reported in Tables 4 and 5 for each of the target chamber concentrations tested. The baseline-corrected average material demands (MD_{μ}) and mass fluxes to the material surfaces (J_{ν}) are also reported in Tables 4 and 5. The data in the table includes (in order from left to right) the average chamber temperature, average concentration of ClO₂ in the feed to the chamber, the time to achieve a chamber CT of 12,000 ppmv \cdot hr, the baseline-subtracted difference between the total inlet and chamber cumulative CT values, the material demand calculated according to Equation 4, and the mass flux calculated according to Equation 6. The average values for these columns described above for the

three runs at each condition (1000 ppmv and 2000 ppmv target chamber concentrations) are presented together with the corresponding standard deviations (\pm SD). The inlet and outlet CT values for each experiment used for the calculation of the average values presented in Tables 4 and 5 can be found in Table E2 of Appendix E.

The baseline-corrected impact of the materials on the CIO_2 feed required to achieve the target conditions can be determined by a comparison of the material demand or mass flux between each material type and the baseline, and among each material type. This can be done for both the 1000 ppmv and 2000 ppmv target chamber CIO_2 concentrations. In addition, a comparison of the material demand or mass flux for a single material at the 1000 ppmv and 2000 ppmv conditions may also indicate the extent of the impact of the material on the required CIO_2 feed to the chamber to achieve and maintain the target concentration. The following subsections of Section 6.2 discuss these comparisons and the statistical significances of the difference observed.

Table 4. Baseline-Corrected Material Demand Test Results for Each Material Type (Target Chamber Concentration = 1000 ppmv CIO₂)

| Tests | Average Chamber Temperature (°C) | Average Feed Concentration (ppmv) | Time (t, in hr) to reach target CT | ∆CT _k (ppmv-hr) | MD _k (ppmv-hr/hr m²) | J _k (g/hr m²) |
|----------------|---|---|---|-------------------------------|------------------------------------|-----------------------------|
| Carpet | 27.2 ± 0.4 | 1489.6 ± 26.8 | 10.77 ± 0.07 | 1960.7 ± 278.2 | 379.3 ± 53.9 | 0.33 ± 0.05 |
| Steel | 26.0 ± 0.3 | 1125.9 ± 45.4 | 10.85 ± 0.22 | -1832.0 ± 300.0 | -351.9 ±58.1 | -0.31 ± 0.05 |
| Wallboard | 28.4 ± 1.9 | 1683.4 ± 30.7 | 10.62 ± 0.06 | 3801.7 ± 397.1 | 737.5 ± 77.1 | 0.64 ± 0.07 |
| Ceiling Tile | 28.3 ± 0.2 | 2146.8 ± 42.9 | 10.84 ± 0.07 | 9213.5 ± 560.4 | 1811.9 ± 110.8 | 1.58 ± 0.10 |
| Wood | 29.6 ± 0.3 | 1770.2 ± 59.8 | 10.65 ± 0.14 | 4774.9 ± 769.0 | 922.0 ± 149.0 | 0.80 ± 0.13 |
| Concrete Block | 28.5 ± 0.6 | 1375.4 ± 42.3 | 10.91 ± 0.16 | 833.4 ± 615.8 | 154.3 ± 114.0 | 0.13 ± 0.10 |

Table 5. Baseline-Corrected Material Demand Test Results for Each Material Type(Target Chamber Concentration = 2000 ppmv ClO₂)

| Tests | Average Chamber Temperature (°C) | Average Feed Concentration (ppmv) | Time (t, in hr) to reach target CT | ∆CT _k (ppmv-hr) | MD _k (ppmv-hr/hr m²) | J _k (g/hr m²) |
|----------------|---|---|---|-------------------------------|------------------------------------|-----------------------------|
| Carpet | 26.9 ± 1.2 | 2978.2 ± 246.0 | 5.94 ± 0.12 | 2609.0 ± 784.7 | 915.1 ± 275.8 | 0.80 ± 0.24 |
| Steel | 25.7 ± 0.1 | 2342.7 ± 42.6 | 5.97 ± 0.12 | -1056.2 ± 619.4 | -368.7 ± 216.4 | -0.32 ± 0.19 |
| Wallboard | 27.6 ± 0.7 | 3353.9 ± 108.4 | 6.02 ± 0.09 | 5137.8 ± 530.7 | 1758.3 ± 183.5 | 1.53 ± 0.16 |
| Ceiling Tile | 29.4 ± 1.2 | 3646.7 ± 61.8 | 7.07 ± 0.21 | 10612.3 ± 821.5 | 3199.8 ± 265.3 | 2.79 ± 0.23 |
| Wood | 30.0 ± 0.4 | 2675.9 ± 42.6 | 6.24 ± 0.06 | 1582.7 ± 726.6 | 521.6 ± 239.6 | 0.45 ± 0.21 |
| Concrete Block | 26.2 ± 0.9 | 2327.1 ± 77.3 | 6.22 ± 0.17 | -378.0 ± 613.1 | -122.7 ± 199.1 | -0.11 ± 0.17 |

6.2.1 Carpet

The average difference between the inlet and outlet chamber CT values for the three 1000 ppmv tests at the time the target CT was achieved was $\Delta CT_{mb} = 4015.9.5 \pm 191.8 \text{ ppmv} \cdot$ hr, as listed in Table E2 of Appendix E. Corrected for the amount of fumigant remaining in the chamber at this time, the difference due to material demand was $\Delta CT_{mb-a} = 2856.9$ \pm 183.3 ppmv \cdot hr (Appendix E, Table E2). An extremely significant ($p_{value} = 0.0006$) difference between the baseline (ΔCT_{b-a}) and tests with carpet in the chamber (ΔCT_{mb-a}) was observed, indicating that the carpet had a statistically significant impact on the concentration of ClO₂ within the chamber. The difference in CT after baseline subtraction was $\Delta CT_k = 1960.7 \pm 278.2$ ppmv · hr, as reported in Table 4. Converted to a volume or mass flux, the material demand can be reported as $MD_k = 379.3 \pm 53.9 \text{ ppmv} \cdot \text{hr} \cdot \text{hr}^1$ \cdot m⁻² and J₁ = 0.33 ± 0.05 g \cdot hr⁻¹ \cdot m⁻², respectively. The differences between these values and those reported for the corresponding baseline tests (Table 3) are statistically significant ($p_{value} = 0.0003$ for MD_k and 0.0051 for J_k). The time required to achieve the target CT was not statistically different from that observed for the baseline tests at 1000 ppmv. Concentration profiles (Figures C7a – C9a) and CT profiles (Figures C7b – C9b) are provided in Appendix C.

The average difference between the inlet and outlet chamber CT values for the three 2000 ppmv tests was $\Delta CT_{mb} = 5625.9$ \pm 668.8 ppmv \cdot hr (Appendix E, Table E2). Corrected for the amount of fumigant remaining in the chamber at the time the target CT was achieved, the difference due to material demand was $\Delta CT_{mb-a} = 3373.5 \pm 691.6 \text{ ppmv} \cdot \text{hr}$ (Appendix E, Table E2). As in the 1000 ppmv tests, this value was determined to be very statistically different (p_{value} = 0.0045) from that of the baseline tests (CT_{h-a}) reported in Table 3. The difference in CT after baseline subtraction was $\Delta CT_{\mu} = 2609.0 \pm 784.7 \text{ ppmv} \cdot \text{hr}$, as reported in Table 5. Converted to a volume or mass flux, the material demand can be reported as $MD_k = 915.1 \pm 275.8 \text{ ppmv} \cdot \text{hr} \cdot \text{hr}^{-1}$ \cdot m⁻² and J_k = 0.80 ± 0.24 g \cdot hr⁻¹ \cdot m⁻², respectively. The differences between these values and those reported for the corresponding baseline tests (Table 3) are statistically significant ($p_{value} = 0.0052$ for MD_k and 0.0004 for J_k). The

difference in CT_k of carpet at 1000 ppmv and 2000 ppmv was not statistically significant ($p_{value} = 0.2487$). However, when converted to a volume or mass flux (i.e., normalized for the fumigation time), the differences in material demand (MD_k) and mass flux (J_k) at the two concentrations were statically significant; this indicates that the material demand (and mass flux) is likely a function of the concentration (i.e., not zero order in concentration). The time required to achieve the target CT was not statistically different from that observed for the baseline tests at 2000 ppmv. Concentration profiles (Figures C10a – C12a) and CT profiles (Figures C10b – C12b) are provided in Appendix C.

6.2.2 Painted Steel

The average difference between the inlet and outlet chamber CT values for the three 1000 ppmv tests at the time the target CT was achieved was $\Delta CT_{mb} = 199.0 \pm 292.8$ ppmv · hr, as listed in Table E2 of Appendix E. Corrected for the amount of fumigant remaining in the chamber at this time, the difference due to material demand was $\Delta CT_{mb-a} = -937.7$ ± 215.0 ppmv \cdot hr (Appendix E, Table E2). An extremely significant ($p_{value} = 0.0005$) difference between the baseline $(\Delta CT_{h,a})$ and tests with painted steel in the chamber $(\Delta CT_{mb,a})$ was observed. The difference in CT between the inlet and chamber after baseline subtraction was $\Delta CT_{k} = -1832.0$ \pm 300.0 ppmv \cdot hr, as reported in Table 4. Converted to a volume or mass flux, the material demand can be reported as $MD_{\mu} = -351.9 \pm 58.1 \text{ ppmv} \cdot \text{hr} \cdot \text{hr}^{-1} \cdot \text{m}^{-2}$ and $J_{\mu} = -0.31 \pm$ $0.05 \text{ g} \cdot \text{hr}^{-1} \cdot \text{m}^{-2}$, respectively. The differences between these values and those reported for the corresponding baseline tests (Table 3) are statistically significant ($p_{value} = 0.0004$ for MD_{ν} and 0.0333 for J_{ν}). However, a negative demand does not make physical sense since this would mean that additional ClO₂ was generated within the chamber due to the presence of the painted steel. This was certainly not the case and is an artifact of the measurement method; the minor demand of the painted steel is within the limits of detection of the experimental method used. Further tests would need to be performed to understand this response. This result is discussed further in Section 6.4. The time required to achieve the target CT was not statistically different from that observed for the baseline tests at 1000 ppmv. Concentration

profiles (Figures C13a - C15a) and CT profiles (Figures C13b - C15b) are provided in Appendix C.

The average difference between the inlet and outlet chamber CT values for the three 2000 ppmv tests was $\Delta CT_{mb} = 1964.0$ \pm 446.6 ppmv \cdot hr (Appendix E, Table E2). Corrected for the amount of fumigant remaining in the chamber at the time the target CT was achieved, the difference due to material demand was $\Delta CT_{mb-a} = -291.7 \pm 496.2 \text{ ppmv} \cdot \text{hr}$ (Appendix E, Table E2). The difference between this value and that determined for the baseline tests (ΔCT_{h-a} , Table 3) was determined to be statistically significant ($p_{value} =$ 0.0418). The difference in CT after baseline subtraction was $\Delta CT_{L} = -1056.2 \pm 619.4 \text{ ppmv} \cdot \text{hr}$, as reported in Table 5. Converted to a volume or mass flux, the material demand can be reported as $MD_{k} = -368.7 \pm 216.4 \text{ ppmv} \cdot \text{hr} \cdot \text{hr}^{-1}$. m^{-2} and $J_{k} = -0.32 \pm 0.19 \text{ g} \cdot \text{hr}^{-1} \cdot m^{-2}$, respectively. The differences between these values and those reported for the corresponding baseline tests (Table 3) are statistically significant ($p_{value} = 0.033$ for MD_k and 0.0003 for J_k). The difference in CT (Δ CT_k) due to the material was not determined to be statistically significantly different from the baseline-corrected average difference observed in the 1000 ppmv tests ($P_{value} = 0.1224$). Similarly, the material demand (MD_{k}) and mass flux (J_{k}) determined at 1000 ppmv and 2000 ppmv are not statistically significantly different (P_{value} = 0.9029 for MD_k and P_{value} = 0.934 for J_k). As discussed previously, the negative demand (or flux) is likely an artifact of the testing and not due to additional generation of ClO, due to the painted steel in the chamber. The average time required to achieve the target CT at 2000 ppmv was not statistically different from the average time observed for the respective baseline tests. Concentration profiles (Figures C16a – C18a) and CT profiles (Figures C16b – C18b) for the 2000 ppmv tests are provided in Appendix C.

6.2.3 Gypsum Wallboard

The average difference between the inlet and outlet chamber CT values for the three 1000 ppmv tests at the time the target CT was achieved was $\Delta CT_{mb} = 5856.3 \pm 331.4$ ppmv · hr, as listed in Table E2 of Appendix E. Corrected for the amount of fumigant remaining in the chamber at this time, the difference due to material demand was $\Delta CT_{mb-a} = 4697.9$ \pm 337.5 ppmv · hr (Appendix E, Table E2). An extremely significant ($p_{value} = 0.0001$) difference between the baseline $(\Delta CT_{h_{a}})$ and tests with painted wallboard in the chamber (ΔCT_{mb}) was observed, indicating that the material had a statistically significant impact on the concentration of ClO within the chamber. The difference in CT after baseline subtraction was $\Delta CT_k = 3801.7 \pm 397.1$ ppmv · hr, as reported in Table 4. Converted to a volume or mass flux, the material demand can be reported as $MD_k = 737.5 \pm 77.1 \text{ ppmv} \cdot \text{hr}$ \cdot hr⁻¹ \cdot m⁻² and J₁ = 0.64 ± 0.07 g \cdot hr⁻¹ \cdot m⁻², respectively. The differences between these values and those reported for the corresponding baseline tests (Table 3) are extremely statistically significant ($p_{value} = 0.0001$ for MD_k and 0.0001 for J_{μ}). The time required to achieve the target CT was not statistically different from that observed for the baseline tests at 1000 ppmv. Concentration profiles (Figures C19a – C21a) and CT profiles (Figures C19b - C21b) are provided in Appendix C.

The average difference between the inlet and outlet chamber CT values for the three 2000 ppmv tests was $\Delta CT_{mb} = 8135.7$ \pm 419.1 ppmv \cdot hr (Appendix E, Table E2). Corrected for the amount of fumigant remaining in the chamber at the time the target CT was achieved, the difference due to material demand was $\Delta CT_{mb-a} = 5902.2 \pm 530.7 \text{ ppmv} \cdot \text{hr}$ (Appendix E, Table E2). As in the 1000 ppmv tests, this value was determined to be extremely statistically different (p_{value} = 0.0001) from that of the baseline tests (ΔCT_{ha}) reported in Table 3. The difference in CT after baseline subtraction was $\Delta CT_{k} = 5137.8 \pm 530.7 \text{ ppmv} \cdot \text{hr}$, as reported in Table 5. Converted to a volume or mass flux, the material demand can be reported as $MD_{k} = 1768.3 \pm 183.5 \text{ ppmv} \cdot \text{hr} \cdot \text{hr}^{-1} \cdot \text{m}^{-2}$ and $J_{k} = 1.58 \pm 0.16 \text{ g} \cdot \text{hr}^{-1} \cdot \text{m}^{-2}$, respectively. The differences between these values (MD $_{k}$, and J $_{k}$) and those reported for the corresponding baseline tests (Table 3) are extremely statistically significant ($p_{value} = 0.0001$ for MD_k , and 0.0001 for J_k). The difference in CT_k of painted wallboard at 1000 ppmv and 2000 ppmv was statistically significant (p_{value} = 0.0251); similarly, the material demand and mass flux at 1000 ppmv were statistically different ($p_{value} = 0.0009$ for MD_k and $p_{value} = 0.0009$ for J_k) from the demand and flux at 2000 ppmv. These results suggest a nonzero dependence of the demand on the chamber concentration. The time required to achieve the target CT was not statistically different from that observed for the baseline tests at 2000 ppmv. Concentration profiles (Figures C22a - C24a) and CT profiles (Figures C22b - C24b) are provided in Appendix C.

6.2.4 Ceiling Tile

The average difference between the inlet and outlet chamber CT values for the three 1000 ppmv tests at the target CT was achieved was $\Delta CT_{mb} = 11254.4 \pm 552.9 \text{ ppmv} \cdot \text{hr}$, as listed in Table E2 of Appendix E. Corrected for the amount of fumigant remaining in the chamber at this time, the difference due to material demand was $\Delta CT_{mb-a} = 10109.7 \pm 519.9$ ppmv · hr (Appendix E, Table E2). This value was extremely statistically different ($p_{value} = 0.0001$) from the baseline (ΔCT_{h_a}) test results reported in Table 3; this difference indicates that the ceiling tile had a pronounced impact on the concentration of ClO₂ within the chamber. The difference in CT after baseline subtraction was $\Delta CT_k = 9213.5 \pm 560.4$ ppmv · hr, as reported in Table 4. Converted to a volume or mass flux, the material demand can be reported as $MD_{\mu} =$ $1811.9 \pm 110.8 \text{ ppmv} \cdot \text{hr} \cdot \text{hr}^{-1} \cdot \text{m}^{-2} \text{ and } \text{J}_{\perp} = 1.58 \pm 0.10 \text{ g} \cdot \text{m}^{-2}$ $hr^{-1} \cdot m^{-2}$, respectively. The differences between these values and those reported for the corresponding baseline tests (Table 3) are extremely statistically significant ($p_{value} = 0.0001$ for MD_k and 0.0001 for J_k). The time required to achieve the target CT was not statistically different from that observed for the baseline tests at 1000 ppmv. Concentration profiles (Figures C25a – C27a) and CT profiles (Figures C25b – C27b) are provided in Appendix C.

The average difference between the inlet and outlet chamber CT values for the three 2000 ppmv tests was $\Delta CT_{mb} = 13723.1 \pm 745.9$ ppmv \cdot hr (Appendix E, Table E2). Corrected for the amount of fumigant remaining in the chamber at the time the target CT was achieved, the difference due to material demand was $\Delta CT_{mb-a} = 11376.8$ \pm 733.1 ppmv · hr (Appendix E, Table E2). As in the 1000 ppmv tests, this value was determined to be extremely statistically different ($p_{value} = 0.0001$) from that of the baseline tests (ΔCT_{b-a}) reported in Table 3. The difference in CT after baseline subtraction was $\Delta CT_{\mu} = 10612.3 \pm 821.5 \text{ ppmv} \cdot \text{hr}$, as reported in Table 5. Converted to a volume or mass flux, the material demand can be reported as $MD_{L} = 3199.8 \pm$ 265.3 ppmv \cdot hr \cdot hr⁻¹ \cdot m⁻² and J₁ = 2.79 ± 0.23 g \cdot hr⁻¹ \cdot m⁻², respectively. The differences between these values (MD₁, and J_{ν}) and those reported for the corresponding baseline tests (Table 3) are extremely statistically significant ($p_{value} = 0.0001$ for MD_k, and 0.0001 for J_k). The difference in CT_k of ceiling tile at 1000 ppmv and 2000 ppmv was not quite statistically significant ($p_{value} = 0.0715$); however, the material demand and mass flux at 1000 ppmv were very statistically different $(p_{value} = 0.0011 \text{ for } MD_k \text{ and } p_{value} = 0.0011 \text{ for } J_k) \text{ from}$ the demand and flux at 2000 ppmv. Concentration profiles (Figures C28a - C30a) and CT profiles (Figures C28b -C30b) are provided in Appendix C.

6.2.5 Wood

The average difference between the inlet and outlet chamber CT values for the three 1000 ppmv tests at the time the target CT was achieved was $\Delta CT_{mb} = 6817.9 \pm 707.1 \text{ ppmv} \cdot \text{hr}$, as listed in Table E2 of Appendix E. Corrected for the amount of fumigant remaining in the chamber at this time, the difference due to material demand was $\Delta CT_{mb-a} = 5671.1 \pm 740.0 \text{ ppmv}$ \cdot hr (Appendix E, Table E2). An extremely significant ($p_{value} =$ 0.0004) difference between the baseline (Δ CTb-a) and tests with wood in the chamber (ΔCT_{mb-a}) was observed, indicating that the material had a statistically significant impact on the concentration of ClO₂ within the chamber. The difference in CT after baseline subtraction was $\Delta CT_{\mu} = 4774.9 \pm 769.0$ ppmv \cdot hr, as reported in Table 4. Converted to a volume or mass flux, the material demand can be reported as $MD_{L} =$ $922.0 \pm 149.0 \text{ ppmv} \cdot \text{hr} \cdot \text{hr}^{-1} \cdot \text{m}^{-2} \text{ and } \text{Jk} = 0.80 \pm 0.13 \text{ g} \cdot \text{m}^{-2}$ $hr^{-1} \cdot m^{-2}$, respectively. The differences between these values and those reported for the corresponding baseline tests (Table 3) are statistically significant ($p_{value} = 0.0005$ for MD_k and 0.0258 for J_{μ}). The time required to achieve the target CT was not statistically different from that observed for the baseline tests at 1000 ppmv. Concentration profiles (Figures C31a -C33a) and CT profiles (Figures C31b – C33b) are provided in Appendix C.

The average difference between the inlet and outlet chamber CT values for the three 2000 ppmv tests was $\Delta CT_{mb} = 4638.3 \pm 637.4$ ppmv \cdot hr (Appendix E, Table E2). Corrected for the amount of fumigant remaining in the chamber at this time, the difference due to material demand was $\Delta CT_{mb-a} = 2347.1 \pm 624.8$ ppmv \cdot hr (Appendix E, Table E2). As in the 1000 ppmv tests, this value was determined to be statistically different ($p_{value} = 0.0196$) from that of the baseline test results (ΔCT_{b-a}) reported in Table 3. The difference in CT after baseline subtraction was $\Delta CT_k = 1582.7 \pm 726.6$ ppmv \cdot hr, as reported in Table 5. Converted to a volume or mass flux, the material demand can be reported as MD_k = 521.6 \pm 239.6 ppmv \cdot hr \cdot hr⁻¹ \cdot m⁻² and J_k = 0.45 \pm 0.21 g \cdot hr⁻¹ \cdot m⁻², respectively. The differences between these values (MD_k and J_k) and those reported for the corresponding baseline tests

(Table 3) are statistically significant ($p_{value} = 0.0241$ for MD_k and 0.0005 for J_k). The baseline-corrected difference between the inlet and chamber CT from the 1000 and 2000 ppmv tests were determined to be statistically different ($p_{value} = 0.0064$). However, the material demand (MD_k) and mass flux (J_k) of the wood for ClO₂ at a target concentration of 1000 ppmv was not different ($p_{value} = 0.0695$ for MD_k and $p_{value} = 0.0701$ for J_k) from the demand at 2000 ppmv. The fact that these normalized (for time and surface area) values are not different potentially indicates that the demand is not highly dependent on chamber concentration. The time required to achieve the target CT was not statistically different from that observed for the baseline tests at 2000 ppmv. Concentration profiles (Figures C34a – C36a) and CT profiles (Figures C34b – C36b) are provided in Appendix C.

6.2.6 Concrete

The average difference between the inlet and outlet chamber CT values for the three tests at a target chamber concentration of 1000 ppmv at the time the target CT was achieved was $\Delta CT_{mb} = 2904.9 \pm 581.7 \text{ ppmv} \cdot \text{hr}$, as listed in Table E2 of Appendix E. Corrected for the amount of fumigant remaining in the chamber at this time, the difference due to material demand was $\Delta CT_{mb-a} = 1729.6 \pm 579.1 \text{ ppmv} \cdot \text{hr}$ (Appendix E, Table E2). This value was not quite statistically different $(p_{value} = 0.070)$ from the average baseline value (ΔCT_{b-a}). The difference in CT due to the material, after baseline subtraction, was $\Delta CT_{\mu} = 833.4 \pm 615.8$ ppmv \cdot hr, as reported in Table 4. Converted to a volume or mass flux, the material demand can be reported as $MD_k = 154.3 \pm 114.0 \text{ ppmv} \cdot \text{hr}$ $hr^{-1} \cdot m^{-2}$ and $Jk = 0.013 \pm 0.10 \text{ g} \cdot hr^{-1} \cdot m^{-2}$, respectively. The differences between these values and those reported for the corresponding baseline tests (Table 3) are not statistically significant ($p_{value} = 0.1131$ for MD_k and 0.2276 for J_k). The time required to achieve the target CT was not statistically different from that observed for the baseline tests at 1000 ppmv. Concentration profiles (Figures C37a - C39a) and CT profiles (Figures C37b - C39b) are provided in Appendix C.

The average difference between the inlet and outlet chamber CT values for the three 2000 ppmv tests at the time the target CT was achieved was $\Delta CT_{mb} = 2580.5 \pm 512.6$ ppmv · hr (Appendix E, Table E2). Corrected for the amount of fumigant remaining in the chamber at this time, the difference due to material demand was $\Delta CT_{mb-a} = 386.5 \pm 488.3$ ppmv · hr (Appendix E, Table E2). The difference between this value and that determined for the baseline tests (ΔCT_{ha} , Table 3) was not determined to be statistically significant (p_{value} = 0.3458). The difference in CT after baseline subtraction was $\Delta CT_{\mu} = -378.0 \pm 613.1$ ppmv · hr, as reported in Table 5, and was not determined to be statistically different from baseline-corrected average difference observed in the 1000 ppmv tests ($p_{value} = 0.0732$). Converted to a volume or mass flux, the material demand can be reported as $MD_k = 521.6$ \pm 239.6 ppmv \cdot hr \cdot hr $^{-1}\cdot$ m $^{-2}$ and J $_{\rm k}$ = 0.45 \pm 0.21 g \cdot hr $^{-1}\cdot$ m⁻², respectively. These values were not determined to be statistically different from the corresponding baseline tests $(p_{value} = 0.2515 \text{ for } MD_k \text{ and } p_{value} = 0.1297 \text{ for } J_k)$. The differences in these determined values $(\Delta CT_k, MD_k, \text{ and } J_k)$ between the 1000 and 2000 ppmv tests were not statistically

significant ($p_{value} = 0.0732$ for ΔCT_k , $p_{value} = 0.1047$ for MD_k and $p_{value} = 0.1028$ for J_k). As discussed for the painted steel, the negative demand is likely an artifact of the testing and not due to additional generation of ClO₂ due to the concrete in the chamber. In this case, the value is not different from zero and is well within the analytical noise of the system. The times required to achieve the target CT at 1000 and 2000 ppmv target concentrations were not statistically different from the times observed for the respective baseline tests. Concentration profiles (Figures C40a – C42a) and CT profiles (Figures C40b – C42b) for the 2000 ppmv tests are provided in Appendix C. 6.3 Total CT Demand for Baseline and Materials

The total CT for the affluent and effluent is shown in Table 6 for the baseline and each material during the 1000 and 2000 ppmv tests. The total CT for each material includes the sum of concentration \cdot time over the three phases of the test, the initial or ramp-up phase, the steady-state or decontamination (decon) phase, and the aeration phase. The file number is the date of the test, month/day/year.

| Table 6. Total Feed and Effluent CT for Each Material Te | est |
|--|-----|
|--|-----|

| | | Experiment | Total Feed CT | Total Effluent CT |
|---------------|----------------------|---------------|---------------|-------------------|
| Material | Concentration (ppmv) | (file number) | (ppmv · hr) | (ppmv · hr) |
| Baseline | 1000 | 011906 | 14216 | 13514 |
| | 1000 | 020106 | 14253 | 13572 |
| | 1000 | 020206 | 14559 | 13563 |
| | 2000 | 011806 | 16309 | 15637 |
| | 2000 | 041905 | 17063 | 15707 |
| | 2000 | 042005 | 15501 | 14638 |
| Carpet | 1000 | 040506 | 16989 | 13453 |
| | 1000 | 041006 | 17330 | 13477 |
| | 1000 | 041106 | 17151 | 13418 |
| | 2000 | 033006 | 18561 | 15693 |
| | 2000 | 040406 | 19969 | 14681 |
| | 2000 | 040606 | 19630 | 15035 |
| Concrete | 1000 | 112905 | 15803 | 13571 |
| | 1000 | 120105 | 16055 | 13581 |
| | 1000 | 120505 | 15568 | 13422 |
| | 2000 | 101105 | 16275 | 16543 |
| | 2000 | 101205 | 15221 | 16907 |
| | 2000 | 101305 | 16405 | 17397 |
| Painted Steel | 1000 | 092905 | 12879 | 13716 |
| | 1000 | 100405 | 13267 | 13801 |
| | 1000 | 100505 | 12714 | 13654 |
| | 2000 | 091905 | 15885 | 17048 |
| | 2000 | 092105 | 15912 | 17250 |
| | 2000 | 092705 | 14982 | 17690 |
| Ceiling Tile | 1000 | 030106 | 24129 | 13712 |
| | 1000 | 032806 | 25163 | 13670 |
| | 1000 | 032906 | 24223 | 13767 |
| | 2000 | 022306 | 27631 | 15428 |
| | 2000 | 022706 | 28607 | 15239 |
| | 2000 | 022806 | 27156 | 15264 |
| Wallboard | 1000 | 041806 | 19795 | 13490 |
| | 1000 | 042006 | 18772 | 13346 |
| | 1000 | 042406 | 19490 | 13514 |
| | 2000 | 041206 | 22278 | 14606 |
| | 2000 | 041306 | 22851 | 14623 |
| | 2000 | 041706 | 22034 | 14609 |
| Wood | 1000 | 020706 | 19683 | 13501 |
| | 1000 | 020906 | 18699 | 13570 |
| | 1000 | 021306 | 20411 | 13546 |
| | 2000 | 120605 | 22554 | 16476 |
| | 2000 | 120705 | 17820 | 16442 |
| | 2000 | 120805 | 17394 | 16752 |

6.4 Discussion

The average feed concentration for the baseline study was 1278.7 ± 50.6 ppmv and 2327.4 ± 246.0 ppmv (Table 3) for the 1000 ppmv and 2000 ppmv exposures, respectively. The averages include the ramp-up and steady-state phases. Similarly, the average feed concentrations required to achieve and maintain the target concentrations with the materials in the chamber are shown in Table 4 (1000 ppmv) and Table 5 (2000 ppmv). The average concentration can be used to qualitatively rank the chlorine dioxide demand of the materials, as follows: ceiling tile > wallboard \approx wood > carpet > concrete > steel \approx baseline for the 1000 ppmv exposures. The ranking for the 2000 ppmv tests was ceiling tile > wallboard > wood > carpet > concrete \approx steel \approx baseline.

Ceiling tile required a higher average feed concentration than the baseline test to reach and maintain the effluent within the target concentration limits over the 0 - 12,000 CT range. A feed concentration 68% higher than the feed in the baseline tests was required for the 1000 ppmv tests and 57% higher for the 2000 ppmv tests. The times to reach the target concentrations were observed to be significant longer for the ceiling tile (Figures C25a - C30a) than for the other materials (concentration profiles in Appendix C). The observation is especially true for the 2000 ppmv test and is also reflected in the time required to achieve 12,000 CT (Table 5). This time is reflective of the strain on the generation system due to the material demand; however, it should be mentioned that the generator had adequate capacity at the scale tested to achieve the desired target concentrations and CT values. Conversely, steel required an average concentration of only $1125.9 \pm$ 45.4 ppmv and 2342.7 ± 42.6 ppmv to obtain and maintain the target concentration in the 1000 ppmv and 2000 ppmv tests, respectively; these concentrations were not statistically different from those required for the baseline tests.

A similar qualitative ranking for chlorine dioxide demand was made using the total feed CT (Table 6) since the exposed surface area was maintained nearly the same for all material types. The average total feed CT for the 1000 ppmv baseline tests over the entire fumigation duration [ramp-up, steadystate (decon), and aeration phases] was 14342.7 ± 188.3 ppmv \cdot hr, and 16291.0 \pm 781.2 ppmv \cdot hr for the 2000 ppmv tests. The average total feed CT for ceiling tile was $24505.0 \pm$ 571.8 ppmv \cdot hr for the 1000 ppmv tests and 27798.0 \pm 739.8 ppmv · hr for the 2000 ppmv tests (to achieve the effluent CT of 12,000 ppmv · hr). These required feed CT values were 71% higher than the average feed CT of the respective (1000 ppmv or 2000 ppmv) baseline tests. The qualitative ranking based on feed CT for the 1000 ppmv and 2000 ppmv tests was ceiling tile > wallboard > wood > carpet > concrete \geq steel. This ranking is in agreement with those shown above based upon the feed concentration requirements.

These qualitative rankings agreed well with the ranking based on the material demand (MD_k) and mass flux (J_k) (Table 4) determined for the 1000 ppmv tests: ceiling tile > wood ≥ wallboard > carpet > concrete > steel. The ranking for materials in the 2000 ppmv tests was ceiling tile >

wallboard > carpet > wood > concrete \approx steel. The mass fluxes for carpet, painted wallboard, and ceiling tile at 2000 ppmv were approximately twice the fluxes at 1000 ppmv. This difference is suggestive of a nonzero order dependence on the concentration of the reaction of ClO₂ with materials; however, the limited concentration study performed here does not allow for further determination of this dependence. Additional studies are being performed by EPA at facilities in Research Triangle Park, NC. There was no difference in average material demand value or mass flux determined for the 1000 ppmv compared to the 2000 ppmv tests for wood, steel, and concrete. With respect to wood, although the difference in the feed and effluent CT values (Δ CT,) for the 1000 and 2000 ppmv tests was statistically different, normalization to fumigation time and surface area (e.g., MD,) resolved the difference.

The concrete and steel exhibited the lowest material demand of the materials investigated. The difference between the baseline and the concrete tests was not statistically different. This suggests a very minimal, if any, reaction of chlorine dioxide with the concrete coupons used in these tests. While the statistics suggested that the tests with steel were different from the baseline, no net mass flux of chlorine dioxide to the materials was observed. More tests are required to determine whether the negative fluxes are either within the experimental variability or an artifact of the detection system. In either case, the calculated flux is minimally significant.

7.0 OPERATIONAL PROBLEMS ENCOUNTERED

Corrosion due to the interaction of chlorine dioxide and moisture with electronic items contributed to numerous equipment failures during the material demand tests. Corrosion was observed on the stainless steel support bars inside the exposure chamber, inside flow meters, on all metal parts inside the INTERSCAN detectors, and inside the temperature and humidity sensors. Unfortunately, the frequency of failures was not recorded. Listed below in bullet format are the most significant problems encountered.

- Corrosion on metal parts of flow meters combined with the moisture in the air stream produced a metal oxide paste that migrated up the tube causing the float to stick.
- The pump (used for sampling the stream) inside the INTERSCAN detectors leaked. The leaks were small; however, chlorine dioxide caused failures of the potentiometers and circuit boards. Attempts were made to seal the new potentiometers with silicone, but the fix was only temporary. The circuit boards were replaced. The pumps were removed from the INTERSCAN detectors, and a single pump (Cole Parmer, Model number 075360-40) was used to sample the feed and effluent. A photograph of the interior of an INTERSCAN detector showing corroded parts due to exposure to chlorine dioxide is provided in Figure 8. The chlorine dioxide CT for the detector is not available.
- Failures due to corrosion occurred with the General Eastern Humiscan and Hobo® temperature-humidity

sensors. Because of cost, the Humiscan was replaced by the Hobo[®] sensor. Corrosion at the USB port on the Hobo[®] occurred frequently. Attempts to apply a protective coating further delayed but did not eliminate the failures. Significantly longer times occurred only when the USB cable was soldered to the Hobo[®] circuit board and protected with liquid electrical tape. A backup Hobo[®] was used during the tests because failure was unpredictable.

- Most of the sodium chlorite cartridges received from CDG Technology, Inc. leaked. The cartridges had to be pressure checked, repaired, and checked again before being installed in the generator.
- Poor temperature control in the laboratory during winter months resulted in condensation inside the exposure chamber. A plastic tent had to be erected around the exposure chamber and the area heated with a small hot air blower to maintain the proper test environment. Condensation was not observed inside the exposure chamber during any test either with or without the plastic tent configuration.
- Chlorine cylinder regulators and flow meters did not maintain set values; manual adjustments were required almost constantly.
- Fluctuations, possibly due to changing the pressure equilibrium inside the laboratory by opening the door to the corridor, were observed in the INTERSCAN detectors. The fluctuations were not always observed and were minimized by entering and exiting a side office door. The fluctuations were infrequent, momentary, and relatively small in magnitude and, therefore, not consider significant errors.

 Corrosion-related failure occurred with the circulation fan inside the exposure chamber. The chlorine dioxide CT value on the fan shown in Figure 9 was not available.

8.0 QUALITY ASSURANCES FINDINGS

Three technical audits of the chlorine dioxide exposure process were conducted over the course of the program.

The first technical audit, conducted 22–23 February 2005, was a control run using wood. This run ended up being aborted when, after more than 24 hours had passed, the chamber was unable to reach the minimum starting humidity. The wood samples absorbed so much moisture from the ambient air that it was not possible to equilibrate the humidity in the chamber. This resulted in a change to the sample storage protocol for the wood samples to prevent this problem in future testing.

A second technical audit was conducted on 17 August 2005. The chamber concentration recorded by the sensors reached the test level far more rapidly than should have been possible. Corrective actions taken by the testers failed to explain the unusual behavior of the chamber, and the test run was aborted. Further work corrected the problem.

A third technical audit was conducted on 19 September 2005. With the exception of a minor deviation in the marking scheme from the IOP— the test team used the acronym SS to designate a new lot of structural steel instead of S (as was specified in the IOP)—everything went as planned. All other operations were in accordance with the IOP and SOP.

Figure 8. Photographs of the Interior of an INTERSCAN Detector (Not exposed to Chlorine Dioxide)



Figure 9. Comparison of Chamber Fans Exposed to Chlorine Dioxide and VHP®.



Data quality audits were conducted on 20 of the 57 chlorine dioxide deposition velocity tests (35%). All were found to be acceptable, in accordance with the QAPP.

9.0 CONCLUSIONS

The chlorine dioxide material demand tests showed that the feed concentration and time required to reach the target concentration (1000 and 2000 ppmv) were a function of building material. The chlorine dioxide demand for the building materials over the 01–2,000 CT range was (from highest to lowest) ceiling tile > wood ≥ gypsum wallboard > carpet > concrete = steel for the 1000 ppmv tests, and ceiling tile > gypsum wallboard > wood > carpet > concrete = steel for the 2000 ppmv tests. Results for concrete and steel were not statistically different from the baseline in unpaired Student's t Tests at $\alpha = 0.05$.

10. LITERATURE CITED

- Larsen, L., Harper, B. G., Rome, W., Ramachadran, C., Westwood, S., *Abbreviated Test Report for the Laboratory* Validation of Chlorine Dioxide Decontamination, WDTC-TR-02-059, West Desert Test Center, Dugway Proving Ground, UT, Sep 2002, UNCLASSIFIED Report.
- McDonnell, G., Grignol, G., Antloga, K., "Vapour-Phase Hydrogen Peroxide Decontamination of Food Contact Surfaces," *Dairy, Food Environmental Sanitation*, Vol. 22, 2002, pp. 868–873.
- 3. Jahnke, M. and Lauth, G., "Biodecontamination of a Large Volume Filling Room with Hydrogen Peroxide," *Pharm. Eng.*, Vol. 17(4), Jul–Aug 1997, pp. 96–108.
- 4. Brickhouse, Mark D. "Quality Assurance Project Plan and Work Plan for Deposition Velocity Studies: Materials Sorption of Vaporized Hydrogen Peroxide or Chlorine Dioxide," DSQAPP2004DV, 2004.
- 5. "Quality Management Plan (QMP) for the National Homeland Security Research Center (NHSRC) Office of Research and Development (ORD)," U.S. Environmental Protection Agency (U.S. EPA), 2003.
- 6. "Quality Management Plan for Environmental Programs," Edgewood Chemical Biological Center Research, Development and Engineering Command, 2003.
- 7. "EPA Guidance on Environmental Data Verification and Data Validation, EPA QA/G-8," U.S. Environmental Protection Agency, 2002.
- 8. "EPA Guidance for Data Quality Assessment, Practical Methods for Data Analysis, EPA QA/G-9," U.S. Environmental Protection Agency, 2000.
- 9. "EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5," U.S. Environmental Protection Agency, 2001.
- 10. "EPA Guidance for Quality Assurance Project Plans. EPA QA/G-5," U.S. Environmental Protection Agency, 2002.
- 11. Brickhouse, M. D., Lalain, T. A., Bartram, P. W., Hall, M. R., Hess, Z. A., and Zander, Z. B., *Effects of Vaporized Decontamination Systems on Selected Interior Building Materials: Chlorine Dioxide*, Unpublished Manuscript.
- 12. Lalain, T. A., IOP DS04016, "Placement of Coupons in Glove Boxes for Material Demand Velocity/Material Compatibility Testing for the EPA Program," Mar. 2005.
- 13. Bartram, P. W., IOP DSO4017, "Procedure for the Calibration of INTERSCAN Detectors," Aug. 2005.
- 14. Hall, M. R., IOP DS04002, "Titration of Chlorine Dioxide Samples," Feb. 2005.
- CDG Technology, INC., "Chlorine Dioxide MSDS," in *Chlorine Dioxide Bench-Scale Generator Manual*, 140 Webster Street, Bethlehem, PA 18015.
- 16. Hall, M. R. and Bartram, P. W., SOP RNG-108, "Materials Exposure to Chlorine Dioxide," July 05.
- 17. Bartram, P. W., IOP DS04014, "Procedure for the Operation of the Chlorine Dioxide Glove Box and Exposure of Material," Oct. 05.
- 18. Skoog, D. A. and Leary, J. J., Principles of Instrumental Analysis, Harcourt Brace College Publishers, 1992.

Appendix A

Detailed Coupon Preparation and Inspection Procedures

Appendix A: Detailed Coupon Preparation and Inspection Procedures (From the Quality Assurance Project Plan, Document Number: DSQAPP04DV, Version 3, 10 December 2004)

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)

- Stock Item Description: 2 x 4 x 8 KD WW/SPF Stud
- Supplier/Source: Home Depot, Edgewood, Maryland
- Coupon Dimensions: $10 \text{ in. } x \ 1 \ 1/2 \text{ in. } x \ 1/2 \text{ in. }$
- Preparation of Coupon:
- The machined ends of the stock will be discarded by removing > 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: 1/2 in. 4 ft. x 8 ft. Drywall
- Supplier/Source: Home Depot, Edgewood, Maryland
- Coupon Dimensions: 6 in. x 6 in. x 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, 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 Block

- Stock Item Description: 8 in. x 16 in. x 1 1/2 in. concrete block cap
- Supplier/Source: York Supply, Aberdeen, Maryland
- Coupon Dimensions: 4 in. x 8 in. x 1 1/2 in.
- Preparation of Coupon:
- Coupons will be cut from stock using a water-jet.
- Four coupons will be cut from each stock piece.

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 machined edge.
- The machined edge will be discarded by removing > 1/2 in.

Painted Structural Steel

- Stock Item Description: A572 Grade 50, 4 ft. x 8 ft. x 1/4 in.
- Supplier/Source: Specialized Metals
- Coupon Dimensions: 1/4 in. x 12 inches total, dog bone shaped with 2 in. wide at ends, 3/4 in. wide at center
- Preparation of Coupon:
- Coupons will be cut from stock using a water-jet.
- A visual observation will be conducted of each coupon to determine whether size and shape have deviated from dimension and been 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 corrison 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.

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 1/2 in. $\pm 1/32$ in. |
|--|---|
| Latex-Painted Gypsum Wallboard | 6 in. $\pm 1/16$ in. x 6 in. $\pm 1/16$ in. x $1/2$ in. $\pm 1/16$ in. |
| Concrete Block | 4 in. $\pm 1/2$ in. x 8 in. $\pm \frac{1}{2}$ in. x 1.5 in. $\pm 3/16$ 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 |
| 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

| Characte | er | Explanation | | |
|----------|--------------------|--|--|--|
| 1 | Material | | | |
| | W | = wood | | |
| | G | = gypsum | | |
| | S | = A572 steel | | |
| | Т | = acoustic ceiling tile | | |
| | С | = concrete cinder block | | |
| | R | = carpet | | |
| | В | = circuit breakers | | |
| 2 | Fumigar | ıt: | | |
| | \mathbf{V} | = ClO ₂ | | |
| | Ν | = no fumigant | | |
| | Test star | t date | | |
| 3 | year | for example: $4 = 2004$ | | |
| 4,5 | mon | th for example: $06 = $ June | | |
| 6,7 | day | for example: $10 =$ the 10th of a month | | |
| 8,9 | Glove b | ox position (see IOP DS04016 Figure 1) | | |
| Example | GV4101 | 104 | | |
| | Gypsum sample r | wallboard with chlorine dioxide having a test start date of October 11th, 2004, and is number 4. | | |
Figure B1. Coupon Placement in Chamber



Appendix C

Typical Time/Concentration and Time/CT Profiles for Chlorine Dioxide Tests

Figure C1. Baseline Profiles at 1000 ppmv Chlorine Dioxide (19 Jan 06).



Vapor Concentration Throughout Run

b. CT versus Time Profile



CT Throughout Run

Figure C2. Baseline Profiles at 1000 ppmv Chlorine Dioxide (01 Feb 06).





b. CT versus Time Profile



CT Throughout Run

Figure C3. Baseline Profiles at 1000 ppmv Chlorine Dioxide (02 Feb 06).





b. CT versus Time Profile



Figure C4. Baseline Profiles at 2000 ppmv Chlorine Dioxide (18 Jan 06).



Vapor Concentration Throughout Run

b. CT versus Time Profile



CT Throughout Run

Figure C5. Baseline Profiles at 2000 ppmv Chlorine Dioxide (19 Apr 06).









b. CT versus Time Profile

Figure C6. Baseline Profiles at 2000 ppmv Chlorine Dioxide (20 Apr 06).

a. Concentration versus Time Profile



Vapor Concentration Throughout Run

b. CT versus Time Profile

CT Throughout Run



Figure C7. Profiles for Chlorine Dioxide at 1000 ppmv on Carpet (05 Apr 06).

a. Concentration versus Time Profile



Vapor Concentration Throughout Run

b. CT versus Time Profile



CT Throughout Run

Figure C8. Profiles for Chlorine Dioxide at 1000 ppmv on Carpet (10 Apr 06).





b. CT versus Time Profile



Figure C9. Profiles for Chlorine Dioxide at 1000 ppmv on Carpet (11 Apr 06).



Vapor Concentration Throughout Run

b. CT versus Time Profile



Figure C10. Profiles for Chlorine Dioxide at 2000 ppmv on Carpet (30 Mar 06).

a. Concentration versus Time Profile



b. CT versus Time Profile



Figure C11. Profiles for Chlorine Dioxide at 2000 ppmv on Carpet (04 Apr 06).

a. Concentration versus Time Profile



b. CT versus Time Profile



Figure C12. Profiles for Chlorine Dioxide at 2000 ppmv on Carpet (06 Apr 06).

a. Concentration versus Time Profile



b. CT versus Time Profile



Figure C13. Profiles for Chlorine Dioxide at 1000 ppmv on Painted Steel (29 Sep 05).



Vapor Concentration Throughout Run

b. CT versus Time Profile



CT Throughout Run

Figure C14. Profiles for Chlorine Dioxide at 1000 ppmv on Painted Steel (04 Oct 05).



Vapor Concentration Throughout Run

b. CT versus Time Profile



CT Throughout Run

Figure C15. Profiles for Chlorine Dioxide at 1000 ppmv on Painted Steel (05 Oct 05).

a. Concentration versus Time Profile



b. CT versus Time Profile



CT Throughout Run

Figure C16. Profiles for Chlorine Dioxide at 2000 ppmv on Painted Steel (19 Sep 05).



Vapor Concentration Throughout Run

b. CT versus Time Profile

CT Throughout Run



Figure C17. Profiles for Chlorine Dioxide at 2000 ppmv on Painted Steel (21 Sep 05).





b. CT versus Time Profile



CT Throughout Run

Figure C18. Profiles for Chlorine Dioxide at 2000 ppmv on Painted Steel (27 Sep 05).

a. Concentration versus Time Profile



b. CT versus Time Profile

CT Throughout Run --- CT, Enclosure CT, Feed CIO₂ CT (ppmv*h) Time (hour)

Figure C19. Profiles for Chlorine Dioxide at 1000 ppmv on Wallboard (18 Apr 06).

a. Concentration versus Time Profile



b. CT versus Time Profile



CT Throughout Run

Figure C20. Profiles for Chlorine Dioxide at 1000 ppmv on Wallboard (20 Apr 06).

a. Concentration versus Time Profile



b. CT versus Time Profile



Figure C21. Profiles for Chlorine Dioxide at 1000 ppmv on Wallboard (24 Apr 06).



Vapor Concentration Throughout Run

b. CT versus Time Profile



C-22

Figure C22. Profiles for Chlorine Dioxide at 2000 ppmv on Wallboard (12 Apr 06).

a. Concentration versus Time Profile



b. CT versus Time Profile

CT Throughout Run



Figure C23. Profiles for Chlorine Dioxide at 2000 ppmv on Wallboard (13 Apr 06).



Vapor Concentration Throughout Run

b. CT versus Time Profile



CT Throughout Run

Figure C24. Profiles for Chlorine Dioxide at 2000 ppmv on Wallboard (17 Apr 06).

a. Concentration versus Time Profile



b. CT versus Time Profile



Figure C25. Profiles for Chlorine Dioxide at 1000 ppmv on Ceiling Tile (01 Mar 06).

a. Concentration versus Time Profile





b. CT versus Time Profile



CT Throughout Run

Figure C26. Profiles for Chlorine Dioxide at 1000 ppmv on Ceiling Tile (28 Mar 06).

a. Concentration versus Time Profile



b. CT versus Time Profile



CT Throughout Run

Figure C27. Profiles for Chlorine Dioxide at 1000 ppmv on Ceiling Tile (29 Mar 06).





b. CT versus Time Profile



CT Throughout Run

Figure C28. Profiles for Chlorine Dioxide at 2000 ppmv on Ceiling Tile (23 Feb 06).

a. Concentration versus Time Profile



b. CT versus Time Profile



CT Throughout Run

Figure C29. Profiles for Chlorine Dioxide at 2000 ppmv on Ceiling Tile (27 Feb 06).





b. CT versus Time Profile



CT Throughout Run

Figure C30. Profiles for Chlorine Dioxide at 2000 ppmv on Ceiling Tile (28 Feb 06).



Vapor Concentration Throughout Run

b. CT versus Time Profile

CT Throughout Run



Figure C31. Profiles for Chlorine Dioxide at 1000 ppmv on Wood (07 Feb 06).





b. CT versus Time Profile



Figure C32. Profiles for Chlorine Dioxide at 1000 ppmv on Wood (09 Feb 06).





b. CT versus Time Profile



Figure C33. Profiles for Chlorine Dioxide at 1000 ppmv on Wood (13 Feb 06).





b. CT versus Time Profile



CT Throughout Run
Figure C34. Profiles for Chlorine Dioxide at 2000 ppmv on Wood (06 Dec 05).





b. CT versus Time Profile



CT Throughout Run

Figure C35. Profiles for Chlorine Dioxide at 2000 ppmv on Wood (07 Dec 05).



Vapor Concentration Throughout Run

b. CT versus Time Profile



C-36

Figure C36. Profiles for Chlorine Dioxide at 2000 ppmv on Wood (08 Dec 05).



Vapor Concentration Throughout Run

b. CT versus Time Profile

CT Throughout Run CT, Enclosure CT, Feed CIO₂ CT (ppmv*h) Time (hour)

Figure C37. Profiles for Chlorine Dioxide at 1000 ppmv on Concrete (29 Nov 05).

a. Concentration versus Time Profile



Vapor Concentration Throughout Run

b. CT versus Time Profile



Figure C38. Profiles for Chlorine Dioxide at 1000 ppmv on Concrete (01 Dec 05).

a. Concentration versus Time Profile



Vapor Concentration Throughout Run

b. CT versus Time Profile



Figure C39. Profiles for Chlorine Dioxide at 1000 ppmv on Concrete (05 Dec 05).





b. CT versus Time Profile



Figure C40. Profiles for Chlorine Dioxide at 2000 ppmv on Concrete (11 Oct 05).



Vapor Concentration Throughout Run

b. CT versus Time Profile



Figure C41. Profiles for Chlorine Dioxide at 2000 ppmv on Concrete (12 Oct 05).

a. Concentration versus Time Profile



Vapor Concentration Throughout Run

b. CT versus Time Profile



Figure C42. Profiles for Chlorine Dioxide at 2000 ppmv on Concrete (13 Oct 05).

a. Concentration versus Time Profile



Vapor Concentration Throughout Run

b. CT versus Time Profile



CT Throughout Run

Appendix D

Excel Data Worksheet for Comparison of INTERSCAN Detectors and Titration Checks

| Table D1. Example of INTERSCAN Detectors and | d Titration Checks | (Baseline 19 Apr 200 | 5) |
|--|--------------------|----------------------|----|
|--|--------------------|----------------------|----|

| | | INLET | | |
|--|----------------------------|---|--|---|
| Titration (time) | Replicate | Interscan (ppmv) | titration (ppmv) | Criteria |
| 1 hr | 1 | 2257 | 2191 | |
| | 2 | 2241 | 2050 | |
| | 3 | 2232 | 1879 | |
| Mean | | 2243.3 | 2040.0 | |
| SD | | 12.7 | 156.2 | |
| Titration / Interscan Agreement | | 10.0% | , ວ | +/- 15% |
| CT/2 | 1 | 2225 | 2052 | |
| | 2 | 2218 | 1990 | |
| | 3 | 2203 | 2033 | |
| Mean | | 2215.3 | 2025.0 | |
| SD | | 11.2 | 31.8 | |
| Titration / Interscan Agreement | | 9.4% | | +/- 15% |
| Final | 1 | 2252 | 2037 | |
| | 2 | 2267 | 2092 | |
| | 3 | 2276 | 2048 | |
| Mean | | 2265 | 2059 | |
| SD | | 12.1 | 29.1 | |
| Titration / Interscan Agreement | | 10.0% | , D | +/- 15% |
| | | OUTLET | | |
| Titration (time) | Replicate | Interscan (ppmv) | titration (ppmv) | Criteria |
| 1 hr | 1 | 2000 | 1970 | |
| | 2 | 1990 | 1786 | |
| | 3 | 1988 | 1818 | |
| Mean | | 1992.7 | 1858 | 2000 - 2500 ppm |
| SD | | 6.4 | 98.3 | |
| Titration / Interscan Agreement | | 7.2% | | +/- 15% |
| CT/2 | 1 | 0070 | 1040 | |
| | 1 | 2073 | 1948 | |
| | 2 | 2073 | 1948 | |
| | 2 | 2073 2078 2064 | 1948 1933 1896 | |
| Mean | 2 | 2073 2078 2064 2071.7 | 1948 1933 1896 1925.7 | 2000 – 2500 ppm |
| Mean SD | 2 3 | 2073 2078 2064 2071.7 7.1 | 1948 1933 1896 1925.7 26.8 | 2000 – 2500 ppm |
| Mean SD Titration / Interscan Agreement | 2 3 | 2073 2078 2064 2071.7 7.1 7.6% | 1948 1933 1896 1925.7 26.8 | 2000 – 2500 ppm +/- 15% |
| Mean SD Titration / Interscan Agreement Final | 1 2 3 1 | 2073 2078 2064 2071.7 7.1 7.6% 2064 | 1948 1933 1896 1925.7 26.8 2024 | 2000 – 2500 ppm +/- 15% |
| Mean SD Titration / Interscan Agreement Final | 1 2 3 1 2 | 2073 2078 2064 2071.7 7.1 7.6% 2064 2085 | 1948 1933 1896 1925.7 26.8 2024 1948 | 2000 – 2500 ppm +/- 15% |
| Mean SD Titration / Interscan Agreement Final | 1 2 3 1 2 3 | 2073 2078 2064 2071.7 7.1 7.6% 2064 2085 2101 | 1948 1933 1896 1925.7 26.8 2024 1948 1897 | 2000 – 2500 ppm +/– 15% |
| Mean SD Titration / Interscan Agreement Final Mean | 1 2 3 1 2 3 | 2073 2078 2078 2064 2071.7 7.1 7.6% 2064 2085 2101 2083.3 | 1948 1933 1896 1925.7 26.8 2024 1948 1897 1956.3 | 2000 – 2500 ppm +/– 15% 2000 – 2500 ppm |
| Mean SD Titration / Interscan Agreement Final Mean SD | 1 2 3 1 2 3 | 2073 2078 2064 2071.7 7.1 7.6% 2064 2085 2101 2083.3 18.6 | 1948 1933 1896 1925.7 26.8 2024 1948 1897 1956.3 63.9 | 2000 – 2500 ppm +/- 15% 2000 – 2500 ppm |

Appendix E Results of Chlorine Dioxide Material Demand Tests

Table E1. Results of Chlorine Dioxide Baseline Demand Tests

| Replicate Number | Feed CT (ppmv hr) | Effluent CT (ppmv hr) | Time (hr) | ∆CT _ь (ppmv hr) | ΔCT _a (ppmv hr) | ∆CT _{ь-a} (ppmv hr) | | | |
|----------------------|----------------------|--------------------------|--------------|-------------------------------|-------------------------------|---------------------------------|--|--|--|
| Baseline – 1000 ppmv | | | | | | | | | |
| 1 | 14000.1 | 12006.4 | 11.5 | 1993.7 | 1065.1 | 928.6 | | | |
| 2 | 13844.1 | 12010.8 | 12010.8 10.7 | | 1160.7 | 672.6 | | | |
| 3 | 14219.3 | 12012.0 | 10.8 | 2207.2 | 1119.8 | 1087.4 | | | |
| Average Std. | 14021.2 | 12009.7 | 11.0 | 2011.4 | 1115.2 | 896.2 | | | |
| Dev. | 188.5 | 3.0 | 0.4 | 187.6 | 48.0 | 209.3 | | | |
| Baseline – 2000 ppmv | | | | | | | | | |
| 1 | 14949.0 | 12005.1 | 6.3 | 2943.9 | 2086.5 | 857.4 | | | |
| 2 | 14353.4 | 12005.0 | 6.5 | 2348.4 | 1992.4 | 356.0 | | | |
| 3 | 15403.8 | 12030.2 | 5.9 | 3373.6 | 2293.6 | 1080.0 | | | |
| Average Std. Dev. | 14902.0 | 12013.4 | 6.2 | 2888.6 | 2124.2 | 764.5 | | | |
| | 526.8 | 14.5 | 0.3 | 514.8 | 154.1 | 370.8 | | | |

Table E2. Results of Chlorine Dioxide Material Demand Tests

| Replicate Number | Feed CT (ppmv hr) | Effluent CT (ppmv hr) | Time (hr) | ∆CT _{mb} (ppmv hr) | ∆CT _a (ppmv hr) | ∆CT _{mb-a} (ppmv hr) | ∆CT _k (ppmv hr) | |
|---------------------------|----------------------|--------------------------|-------------------|--------------------------------|-------------------------------|----------------------------------|-------------------------------|--|
| <u>Carpet – 1000 ppmv</u> | | | | | | | | |
| 1 | 15817.5 | 12009.4 | 10.83 | 3808.1 | 1147.7 | 2660.4 | | |
| 2 | 16195.8 | 12009.9 | 10.70 | 4185.9 | 1162.7 | 3023.2 | | |
| 3 | 16054.4 | 12000.5 | 10.77 | 4053.8 | 1166.6 | 2887.2 | | |
| Average Std. | 16022.6 | 12006.6 | 10.77 | 4015.9 | 1159.0 | 2856.9 | 1960.7 | |
| Dev. | 191.1 | 5.3 | 0.07 | 191.7 | 10.0 | 183.3 | 278.2 | |
| | | · | Carpet – 20 | 000 ppmv | • | | • | |
| 1 | 16950.4 | 12026.1 | 6.07 | 4924.2 | 2268.7 | 2655.5 | | |
| 2 | 18278.6 | 12022.4 | 5.92 | 6256.2 | 2220.9 | 4035.3 | | |
| 3 | 17720.1 | 12022.8 | 5.83 | 5697.4 | 2267.7 | 3429.7 | | |
| Average Std. | 17649.7 | 12023.8 | 5.94 | 5625.9 | 2252.4 | 3373.5 | 2609.0 | |
| Dev. | 666.9 | 2.1 | 0.12 | 668.8 | 27.3 | 691.6 | 784.7 | |
| | | | <u>Steel – 10</u> | 00 ppmv | | | | |
| 1 | 12314.8 | 12010.1 | 10.98 | 304.6 | 1165.6 | -861.0 | | |
| 2 | 12426.1 | 12001.8 | 10.60 | 424.4 | 1192.5 | -768.1 | | |
| 3 | 11881.2 | 12013.2 | 10.98 | -131.9 | 1046.2 | -1178.1 | | |
| Average Std. | 12207.4 | 12008.4 | 10.85 | 199.0 | 1134.8 | -935.7 | -1832.0 | |
| Dev. | 287.9 | 5.9 | 0.22 | 292.8 | 77.9 | 215.0 | 300.0 | |
| | | | <u>Steel – 20</u> | 00 ppmv | | | | |
| 1 | 14301.5 | 12017.2 | 6.10 | 2284.3 | 2220.9 | 63.4 | | |
| 2 | 14157.9 | 12004.0 | 5.95 | 2153.9 | 2233.8 | -79.9 | | |
| 3 | 13463.8 | 12009.9 | 5.86 | 1453.8 | 2312.5 | -858.7 | | |
| Average Std. | 13974.4 | 12010.4 | 5.97 | 1964.0 | 2255.7 | -291.7 | -1056.2 | |
| Dev. | 448.0 | 6.6 | 0.12 | 446.6 | 49.6 | 496.2q | 619.4 | |
| Wallboard – 1000 ppmv | | | | | | | | |
| 1 | 18132.2 | 12018.6 | 10.68 | 6113.6 | 1151.7 | 4961.9 | | |
| 2 | 17483.6 | 12001.3 | 10.62 | 5482.4 | 1164.7 | 4317.7 | | |
| 3 | 17987.5 | 12014.7 | 10.57 | 5972.8 | 1158.7 | 4814.1 | | |
| Average Std. | 17867.8 | 12011.5 | 10.62 | 5856.3 | 1158.4 | 4697.9 | 3801.7 | |
| Dev. | 340.5 | 9.1 | 0.06 | 331.4 | 6.5 | 337.5 | 397.1 | |

Table E2 (Continued). Results of Chlorine Dioxide Material Demand Tests

| Replicate | Feed CT | Effluent CT | Time (hr) | ∆CT _{mb} | ∆CT _a | ∆CT _{mb-a} | ∆CT _k | | |
|--------------|----------------------------|-------------|-----------------------|--------------------|------------------|---------------------|------------------|--|--|
| Number | (ppmv nr) | (ppmv nr) |) M/s IIIs s sud | (ppmv nr) | (ppmv nr) | (ppmv nr) | (ppmv nr) | | |
| | | | | | | | | | |
| 1 | 20189.2 | 12004.7 | 6.10 | 8184.5 | 2260.7 | 5923.8 | | | |
| 2 | 20538.5 | 12010.1 | 5.92 | 8528.3 | 2257.7 | 6270.6 | | | |
| 3 | 19711.5 | 12017.2 | 60.3 | 7694.4 | 2182.1 | 5512.3 | F107.0 | | |
| Average Std. | 20146.4 | 12010.7 | 6.02 | 8135.7 | 2233.5 | 5902.2 | 5137.8 | | |
| Dev. | 415.1 | 6.3 | 0.09 | 419.1 | 44.5 | 3/9./ | 530.7 | | |
| 1 | 000704 | 100100 | <u>Ceiling lile –</u> | <u>1000 ppmv</u> | 1000.0 | 00017 | | | |
| 1 | 22978.4 | 12016.6 | 10.88 | 10961.7 | 1080.0 | 9881.7 | | | |
| 2 | 23910.5 | 12018.4 | 10.90 | 11892.1 | 1187.5 | 10704.6 | | | |
| 3 | 22912.8 | 12003.5 | 10.75 | 10909.4 | 1166.6 | 9742.8 | 00105 | | |
| Average Std. | 23267.2 | 12012.8 | 10.84 | 11254.4 | 1144./ | 10109.7 | 9213.5 | | |
| Dev. | 558.0 | 8.1 | 0.08 | 552.9 | 57.0 | 519.9 | 560.4 | | |
| 1 | | 10011 5 | <u>Ceiling Tile –</u> | 2000 ppmv | | 11156.6 | | | |
| | 25475.6 | 12011.5 | 7.13 | 13464.1 | 2307.5 | 11156.6 | | | |
| 2 | 26600.2 | 12036.2 | 7.23 | 14563.9 | 2369.2 | 12194.7 | | | |
| 3 | 25143.4 | 12002.2 | 6.73 | 13141.3 | 2362.2 | 10779.1 | | | |
| Average Std. | 25739.7 | 12016.6 | 7.03 | 13723.1 | 2346.3 | 11376.8 | 10612.3 | | |
| Dev. | 763.5 | 17.6 | 0.26 | 745.9 | 33.8 | 733.1 | 821.5 | | |
| | | | <u>Wood – 10</u> | 000 ppmv | | | | | |
| 1 | 18910.9 | 12017.4 | 10.80 | 6893.5 | 1117.9 | 5775.6 | | | |
| 2 | 18090.1 | 12014.1 | 10.53 | 6076.0 | 1191.5 | 4884.5 | | | |
| 3 | 19490.9 | 12006.7 | 10.62 | 7484.1 | 1130.8 | 6353.3 | | | |
| Average Std. | 18830.0 | 12012.7 | 10.65 | 6817.9 | 1146.7 | 5671.1 | 4774.9 | | |
| Dev. | 703.8 | 5.5 | 0.14 | 707.1 | 39.3 | 740.0 | 769.0 | | |
| | | | <u>Wood – 20</u> | 000 ppmv | | | | | |
| 1 | 17298.5 | 12035.6 | 6.18 | 5262.9 | 2300.5 | 2962.4 | | | |
| 2 | 16678.7 | 12015.5 | 6.23 | 4663.2 | 2297.5 | 2365.7 | | | |
| 3 | 16019.9 | 12031.0 | 6.30 | 3988.9 | 2275.6 | 1713.3 | | | |
| Average Std. | 16665.7 | 12027.3 | 6.24 | 4638.3 | 2291.2 | 2347.1 | 1582.7 | | |
| Dev. | 639.4 | 10.5 | 0.06 | 637.4 | 13.6 | 624.8 | 726.6 | | |
| | | | Concrete Block | <u>– 1000 ppmv</u> | | | | | |
| 1 | 14910.0 | 12009.6 | 10.75 | 2900.4 | 1173.6 | 1726.8 | | | |
| 2 | 15504.3 | 12015.5 | 11.07 | 3488.8 | 1178.6 | 2310.2 | | | |
| 3 | 14338.2 | 12012.7 | 10.90 | 2325.5 | 1173.6 | 1151.9 | | | |
| Average Std. | 14917.5 | 12012.6 | 10.91 | 2904.9 | 1175.3 | 1729.6 | 833.4 | | |
| Dev. | 583.1 | 2.9 | 0.16 | 581.7 | 2.9 | 579.1 | 615.8 | | |
| | Concrete Block – 2000 ppmv | | | | | | | | |
| 1 | 15069.3 | 12002.0 | 6.35 | 3067.3 | 2161.2 | 906.1 | | | |
| 2 | 14045.1 | 11999.5 | 6.28 | 2045.6 | 2108.4 | -62.8 | | | |
| 3 | 14636.9 | 12008.2 | 6.03 | 2628.6 | 2312.5 | 316.1 | | | |
| Average Std. | 14583.8 | 12003.2 | 6.22 | 2580.5 | 2194.0 | 386.5 | -378.0 | | |
| Dev. | 514.2 | 4.5 | 0.17 | 512.6 | 105.9 | 488.3 | 613.1 | | |



Office of Research and Development National Homeland Security Research Center Cincinnati, OH 45268 Official Business Penalty for Private Use \$300



Recycled/Recyclable Printed with vegetable-based ink on paper that contains a minimum of 50% post-consumer fiber content processed chlorine free PRESORTED STANDARD POSTAGE & FEES PAID EPA PERMIT NO. G-35