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Effects of Vaporized Decontamination Systems on Selected Building Interior Materials: Chlorine Dioxide

Office of Research and Development
National Homeland Security Research Center

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Disclaimer

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Preface

To address Homeland Security needs for decontamination, the U.S. Environmental Protection Agency (EPA) established an Interagency Agreement with the U.S. Army Edgewood Chemical and Biological Center (ECBC) to take advantage of ECBC's extensive expertise and specialized research facilities for the decontamination of surfaces contaminated with chemical and biological (CB) warfare agents. The National Homeland Security Research Center (NHSRC) formed a collaboration with ECBC to more completely address the impact of decontaminants on indoor surfaces in buildings. The work was completed under EPA IAG DW 939917-01-0. The work discussed in this report was conducted from November 2003 to October 2006.

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

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The authors thank Dr. David Cullinan (SAIC/Geo-Centers, Inc.) for preparing many coupon run baskets, coupon measurements, and chain-of-custody forms during the time his assigned laboratory was closed.

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List of Acronyms

APG	Aberdeen Proving Grounds
ASTM	American Society for Testing and Materials
CB	chemical and biological
CD	chlorine dioxide
CoC	chain-of-custody
CT	concentration time
CW	chemical warfare
doc	documentation
DS	Decontamination Sciences
ECBC	Edgewood Chemical and Biological Center
EPA	U.S. Environmental Protection Agency
GSA	General Services Administration
H_2O_2	hydrogen peroxide
hr or hrs	hour or hours
IAW	in accordance with
ID	Gant chart representation for task number (on Gant Chart only)
IOP	Internal Operating Procedure
ISO 17025	International Standardization Organization Standard 17025 on Laboratory Quality Procedures
MSDS	Material Safety Data Sheets
N	Newton
NHSRC	National Homeland Security Research Center
QA	quality assurance
QAPP	Quality Assurance Project Plan (QAPP)
QMP	Quality Management Plan
R&D	Research and Development
RDECOM	Research, Development, and Engineering Command (formerly SBCCOM)
RH	relative humidity
SOPs	standing operating procedures ("standard" may also be used in place of "standing" with the same meaning)
TICs	toxic industrial chemicals
TIMs	toxic industrial materials
UL	Underwriters Laboratories
VHP®, VHP	reference to Steris' registered "vaporized hydrogen peroxide" procedure

COUPON SPECIFIC CODING

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

1 Background

The material compatibility studies were designed to determine how decontaminant vapors impact building materials within an enclosed building interior space. Since building interiors contain large surfaces composed of complex material and electrical components such as circuit breakers, data are needed to determine how such materials are affected by exposure to the vapor. Vaporized hydrogen peroxide (VHP®) and chlorine dioxide (CD) 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. The representative building interior materials tested were unpainted concrete cinder block, standard stud lumber (2" x 4" fir), latex-painted ½-inch gypsum wallboard, ceiling suspension tile, painted structural steel, and carpet. The physical properties of the building materials were measured using ASTM test methods. The material compatibility studies also investigated electrical breakers, using Underwriters Laboratories (UL) test methods and the breaker components aluminum, copper, and steel. The samples were studied using specialized chemical testing to determine the type of chlorine containing salts on the metal surface. In addition, visual appearance was documented. This report contains the results for the CD-exposed coupon material compatibility tests. The VHP results are documented in a separate report.

2
Summary of Conclusions

CD-exposed building materials showed some minor changes in integrity but no change in appearance compared to nonexposed samples. The samples were evaluated for outliers using the Dixon's Q-Test in accordance with (IAW) ASTM Method E 178 and for statistically demonstrated differences using the Welch's T-Test.

- **Visual Inspection:** No differences were observed for any of the six main material type coupons after CD exposure and aging compared to before CD exposure.
- **Painted Structural Steel:** The CD-fumigated structural steel coupons showed no change in the maximum load required to break the samples compared to the controls. Minor differences in tensile strength were reported; however, these differences were due to differences in the cross-section area of the coupons that were within the tolerance limits set for quality control. All samples were above the specified tensile strength requirements of the ASTM test (by 20% or more). There is no obvious change in the potential for failure of the steel after fumigation using CD.
- **Gypsum Wallboard:** No statistically significant difference in the resistance to penetration by a nail was observed between the control and CD-fumigated coupons. The differences noted were well within both the 15% variation indicated in the ASTM specification and the standard deviations of the test.
- **Ceiling Tile:** Exposure to CD has no statistically significant effect on the force required to break the ceiling tile coupons compared to the controls.
- **Carpet:** There appears to be a minor increase for the average tuft bind results with exposure to CD, but the difference is smaller than the standard deviations of the individual test results.
- **Concrete Cinder Block:** The fumigated concrete cinder blocks did not exhibit any changes from the control samples. There is no evidence to indicate that fumigation with CD has any effect on the cinder blocks.
- **Wood:** Exposure to high concentrations of CD for short durations appears to reduce the tensile strength of the furring strips prepared from the standard stud lumber, causing them to fail more rapidly (2% decrease at half-target, 15% decrease at target concentration) and at lower applied forces (+1.4% at half-target, −17% at target concentration).
- **Circuit Breakers:** Exposure to CD presented a conflicting picture of the effects on circuit breakers. Under the 60-amp challenge (300% of the rated value), exposed circuit breakers showed no statistically significant difference compared to the controls with respect to the time to trip. Under the 30-amp challenge, the circuit breakers tripped more slowly than the controls. Failure criteria must be established to determine whether this is an acceptable response.
- **Residual Analysis on Metals:** Chlorine dioxide decomposed on aluminum to yield chloride, chlorite, chlorate, and perchlorate anions. The products from the reaction of chlorine dioxide with copper and steel surfaces were chloride, chlorate, and perchlorate. The reaction of chlorine dioxide on steel was the most severe, with chloride the most abundant product. The metal chloride, a decomposition product of the other anions, was the most abundant species on each metal. The number of chloride anions on each metal was similar in the 6-hr (1000 ppm) and 12-hr (2000 ppm) tests.

3
Introduction

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

Since building interiors may contain large surfaces composed of complex materials, the material compatibility studies were designed to determine how decontaminant vapors impact building materials within an enclosed building interior space. The objective of this study was to establish and conduct laboratory test procedures to determine to what degree interior building materials were affected by decontamination using VHP® and CD. The building materials used for testing were 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 encompassed a variety of material compositions and porosities; the materials studied included unpainted concrete cinder block, standard stud lumber (2" x 4" fir, type-II), latex-painted ½-inch gypsum wallboard, acoustical ceiling suspension tile, primer-painted structural steel, and carpet. The material compatibility studies also investigated material(s) related to electrical breaker connections. The physical appearance was documented by visual inspection of the test material, and the physical properties of the building materials were measured using standardized ASTM and Underwriters Laboratories (UL) test methods. Specialized chemical testing was conducted to determine whether chemical changes occurred in select building materials.

4
Experimental Method

The material compatibility testing was conducted in compliance with the Quality Assurance Project and Work Plan[1] developed under the Quality Management Plans[2,3] and EPA E4 quality system requirements.[4-7]

4.1 Coupon Preparation

Test coupons were prepared in accordance with the ASTM testing requirements for material compatibility testing. The coupons were cut from stock material in accordance with (IAW) the procedure in Appendix B of the QAPP[8], which has been reproduced as Appendix B of this report. Coupons were prepared by obtaining a large enough quantity of material that multiple test samples could be obtained with uniform characteristics (e.g., test coupons were all cut from the interior rather than the edge of a large piece of material). The building materials studied, including supplier and coupon dimensions, are provided in Table 4.1 and shown in Figure 4.1., see page 5.

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

The material compatibility studies also investigated materials related to electrical breaker connections such as intact circuit breakers and component metals aluminum, copper, and steel. The circuit breakers were one-pole circuit breakers (HOM120, 2400 watts, 120/240 volts, 20 amperes).

4.2 Coupon Exposure: Wood, Wallboard, Ceiling Tile, Steel, Carpet, and Concrete Cinder Block

The process for exposing the building material samples to CD and results for the material demand study are documented in a separate report titled “Material Demand Studies: Materials Sorption of Chlorine Dioxide,” by Phil Bartram et. al. This testing followed the operating procedures discussed with the sponsor and is not specific to any particular vendor. A brief overview of the exposure process is provided in this section. The material demand report contains the detailed test information and results.

The coupons were placed in the exposure chamber, which had been conditioned to achieve the target relative humidity (RH) of 75% and target temperature of 75 °F. The vapor generator was operated to maintain the chamber concentration within specified ranges. The target concentration was 2000-ppm CD for six hours for a total concentration-time (CT) value of 12,000 ppm-hrs. The half-target concentration was 1000-ppm CD for twelve hours also for a total concentration-time (CT) value of 12,000 ppm-hrs. The CD tests were conducted with a turnover rate of approximately one air exchange per hour due to the relative stability of CD and to mimic actual treatment conditions. Aeration of the chamber was conducted following the decontamination phase (exposure period) and continued until the vapor concentration fell to/below the levels required by the Risk Reduction Office to ensure safe operation for personnel. The coupons remained in the chamber until aeration was complete. Control samples were prepared using the same procedure as the test runs except with only air (no fumigant) through the chamber. Three replicate runs were done for each sample at each condition. The samples were removed from the chamber, marked with unique sample identifier codes, and visually examined.

4.3 Coupon Exposure: Circuit Breakers

Like the other building materials discussed in Section 4.2, the circuit breakers (Hom220, Home Depot) were placed in the exposure chamber and exposed to fumigant. After exposure to the decontaminant, the circuit breakers were stored in a fume hood for two days and then placed in storage under load for three months. Each set of circuit breakers was inserted into an electrical box (8 spaces, 16 circuits, 100 amp max from square D, Home Depot # 577-340). The circuit breaker box was wired with 12-gauge, 20-amp wire into the 120-V outlet. Each circuit breaker was wired in series with an electrical lamp (s513e) with an outlet box (s110e) manufactured by Thomas & Bretts (Home Depot # c214477 and b214426, respectively). The load in each lamp was a Phillips 40-watt light bulb (Philips and Sylvania, Home Depot). Current was applied to the circuits and monitored. At the end of 90 days, the circuit breakers were tested to determine the effect of CD fumigation.

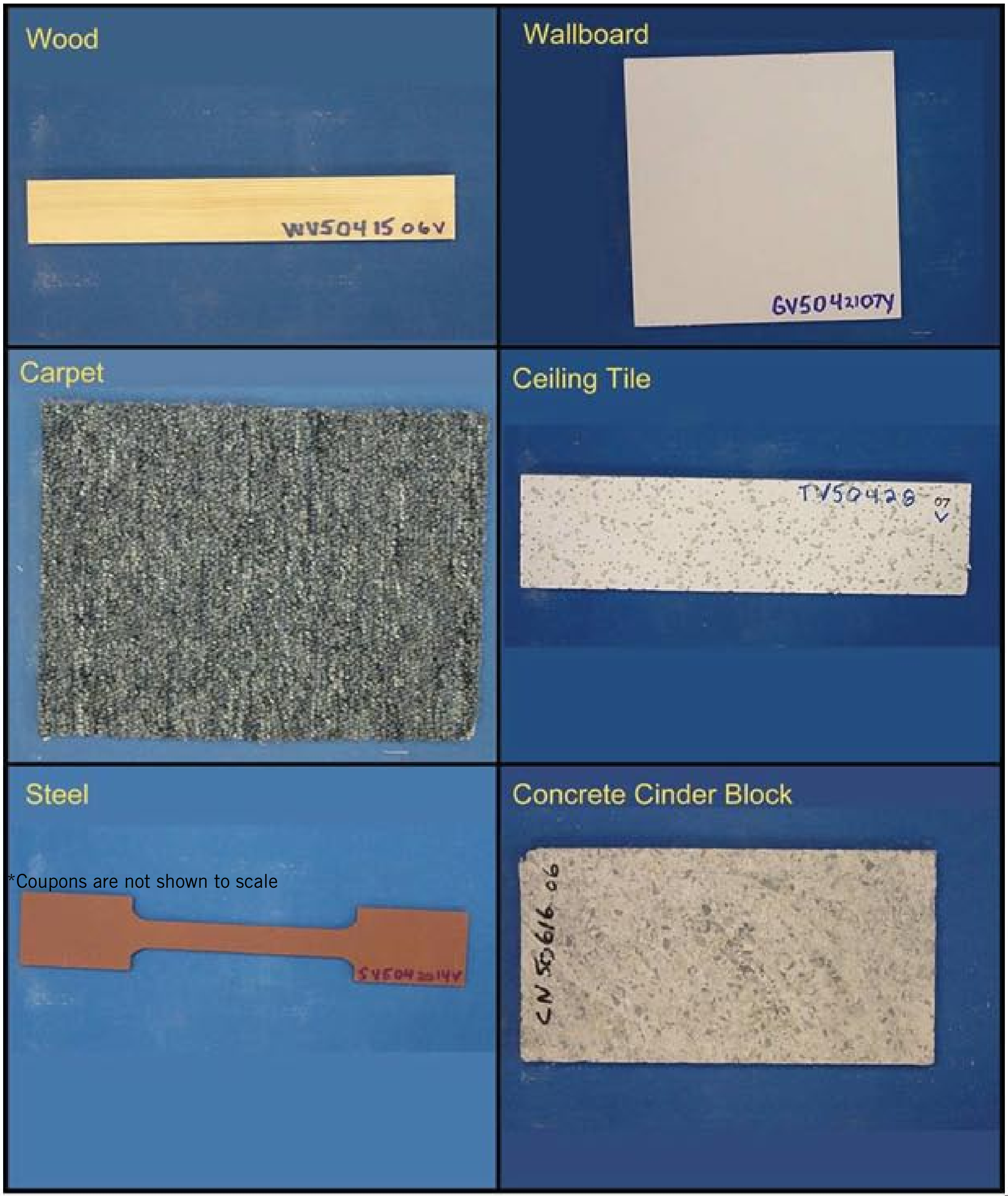
4.4 Visual Inspection

The coupons were visually inspected and digitally photographed upon removal from the chamber. Visual inspection of the coupon surfaces was conducted through side-by-side comparison of the decontaminated test surface and fresh coupons of the same test material. The testing staff looked for changes such as discoloration, blistering, warping, and peeling on the test coupons compared to the fresh coupons. After the visual inspection was completed, the coupon custody was transferred to the Material Compatibility Technical Leader for the three-month aging period and

Table 4.1: Representative Building Interior Materials

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

Figure 4.1: Samples of the Test Coupons*



*Coupons are not shown to scale

material compatibility testing. The coupons were examined again at the time of the material testing and the visual appearance recorded on the data test forms. If the coupon showed dramatic changes compared to a fresh coupon, then the coupon was photographed and the photograph included in the report. Representative photographs of each material type are provided in the report.

4.5 Coupon Aging

The material compatibility studies were conducted using the coupons from the material demand study. The coupons were aged for a minimum of 90 days following exposure to the decontaminant prior to material compatibility testing. The coupons were placed in open containers and stored under ambient conditions. The open container arrangement allowed aging of the coupons in conditions mimicking real-world aging.

4.6 Data Review and Technical Systems Audits

The approved Material Compatibility QAPP specified procedures for the review of data and independent technical system audits. All data were peer reviewed within two weeks of collection. The project quality manager (or designee) was required to audit at least 10% of the data collected. The project quality manager (or designee) performed three 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.

4.7 Physical Testing

An Instron model 5582 was used for the physical property testing. The Instron is a universal testing machine capable of performing tensile, compression, shear, peel, and flexural tests on most materials and components. Each material subsection contains a photograph of the coupon loaded into the test apparatus. The Instron model 5582 specifications are listed in Table 4.2.

Table 4.2: Instron Model 5582 Specifications

Load Capacity:	kN	100
	Kgf	10000
Maximum Speed:	mm/min	500
Minimum Speed:	mm/min	0.001
Maximum Force at Full Speed:	kN	75
Maximum Speed at Full Load:	mm/min	250
Return Speed:	mm/min	600
Position Control Resolution:	µm	0.06
Total Crosshead Travel:	mm	1235
Total Vertical Test Space:	mm	1309
Height:	mm	2092
Width:	mm	1300
Depth:	mm	756
Weight:	kg	862

4.8 Statistical Analyses

The data from the material compatibility testing phase of the systematic decontamination program was subjected to a statistical analysis to determine whether the differences observed among the various test sets were merely the result of random variations in test data or represented actual differences in the performance of the materials as a result of exposure to fumigation chemicals.

Methods used were from the statistical analysis functions embedded within both the Microsoft Excel software and *Practical Statistics for Analytical Chemists*, by Robert L. Anderson, © 1987, Van Nostrand Reinhold Company.

First, the individual coupon sets were tested for statistical outliers that could be eliminated from the data. The Q-Test for outliers was first used to identify potential outliers within a test set. Then the test group of coupons that had undergone similar treatment (controls, half-target, or full-target exposures) was tested. If an outlier identified in the individual coupons was also picked out in the test group analysis, it was eliminated and the statistics (averages and standard deviations) recalculated. However, if the specific data point was not identified as an outlier by both tests, it was retained in the study. Once statistical outliers had been eliminated, the test groups were analyzed to determine whether they were statistically significantly different — that is, to determine whether the treatment with the chosen fumigant had a detectable effect on the sample.

The primary test used was the Welch's T-test; the two-tailed, heteroscedastic test was used for the analysis. Welch's T-test values were calculated to compare the test groups, and results are reported for the 95% level of confidence. The percent level of confidence reported indicates the confidence that the two sample groups being compared are, in fact, different, and represent truly different samples. A 95% level of confidence indicates that there is a 5% chance that the two samples are, in fact, subparts of the same population. If a comparison determines that a sample is significantly different at the X% level of confidence, it is also significantly different at any lower level of confidence.

Determination that a control and exposed sample are statistically different implies that the treatment had some detectable effect on the material. Statistically different results do not imply that the material will fail as a result of treatment, unless the material no longer meets specifications. In some cases measured values may vary by several percent; however, there is no statistically detectable difference. It cannot be assumed that this difference is real unless the difference is statistically detected (e.g., by a Welch's T-test).

4.9 Chemical Testing: Ion Chromatography

The program also included additional CD tests of metals commonly used in electrical applications. Metal samples were exposed to CD and analyzed for the presence of chloride, chlorite, chlorate, and perchlorate anions in aqueous CD decontamination matrices using ion chromatograph (IC) with conductivity detection. The results of this study are discussed in Section 13 of this report.

5
Post-Fumigation Inspection Results

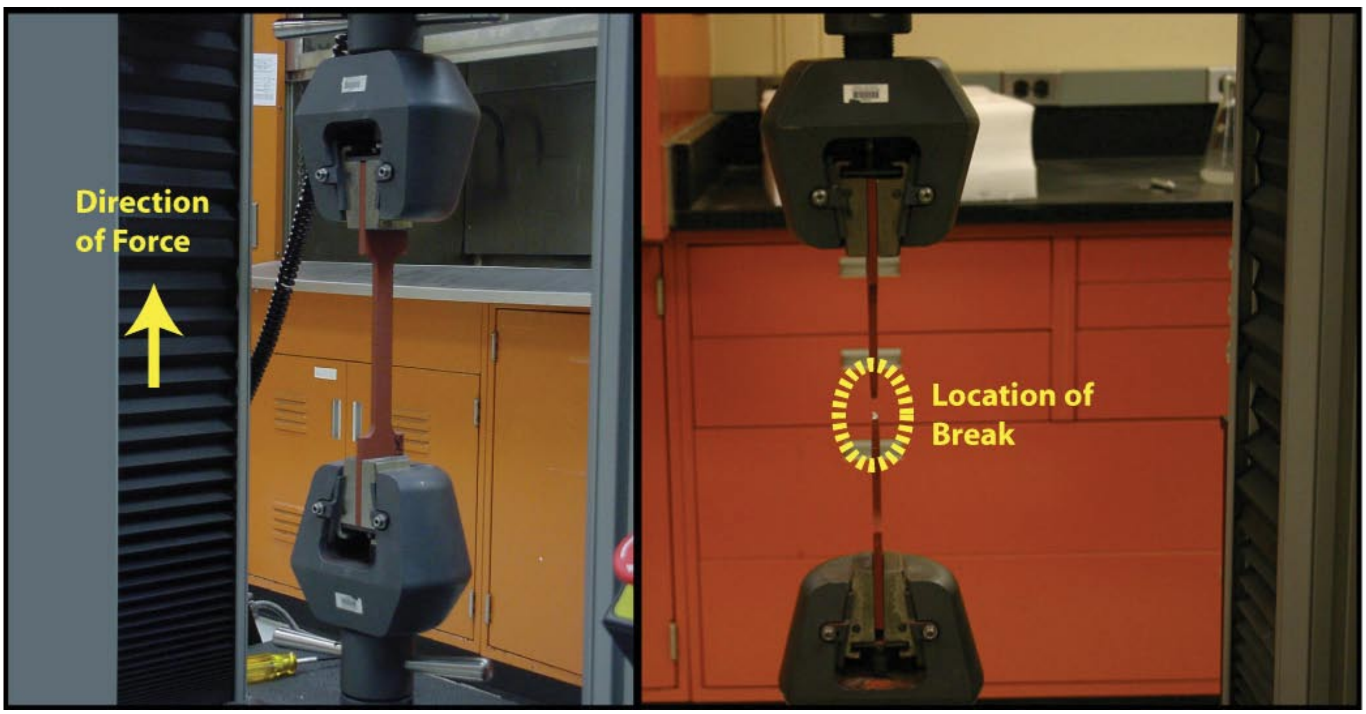
The coupons were visually inspected prior to fumigation, immediately after fumigation (post-fumigaiton), and after storage (post-storage) at time of material testing. Carpet coupons were inspected for any frayed tufts, pulled loops, and other noticeable defects. Concrete coupons were inspected for cracks, chips — particularly at the corners — any raised ridge sections, and other noticeable defects. Steel coupons were inspected for rust, peeling paint, any ridged sections on the small I-beam cross section, and any other noticeable defects. Tile coupons were inspected for crushed corners and edges, and any other noticeable defect. Wallboard coupons were inspected for any damage to the paper section, as well as any other noticeable defects. Wood coupons were inspected for any knots, missing knots, splitting, and other noticeable defects. The post-fumigation and post-storage inspections were compared to the initial inspections. No differences were observed for any of the coupons after CD exposure and aging compared to before CD exposure.

6
Evaluation of Structural Steel

6.1 Introduction
The effects of CD on the physical integrity of steel were investigated using the tension test as described in ASTM test method A370-03a "Standard Test Methods and Definitions for Mechanical Testing of Steel Products," Sections 5 to 13. The tension test was used to determine the integrity of steel coupons exposed to vaporous decontaminant compared to unexposed (control) steel coupons.

6.2 Sample Preparation and Testing
The steel coupons were removed from storage, visually inspected, and measured to confirm that they were within coupon specifications listed in Appendix A. The coupons from chamber positions 1, 4, 7, 10, and 16 were selected for testing in order to obtain representation throughout the test chamber. The coupons were used "as is" without any additional preparation. The testing was conducted in accordance with ASTM Test Method A370-03a. The Instron fixture for the steel test was installed into the Instron universal testing machine prior to testing. The testing machine operation and calibration verification was conducted by suspending a certified weight from the fixture and recording the weight. Three sets of five coupons were tested for each concentration (full-target and half-target) and four sets were tested for the controls (0 ppm). The load required to rupture the steel coupons was measured in Newtons (N). The tensile strength is the maximum tensile stress that a material is capable of sustaining and is calculated by dividing the amount of force required to rupture a specimen by the specimen cross-sectional area. No precision or bias requirements have been established for this test method. The results for control coupons were compared against the results for decontaminant-exposed samples. A statistical analysis of the data was conducted to determine whether the decontaminant-exposed steel coupon results were statistically different compared to the control steel coupons results.

Figure 6.1: Steel Coupon Set



6.3 Results

The coupons were stored for at least 90 days after fumigation. The actual number of storage days was based on the arrival of the Instron fixture for testing. The coupons for a particular fumigation trial were studied after a similar number of days in storage. A photograph of a representative steel sample before and after testing is provided in Figure 6.1. Values for the load required to rupture the steel coupons, the tensile strength results, and the number of days in storage before testing are provided in Table 6.1.

6.4 Discussion

The steel studied was an A572 Grade 50 high-strength, structural steel. The minimum tensile strength requirement is 450 N/mm^2. Both the control coupons and CD-exposed coupons met this minimum specification.

Of the 50 coupons tested in this portion of the program, only one generated a result that could be rejected as a statistical outlier from within its individual test set at the Q=0.99 level of confidence: Coupon 5 from test set SN50302, which had a tensile strength value significantly below the others in its test set. Within test groups (control samples, half-target concentration samples, and full-target concentration samples), similar statistical analysis showed that none of the coupon sets could be eliminated as statistical outliers. Therefore, Coupon 5 was retained for this analysis.

The values for the maximum load for the steel coupons were determined to be 61136 ± 1347 N for the control samples, 62308 ± 1560 N for the half-target samples, and 62639 ± 1538 N for the full-target coupons. The value for the tensile strength of the steel coupon is the maximum load (Newton) divided by the cross-sectional area (mm^2) of the coupon at the break point. The values for the tensile strength of the steel coupons were calculated to be 553 ± 14 N/mm^2 for the control coupons, 545 ± 24 N/mm^2 for the half-target coupons, and 554 ± 20 N/mm^2 for the full-target coupons.

With regard to the data from the test group of coupons, the average maximum load values for the CD-exposed coupons differed by less than 3% from the control samples, and the tensile strengths varied by about 1%. The Welch's T-test was used to determine whether any of the groups of samples were statistically different from the others. The tensile strength results showed no statistical difference between the controls, half-, or full-target concentration samples at the 95% confidence level. The average maximum load values for the half-target or full-target coupon samples, however, were statistically different from the control samples at the 95% confidence level. This is a result of slight differences in the cross-sectional areas between the groups that were still within the target tolerance values for quality control and not due to an effect of the fumigation process.

Table 6.1: CD Steel Coupon Test Results

Maximum Load	Control Samples (0 ppm) Tension Test Results, N				Half-Target Concentration (1000–1250 ppm) Results, N			Target Concentration (2000–2500 ppm) Results, N		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon set	SN50302	SSN51018	SSN50525	SN50228	SSD50928	SSD51004	SSD51005	SSD50919	SSD50921	SSD50927
Coupon 1	60616	60025	61501	61175	62076	63593	62285	60049	63305	64290
Coupon 2	60916	58303	61041	61559	63920	63181	64543	64989	62608	63527
Coupon 3	61191	63318	62511	60806	61906	58890	63163	60248	62549	59927
Coupon 4	60890	59384	60302	60731	61904	62222	59911	62529	63792	63872
Coupon 5	61049	64803	61698	60900	64228	61673	61122	61831	63135	62932
Test Average	60932	61167	61410	61034	62807	61912	62205	61929	63078	62909
Standard Deviation	214	2763	817	338	1164	1852	1791	2006	516	1740
Test Set Average ± Standard Deviation	61136 ± 1347				62308 ± 1560			62639 ± 1538		
Tensile Strength	**Control Samples (0 ppm) Tensile Strength, N/mm²**				**Half-Target Concentration (1000–1250 ppm) Results, N/mm²**			**Target Concentration (2000–2500 ppm) Results, N/mm²**		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon set	SN50302	SSN51018	SSN50525	SN50228	SSD50928	SSD51004	SSD51005	SSD50919	SSD50921	SSD50927
Coupon 1	561	527	569	537	575	558	546	556	586	536
Coupon 2	564	540	535	570	561	554	538	570	522	529
Coupon 3	567	555	548	563	573	545	475	558	579	526
Coupon 4	564	550	558	562	543	546	526	579	560	560
Coupon 5	536	540	571	534	563	541	536	542	554	552
Test Average	558	542	557	553	563	549	524	561	560	541
Standard Deviation	13	11	15	17	13	7	29	14	25	15
Test Set Average ± Standard Deviation	553 ± 14				545 ± 24			554 ± 20		
Number of Days in Storage	**Control Samples (0 ppm) Days**				**Half-Target Concentration Days**			**Target Concentration Days**		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon set	SN50302	SSN51018	SSN50525	SN52228	SSD50928	SSD51004	SSD51005	SSD50919	SSD50921	SSD50927
Coupon 1	103	159	96	98	173	168	167	220	218	181
Coupon 2	103	159	96	98	173	168	167	220	218	181
Coupon 3	103	159	96	98	173	168	167	220	218	181
Coupon 4	103	159	96	98	173	168	167	220	218	181
Coupon 5	103	159	96	98	173	168	167	220	218	181
Test Set Average ± Standard Deviation	114 ± 27				169 ± 3			206 ± 19		

Note: The cell highlighted in yellow indicates that the data point was statistically identified as an outlier within its test set but not within the test group (four control groups); therefore, the value was retained.

7
Evaluation of Gypsum Wallboard

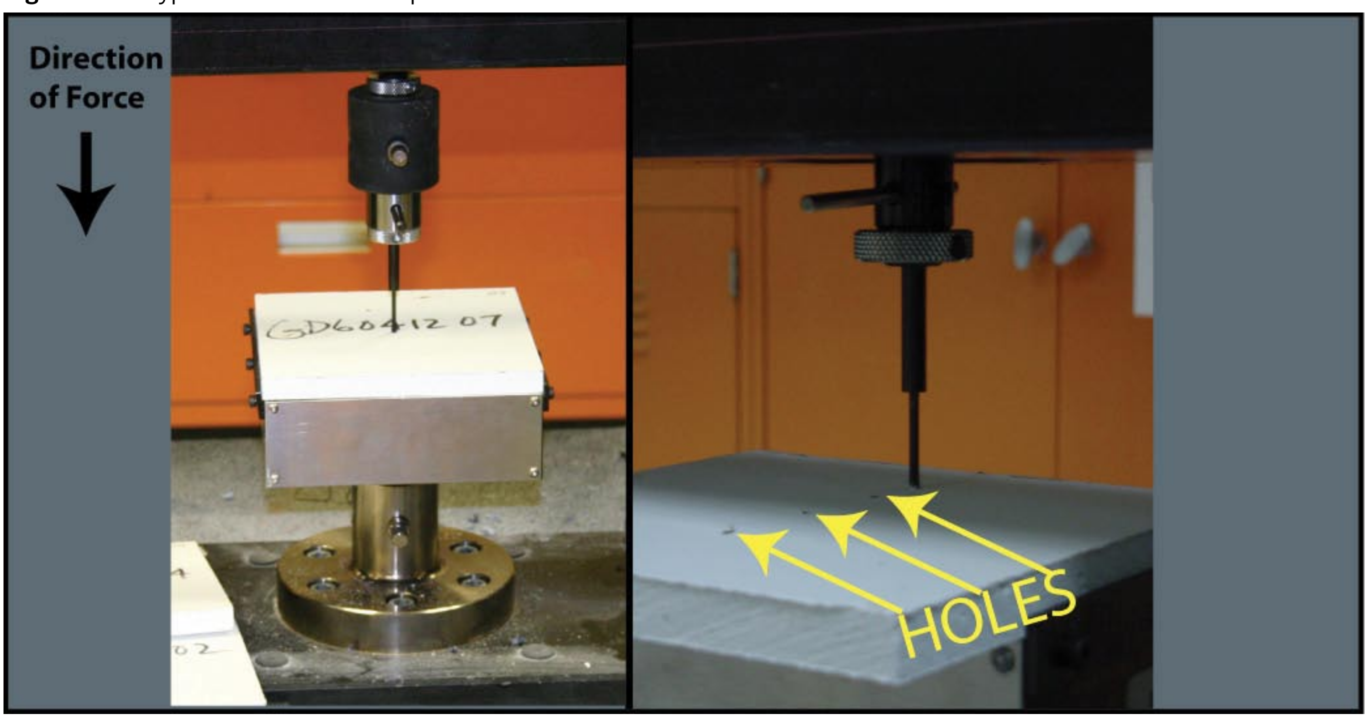
7.1 Introduction

The effects of CD fumigation on the physical integrity of gypsum wallboard were investigated using the nail pull-through resistance test method B as described in ASTM Test Method C473-03 “Standard Test Methods for Physical Testing of Gypsum Panel Products,” Section 13. The test measures the ability of the wallboard to resist nail pull-through by determining the load required to push a standard nail through the wallboard. The ASTM test was used to determine the integrity of the gypsum wallboard coupons exposed to vaporous decontaminant compared to unexposed (control) gypsum wallboard coupons.

7.2 Sample Preparation and Testing

The gypsum wallboard coupons were removed from storage, visually inspected, and measured. The coupons from chamber positions 1, 2, 4, 5, and 7 were selected for testing in order to obtain representation throughout the test chamber. The samples were brought to moisture equilibrium such that the weight of the sample did not change by more than 0.2% on successive weighings at a minimum interval of two hours. The sample preparation was conducted within a range of 15–25 °C and 48–75% RH. The testing was conducted in accordance with ASTM Test Method C473-03. The Instron fixture for the gypsum wallboard test was installed prior to testing. The Instron universal testing machine operation was verified by suspending a certified weight from the fixture and recording the weight. Three sets of five coupons were tested for each concentration (full-target and half-target) and four sets were tested for the controls (0 ppm). The force required to drive a nail shank through the wallboard coupons was measured in N. The ASTM method indicates that any coupon measurement in the series that varies 15% more than the average needs to be discarded. The method states that if 15% of the coupons deviate from the average, the test will be repeated. No additional precision or bias requirements have been determined for this test by ASTM. The results for control coupons were compared against the results for decontaminant-exposed coupons. A statistical analysis of the data was conducted to determine whether the decontaminant-exposed coupon results were statistically different from the control coupon results.

Figure 7.1: Gypsum Wallboard Coupon Test



7.3 Results

The coupons were stored for at least 90 days after fumigation. The actual number of storage days was based on the arrival of the Instron fixture for testing. The coupons for a particular fumigant were studied at the same number of days.

A photograph of a gypsum wallboard sample before and after testing (i.e., holes) is provided in Figure 7.1. Values for the load required to push the nail through the wallboard coupons and number of days in storage before testing are provided in Table 7.1.

Table 7.1: Gypsum Wallboard Coupon Test Results for Maximum Load

Force	Control Samples (0 ppm) Tension Test Results, N				Target Concentration (2000–2500 ppm) Results, N			Half-Target Concentration (1000–1250 ppm) Results, N		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon set	GN50316	GN50301	GN50518	GN50512	GD50417	GD60412	GD60413	GD60418	GD60420	GD602424
Hole 1	64.2	55.1	43.4	45.3	57.1	40.2	67.88	61.29	56.33	47.96
Hole 2	62.3	44.1	37.3	41.6	69.4	49.6	65.32	52.19	47.72	50.49
Hole 3	60.8	48.8	37.0	47.2	72.1	43.9	70.30	65.02	62.27	63.78
Hole 4	71.0	54.3	38.3	52.0	56.4	47.8	69.5	55.19	55.18	57.92
Hole 5	61.0	45.8	43.8	51.2	67.4	41.0	73.48	58.36	57.50	59.45
Test Average	63.8	49.6	39.9	47.5	64.5	44.5	69.3	58.4	55.8	55.92
Standard Deviation	4.2	5.0	3.4	4.3	7.3	4.1	3.0	5.0	5.3	6.5
Test Set Average ± Standard Deviation	50.2 ± 9.7				59.4 ± 12.1			56.7 ± 5.4		
Days in Storage	175	190	349	355	157	161	163	157	155	151

7.4 Discussion

The wallboard tension test results were analyzed for potential statistical outliers using the Q-test and for differences between the control and exposed samples using Welch's T-test. Although there was a great deal of scatter in the data (the data ranged from 37.0 to 73.5 N, the standard deviations of the results were between 9 and 21% of the mean value within the various test groups); therefore, none of the individual coupons were determined to be outliers at the Q=0.99 confidence level.

The average tension test results were 50.2 ± 9.7 N for the control group, 56.7 ± 5.4 N for the half-target group, and 59.4 ± 12.1 N for the full-target group. The differences were not determined to be statistically significant at the 95% confidence interval as determined by the Welch's T-test. Therefore, it did not appear that exposure to the CD fumigation process impacted the wallboard with respect to the ASTM tension test used for analysis.

8
Evaluation of Acoustical Ceiling Tile

8.1 Introduction

The effects of CD on the physical integrity of ceiling tile were investigated using the transverse strength test as described in ASTM Test Method C367-99 “Standard Test Methods for Strength Properties of Prefabricated Architectural Acoustical Tile or Lay-In Ceiling Panels” Sections 1, 3–5, and 21–29. The test measures the force required to cause the tile to break. The ASTM test was used to determine the integrity of ceiling tile coupons exposed to vaporous decontaminant compared to unexposed (control) ceiling tile coupons.

8.2 Sample Preparation and Testing

The acoustical ceiling tile samples were removed from storage, visually inspected, and measured. The samples were brought to moisture equilibrium such that the weight of the sample did not change by more than 1% on successive weighings at a minimum interval of two hours. The sample preparation was conducted within a range of 18–24 °C and 48–75% RH. The testing was conducted in accordance with ASTM Test Method C367-99. The Instron fixture for the ceiling tile test was installed prior to testing. The Instron universal testing machine operation was verified by suspending a certified weight from the fixture and recording the weight. For each test, the coupons from chamber positions 1 through 8 were selected for testing; this selection consisted of all coupons placed in the chamber during a single fumigation trial. Ceiling tile coupons were tested in two directions — with the mandrel parallel to the axis of the test machine (hereafter referred to as “machine direction”) and with the mandrel perpendicular to the axis (“cross-machine direction”). Three sets of four machine-direction coupons and four cross-machine-direction coupons were tested for each concentration (full-target and half-target) and four sets were tested for the controls (0 ppm) for each direction. The load required to break the ceiling tile coupons was measured in N. Figure 8.1 shows a photograph of a coupon loaded into the Instron for the machine and cross-machine direction tests. No precision or bias requirements have been established for this test method. The results of control coupons have been compared to the results for decontaminant-exposed tiles. A statistical analysis of the data was conducted to determine whether the decontaminant-exposed coupon results were statistically different from the control coupon results.

The Modulus of Rupture (MOR) was calculated according to the test method using the following equation:

$$\text{MOR units N/mm}^2\ (\text{lbf/in}^2) = \frac{3 \times P \times L}{2 \times b \times d^2}$$

where P is the maximum load, N (lbf)
L is the length of span, mm (in.)
b is the specimen width, mm (in.)
d is the specimen thickness, mm (in.)

Figure 8.1: Acoustical Ceiling Tile Coupon Test

8.3 Results

The coupons were stored for at least 90 days after fumigation. The actual number of storage days was based on the arrival of the Instron fixture for testing. The coupons for a particular fumigation trial were studied at the same number of days. A photograph of a representative ceiling tile sample before and after testing is provided in Figure 8.2. Values for the load required to break the ceiling tile coupons, the ceiling tile coupon MOR results, and number of days in storage are provided Table 8.1.

Figure 8.2: Representative Break - Acoustical Ceiling Tile Coupons

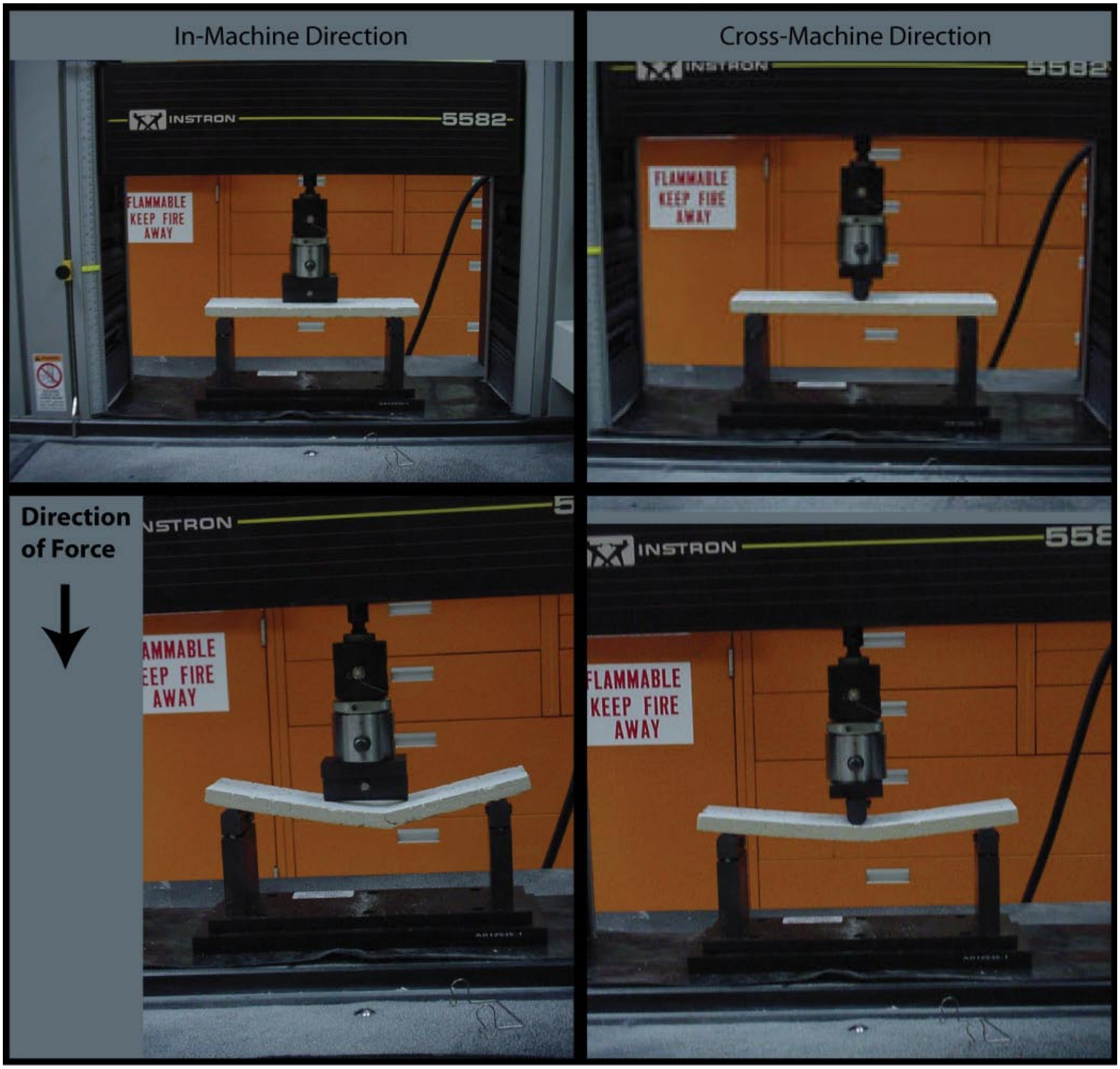


Table 8.1: CD Coupon Test Results for Tile

Maximum Load MACHINE DIRECTION	Control Samples (0 ppm) Tension Test Results, N				Half-Target Concentration (1000–1250 ppm) Results, N			Target Concentration (2000–2500 ppm) Results, N		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon set	TN51024	TN50223	TN50517	TN50519	TD60301	TD60329	TD60328	TD60228	TD60223	TD60227
Coupon 1	37.10	31.27	51.49	38.82	44.48	42.80	41.17	34.31	40.08	32.58
Coupon 2	39.63	29.80	32.19	43.15	41.09	42.80	41.13	41.68	35.96	32.87
Coupon 3	33.44	30.82	36.85	41.70	34.79	36.71	37.14	36.69	36.75	33.19
Coupon 4	36.73	34.54	40.48	42.76	31.88	43.76	41.01	38.21.	40.03	29.99
Coupon 5		31.19								
Test Average	36.73	31.52	40.25	41.61.	38.06	41.52.	40.11	37.72	38.21	32.16
Standard Deviation	2.54	1.78	8.22	1.96	5.75	3.24	1.98	3.09	2.16	1.47
Test Set Average ± Standard Deviation	37.17 ± 5.72				39.90 ± 3.89			36.03 ± 3.56		
Modulus of Rupture MACHINE DIRECTION	Control Samples (0 ppm) Tensile Strength, N/mm^2				Half-Target Concentration (1000–1250 ppm) Results, N/mm^2			Target Concentration (2000–2500 ppm) Results, N/mm^2		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon set	TN51024	TN50223	TN50517	TD50519	TD60301	TD60329	TD60328	TD60228	TD60223	TD60227
Coupon 1	0.86	0.72	1.19	0.91	1.03	0.98	0.95	0.79	0.93	0.75
Coupon 2	0.92	0.69	0.75	1.00	0.95	0.99	0.95	0.97	0.83	0.76
Coupon 3	0.77	0.71	0.85	0.95	0.76	0.85	0.86	0.85	0.85	0.77
Coupon 4	0.85	0.80	0.94	0.99	0.75	1.01	0.95	0.88	0.93	0.69
Coupon 5		0.72								
Test Average	0.85	0.73	0.93	0.96	0.87	0.96	0.93	0.87	0.88	0.74
Standard Deviation	0.06	0.04	0.19	0.04	0.14	0.07	0.05	0.07	0.05	0.03
Test Set Average ± Standard Deviation	0.86 ± 0.13				0.92 ± 0.09			0.83 ± 0.08		
Maximum Load CROSS-MACHINE	Control Samples (0 ppm) Tension Test Results, N				Half-Target Concentration (1000–1250 ppm) Results, N			Target Concentration (2000–2500 ppm) Results, N		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon set	TN51024	TN50223	TN50517	TN50519	TD60301	TD60329	TD06328	TD60228	TD60223	TD60227
Coupon 1	33.47	22.21	32.84	37.52	24.33	33.34	33.62	32.06	26.95	25.00
Coupon 2	21.19	19.57	33.67	36.24	37.33	35.45	32.80	25.40	27.75	25.35
Coupon 3	26.77	26.49	34.32	32.36	31.66	30.79	33.84	25.74	25.37	30.18
Coupon 4	26.80		32.11	33.72	37.16	31.99	27.96	26.88	24.97	26.23
Test Average	27.06	22.76	33.24	34.96	32.62	32.89	32.06	27.52	26.26	26.69
Standard Deviation	5.02	3.49	0.96	2.34	6.12	2.00	2.77	3.09	1.31	2.38
Test Set Average ± Standard Deviation	29.95 ± 5.68				32.52 ± 3.68			26.82 ± 2.22		
Modulus of Rupture CROSS-MACHINE	Control Samples (0 ppm) Tensile Strength, N/mm^2				Half-Target Concentration (1000–1250 ppm) Results, N			Target Concentration (2000–2500 ppm) Results, N		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon set	TN51024	TN50223	TN50517	TN50519	TD60301	TD60329	TD60328	TD60228	TD60223	TD60227
Coupon 1	0.78	0.51	0.76	0.87	0.56	0.77	0.78	0.74	0.62	0.58
Coupon 2	0.49	0.45	0.78	0.84	0.85	0.82	0.76	0.59	0.64	0.59
Coupon 3	0.62	0.61	0.80	0.75	0.73	0.71	0.78	0.60	0.59	0.70
Coupon 4	0.62		0.74	0.78	0.86	0.86	0.65	0.62	0.58	0.61
Test Average	0.63	0.52	0.77	0.81	0.75	0.79	0.74	0.64	0.61	0.62
Standard Deviation	0.12	0.08	0.02	0.05	0.14	0.06	0.06	0.07	0.03	0.06
Test Set Average ± Standard Deviation	0.69 ± 0.13				0.76 ± 0.09			0.62 ± 0.05		
Number of Days in Storage	Control Samples (0 ppm) Days				Half-Target Concentration Days			Target Concentration Days		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon set	TN51024	TN50223	TN50517	TN50519	TD60301	TD60329	TD60328	TD60228	TD60223	TD60227
Days in Storage	200	203	307	305	204	176	177	205	210	206
Test Set Average ± Standard Deviation	254 ± 60				186 ± 16			207 ± 3		

Note: Yellow-highlighted cells indicate that the data point was statistically determined to be an outlier within its test set but not within the test group (four control groups); therefore, the value was retained.

8.4 Discussion

Coupons were tested for maximum load in both the machine direction and cross-machine direction; these values were then used to calculate the MOR for each coupon type and test direction.

For the machine direction tests, the maximum load values were 37.17 ± 5.72 N for the control samples, 39.90 ± 3.89 N for the half-target coupons, and 36.03 ± 3.56 N for the full-target coupons. The MOR values for the machine direction tests were 0.86 ± 0.13 N/mm² for the control samples, 0.92 ± 0.09 N/mm² for the half-target coupons, and 0.83 ± 0.08 N/mm² for the full-target coupons.

For the cross-machine tests, the maximum load values were 29.95 ± 5.68 N for the control samples, 32.52 ± 3.68 N for the half-target coupons, and 26.82 ± 2.22 N for the full-target coupons. The MOR values for the cross-machine direction tests were 0.69 ± 0.13 N/mm² for the control samples, 0.76 ± 0.09 N/mm² for the half-target coupons, and 0.62 ± 0.05 N/mm² for the full-target coupons.

A statistical analysis of the individual test results was conducted to detect potential statistical outliers (Q-test) and determine whether there were any differences between the control and exposed samples (Welch's T-test).

Only one of the individual coupons was determined to be an outlier at the Q=0.99 confidence level (Coupon 3 of test set TD60328, half-target concentration) within its own test set. Because the machine direction maximum load of this coupon was statistically significantly different from others in the test set, the value for the MOR for that coupon was also a statistical outlier. However, when compared to all the other tests and coupons in the test group (half-target, machine direction), the coupon was not significantly different and therefore could not be rejected.

The half-target exposure coupons produced slightly higher maximum load values and higher MOR values (on average) than either the control or full-target coupons for both the machine direction and the cross-machine tests. However, at the 95% confidence level, there were no statistical differences between the control samples and either the half- or full-target concentration samples.

9
Evaluation of Carpet

9.1 Introduction

The effects of CD on the physical integrity of loop pile carpet fibers were investigated using ASTM Test Method C1335-03 “Standard Test Method for Tuft Bind of Pile Yarn Floor Coverings.” The method determines the force required to pull out a tuft of a pile yarn from a floor-covering sample (see Figure 9.1). The ASTM test was used to determine the integrity of loop pile carpet fibers exposed to vaporous decontaminant compared to unexposed (control) loop pile carpet fibers.

9.2 Sample Preparation

The carpet samples were removed from storage, visually inspected, and measured. The coupons from chamber positions 1, 3, 4, 5, and 7 were selected for testing in order to obtain representation throughout the test chamber. The samples were brought to moisture equilibrium such that the weight of the sample did not change by more than 0.2% on successive weighings at a minimum interval of two hours. The sample preparation was conducted within a range of 15–24 °C and 48–75% RH. The testing was conducted in accordance with ASTM Test Method D1335-03. The Instron fixture for the carpet test was installed prior to testing. The Instron universal testing machine operation and calibration verification was conducted by suspending a certified weight from the fixture and recording the weight. Three sets of five coupons were tested for each concentration (full-target and half-target) and four sets were tested for the controls (0 ppm). The load required to pull a carpet loop from the binding was measured in N. No bias requirements have been established for this test method. The results for control coupons were compared to the results for decontaminant-exposed samples. A statistical analysis of the data was conducted to determine whether the decontaminant-exposed coupon results were statistically different from the control coupon results.

9.3 Results

The coupons were stored for at least 90 days after fumigation. The actual number of storage days was based on the arrival of the Instron fixture for testing. The coupons for a particular fumigation trial were studied at the same number of days. The carpet tuft bind results and number of days in storage is provided in Table 9.1.

Figure 9.1: Carpet Coupon Test

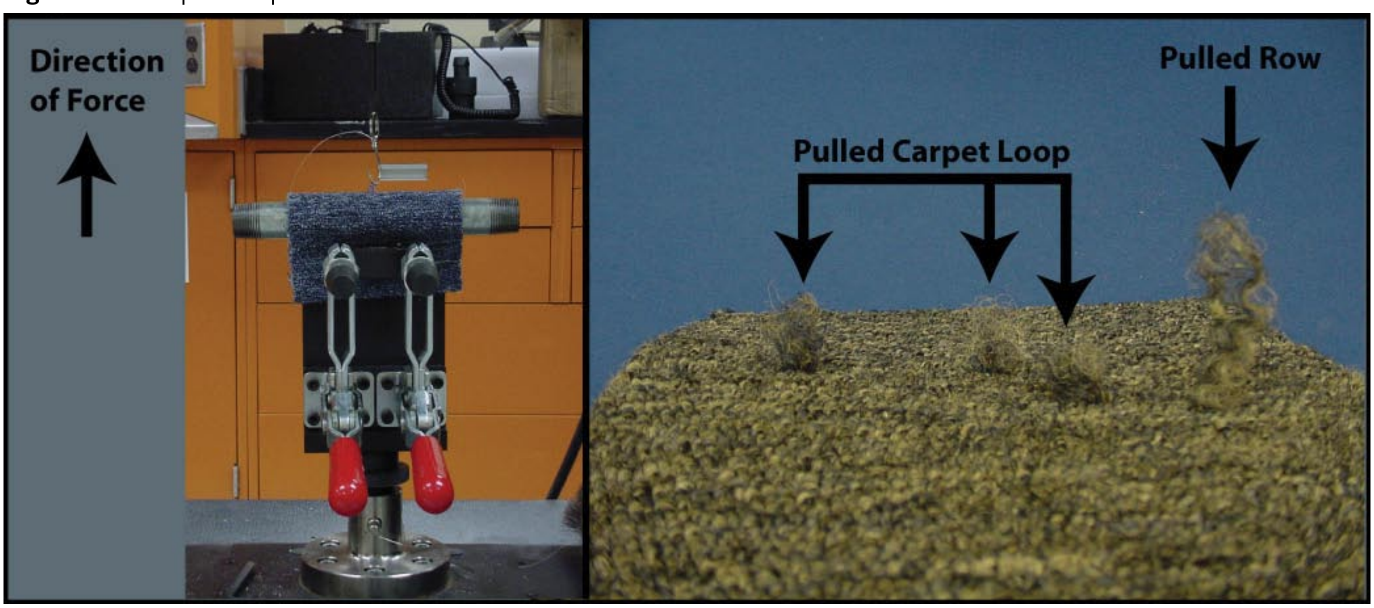


Table 9.1: Carpet Coupon Test Results for Average Tuft Bind - CD Control Samples

Tuft Bind Force	Control Sample (0 ppm) Results, N														
	RN50509					RN50505					RN50304				
Coupon	#1	#2	#3	#4	#5	#1	#2	#3	#4	#5	#1	#2	#3	#4	#5
Loop 1	14.9	10.8	14.3	20.9	21.6	10.8	24.3	11.7	17.0	19.8	15.1	10.3	29.8	12.1	18.1
Loop 2	8.9	17.0	15.0	19.4	16.2	16.2	20.2	11.2	17.3	18.4	20.1	23.6	15.2	13.7	18.4
Loop 3	10.0	18.2	11.8	18.0	16.4	18.1	20.9	12.0	17.7	9.9	21.9	14.7	14.6	13.6	14.7
Loop 4	16.4	13.8			16.8	21.1				16.9	10.4	15.3	20.8		
Loop 5	11.7	16.4				26.8				19.4	18.3		22.3		
Test Average	12.4	15.2	13.7	19.5	17.7	18.6	21.8	11.6	17.4	16.9	17.1	16.0	20.6	13.1	17.1
Standard Deviation	3.2	2.9	1.7	1.4	2.6	5.9	2.2	0.4	0.4	4.0	4.5	5.5	6.2	0.9	2.0
Days	158					162					151				
	RN50224														
Coupon	#1	#2	#3	#4	#5										
Loop 1	11.6	18.8	10.7	17.8	15.8										
Loop 2	13.5	7.0	9.2	12.7	15.2										
Loop 3	18.5	12.8	12.5	16.4	14.4										
Loop 4	21.3	23.6													
Loop 5	13.1	17.1													
Test Average	15.6	15.8	10.8	15.6	15.1										
Standard Deviation	4.1	6.3	1.7	2.6	0.7										
Days	159														
Test Set Average ± Standard Deviation	16.2 ± 4.3														
Tuft Bind Force	Target Concentration (2000–2500 ppm) Results, N														
	RD60330					RD60406					RD60404				
Coupon	#1	#2	#3	#4	#5	#1	#2	#3	#4	#5	#1	#2	#3	#4	#5
Loop 1	26.8	16.3	15.8	17.3	18.9	21.9	15.7	24.6	18.8	14.2	6.2	13.4	23.1	23.2	7.9
Loop 2	24.9	26.3	20.8	15.6	14.1	19.7	22.8	14.4	13.1	17.9	3.5	13.0	14.0	15.1	4.9
Loop 3	26.2	15.1	20.8	15.0	22.8	15.8	21.4	21.9	21.5	15.3	4.7	13.1	15.4	16.4	6.6
Loop 4		20.9			13.3	21.3	21.6	18.8	21.5		28.2		13.2	27.1	6.5
Loop 5		15.4			24.2	16.3	17.0	32.3	18.0		15.4		18.6	22.3	9.5
Test Average	25.9	18.8	19.1	16.0	18.7	19.0	19.7	22.4	18.6	15.8	11.6	13.2	16.9	20.8	7.1
Standard Deviation	1.0	4.8	2.9	1.2	5.0	2.8	3.1	6.7	3.4	1.9	10.4	0.2	4.1	5.0	1.7
Test Set Average ± Standard Deviation	17.5 ± 6.1														
Days	175					168					170				
Tuft Bind Force	Half-Target Concentration (1000–1250 ppm) Results, N														
	RD60411					RD60410					RD60405				
Coupon	#1	#2	#3	#4	#5	#1	#2	#3	#4	#5	#1	#2	#3	#4	#5
Loop 1	19.45	22.13	21.04	25.67	19.95	25.33	10.95	30.17	17.13	11.67	27.00	20.54	32.30	21.78	15.24
Loop 2	19.51	26.96	21.43	29.23	22.03	24.91	15.71	20.02	20.70	16.26	19.56	41.93	25.26	17.50	14.80
Loop 3	12.39	23.43	25.90	31.16	19.34	20.09	23.97	19.52	14.64	23.38	13.40	22.79	11.90	20.73	28.66
Loop 4	14.49						11.58	23.80	15.56	26.55	34.52	13.56	26.04		19.91
Loop 5	22.34						12.10	21.21		11.76	23.13	15.38	42.77		18.84
Test Average	17.6	24.2	22.8	28.7	20.4	23.4	14.9	22.9	17.0	17.9	23.5	22.8	27.7	20.0	19.5
Standard Deviation	4.1	2.5	2.7	2.8	1.4	2.9	5.4	4.4	2.7	6.8	7.9	11.3	11.3	2.2	5.6
Test Set Average ± Standard Deviation	21.3 ± 6.8														
Days	163					164					169				

Notes: The blank cells are samples that were not required to be analyzed, due to meeting the test method sampling criteria of ±15%.
The cells highlighted in yellow were determined to be outliers according to the Q-test at the 99% confidence interval within its test set but not within the test group (four control groups); therefore, the values were retained.

9.4 Discussion

A statistical analysis of the individual test results was conducted to detect potential statistical outliers (Q-test) and determine whether there were any differences between the control and exposed samples (Welch's T-test).

Although there was a great deal of scatter in the data (the standard deviations of the results were between 27 and 35% of the mean value within the various test groups), only one of the individual tuft pulls from a single coupon (Tuft 1, Coupon 3, RD60330) was significantly different from the others from the same carpet coupon. When comparing coupons within test groups, there was one coupon (Coupon 3, RN50224, control group) that was significantly different from others within the test group at the Q=0.99 confidence level. However, it was not statistically different when compared to all the control coupons. There were no statistical outliers in the half-target or full-target concentration tests.

The average tuft bind pull value was 16.2 ± 4.3 N for the control samples, 21.3 ± 6.8 N for the half-target concentration samples, and 17.5 ± 6.1 N for the full-target concentration samples.

A Welch's T-Test analysis was conducted on the samples to determine whether there were statistical differences between the control, half-, and full-target concentration samples. The half-target concentration results were determined to be statistically significantly different from both the control and target samples at the 95% confidence level, an unexpected result due to the large standard deviations of the three sets of data and their respective overlaps. The control and target concentration samples were not found to be significantly different at the 95% confidence level. The lack of difference for the full-target concentration samples and control samples indicates that it is not likely that the difference between the half-target data is due entirely to CD fumigation.

There appears to be a minor increase for the average tuft bind results with exposure to CD, but the trend is smaller than the standard deviations of the individual test results, so it is not clear whether it is an experimental artifact or a real trend.

These test methods show that exposure to CD may have a statistically significant effect on the tuft bind pull tests of carpet; further study is required to define the nature and magnitude of the effect.

10
Evaluation of Concrete Cinder Block

10.1 Introduction

The effects of CD on the physical integrity of concrete cinder block coupons were investigated using the compression test as described in ASTM Test Method C140-03 "Standard Test Methods for Sampling and Testing Concrete Masonry Units and Related Units." The ASTM test was used to determine the integrity of concrete cinder block coupons exposed to vaporous decontaminant compared to unexposed (control) concrete cinder block coupons.

10.2 Sample Preparation and Testing

The concrete cinder block samples were removed from storage, visually inspected, and measured. The coupons from chamber positions 1, 4, and 7 were selected for testing in order to obtain representation throughout the test chamber. The samples were brought to equilibrium in a range of 16–32 °C and less than 80% RH for 48 hours prior to testing. The testing was conducted in accordance with ASTM Test Method C140-03. The Instron fixture for the concrete cinder block test was installed prior to testing. The Instron universal testing machine operation and calibration verification was conducted by suspending a certified weight from the fixture and recording the weight. A photograph of a test sample loaded into the Instron test fixture is shown in Figure 10.1. Three sets of three coupons were tested for each concentration (0 ppm, full-target, and half-target). The load required to rupture the coupons was measured in N. No precision or bias requirements have been established for this test method. The results for control coupons were compared to the results for decontaminant-exposed samples. A statistical analysis of the data was conducted to determine whether the decontaminant-exposed coupon results were statistically different from the control coupon results.

Figure 10.1: Concrete "Cinder Block" Coupon Test

10.3 Results

The coupons were stored for at least 90 days after fumigation. The actual number of storage days was based on the arrival of the Instron fixture for testing. The coupons for a particular fumigation trial were studied at the same number of days. A photograph of a representative concrete cinder block sample before and after testing is provided in Figure 10.2. The coloring difference between the pictures is a result of the room lighting and is not real. Both samples were taken on the same blue color mat. Values for the load required to crush the concrete cinder block coupons, the coupon gross area compressive strength results, and number of days in storage are provided in Table 10.1. The concrete cinder block is a heterogeneous material sample to sample. The break patterns varied from sample to sample; a photograph of each sample is provided in Appendix D.

Figure 10.2: Representative Concrete Coupon Before and After Testing

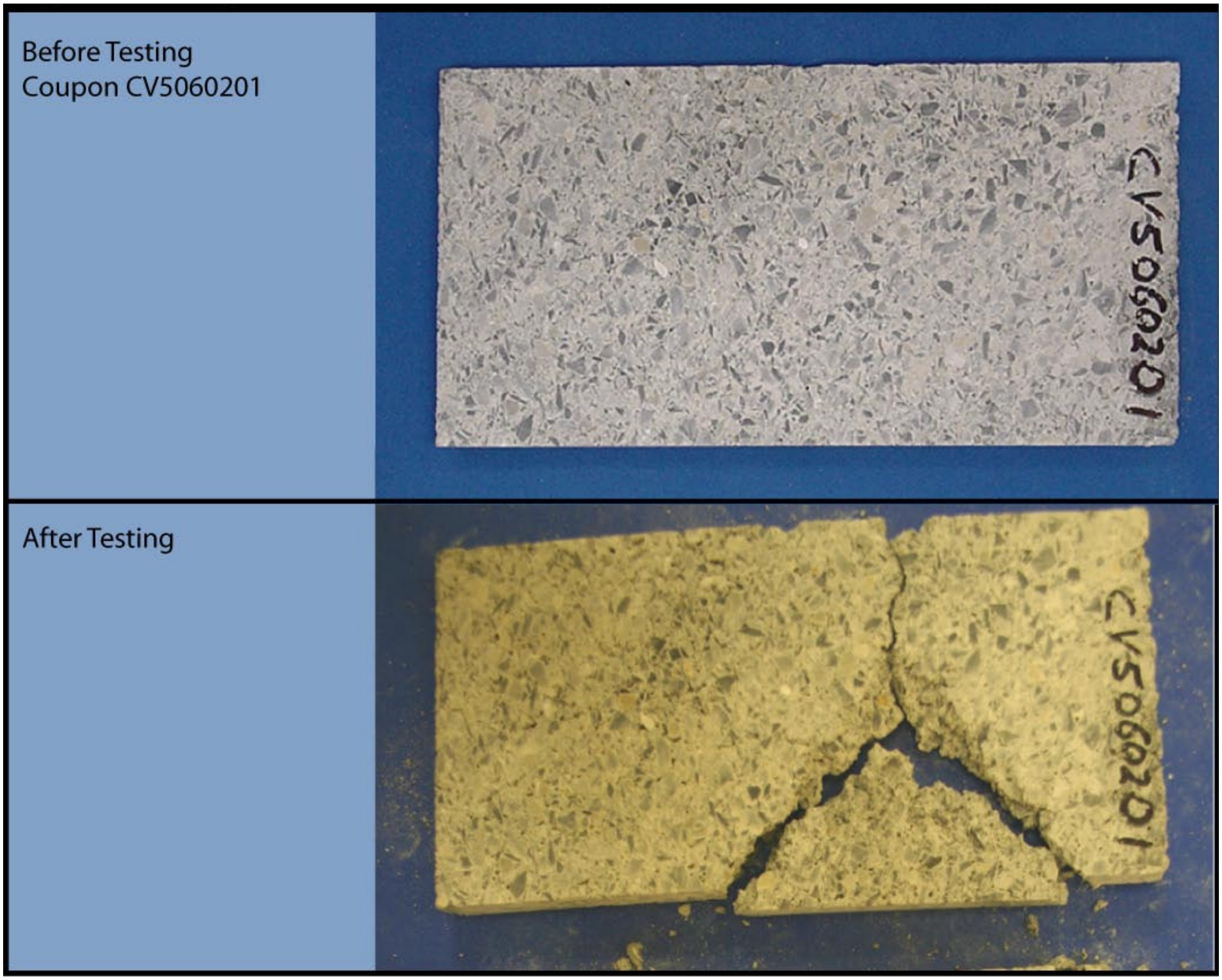


Table 10.1: Coupon Test Results for Concrete Cinder Block

Maximum Load	Control Samples (0 ppm) kgf			Half-Target Concentration (1000–1250 ppm) Results, kgf			Target Concentration (2000–2500 ppm) Results, kgf		
	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon set	CN50510	CN50524	CN51027	CD51129	CD51201	CD51205	CD51011	CD51012	CD51013
Coupon 1	3760	3252	4880	4243	3619	3091	5372	2959	3558
Coupon 2	3112	2711	3011	3001	4458	4107	2871	2376	3839
Coupon 3	2554	2557	3310	3074	2366	4596	3818	2790	4027
Test Average	3142	2840	3734	3439	3481	3931	4020	2708	3808
Standard Deviation	603	365	1004	697	1053	768	1262	300	236
Test Set Average ± Standard Deviation	3239 ± 729			3617 ± 776			3512 ± 898		
Gross Area Compressive Strength	**Control Samples (0 ppm) kgf/mm²**			**Half-Target Concentration (1000–1250 ppm) Results, kgf/mm²**			**Target Concentration (2000–2500 ppm) Results, kgf/mm²**		
	Test 1	**Test 2**	**Test 3**	**Test 1**	**Test 2**	**Test 3**	**Test 1**	**Test 2**	**Test 3**
Coupon set	CN50510	CN50524	CN51027	CD51129	CD51201	CD51205	CD51011	CD51012	CD51013
Coupon 1	1.9	1.5	2.3	2.2	1.7	1.6	2.8	1.4	1.8
Coupon 2	1.5	1.4	1.6	1.3	2.1	1.8	1.4	1.1	2.1
Coupon 3	1.1	1.2	1.7	1.5	1.2	2.2	2.0	1.3	1.8
Test Average	1.5	1.4	1.9	1.7	1.7	1.9	2.0	1.3	1.9
Standard Deviation	0.4	0.2	0.4	0.5	0.5	0.3	0.7	0.1	0.2
Test Set Average ± Standard Deviation	1.6 ± 0.4			1.7 ± 0.4			1.7 ± 0.5		
Number of Days in Storage	**Control Samples (0 ppm) Days**			**Half-Target Concentration Days**			**Target Concentration Days**		
	Test 1	**Test 2**	**Test 3**	**Test 1**	**Test 2**	**Test 3**	**Test 1**	**Test 2**	**Test 3**
Coupon set	CN50510	CN50524	CN51027	CD51129	CD51201	CD51205	CD51011	CD51012	CD51013
Coupon 1	126	127	219	128	126	211	162	211	160
Coupon 2	126	127	219	128	126	211	162	211	160
Coupon 3	126	127	219	128	126	211	162	211	160
Test Set Average ± Standard Deviation	157 ± 46			155 ± 42			178 ± 25		

10.4 Discussion

A statistical analysis of the individual test results was conducted to detect potential statistical outliers (Q-test) and determine whether there was a difference between the control and exposed samples (Welch's T-test). Within individual test runs for both maximum load and gross area compressive strength, there were no statistical outliers.

The values for the maximum load tests for concrete cinder blocks were 3239 ± 729 kgf for the control samples, 3617 ± 776 kgf for the half-target concentration samples, and 3512 ± 898 kgf for the full-target concentration samples. The values calculated for the gross compressive strength of the cinder block samples were 1.6 ± 0.4 kgf/mm² for the control samples, 1.7 ± 0.4 kgf/mm² for the half-target samples, and 1.7 ± 0.5 kgf/mm² for the full-target samples.

Comparing individual test set averages and gross area compressive strength within test groups, there were no statistical outliers.

When the averages for the exposed and nonexposed coupons are compared, we find no statistical differences among them. The Welch's T-test evaluation of the data indicates no statistically significant differences among the means of the exposed and control samples at the 95% confidence level. These test methods show that exposure to CD has no statistically significant effect on the maximum load or the gross area compressive strength of the cinder blocks tested.

11
Evaluation of Wood

11.1 Introduction

The effects of CD on the physical integrity of wood were investigated using the bending edge-wise test as described in ASTM Test Method D4761-02a "Standard Test Methods for Mechanical Properties of Lumber and Wood-Base Structural Material," Sections 6 to 11. The ASTM test was used to determine the integrity of wood coupons exposed to vaporous decontaminant compared to unexposed (control) wood coupons.

11.2 Sample Preparation

The wood samples were removed from storage, visually inspected, and measured. The coupons from chamber positions 1, 4, 7, 10, and 14 were selected for testing in order to obtain representation throughout the test chamber. The samples were brought to moisture equilibrium such that the weight of the sample did not change by more than 0.2% on successive weighings at a minimum interval of two hours. The sample preparation was conducted within a range of 15–25 °C and 48–75% RH. The testing was conducted in accordance with ASTM Test Method D4761-02a. The Instron fixture for the wood test was installed prior to testing. The Instron universal testing machine operation and calibration verification was conducted by suspending a certified weight from the fixture and recording the weight. Three sets of five coupons were tested for each concentration (full-target and half-target), and four sets were tested for the controls (0 ppm). The load required to rupture the wood coupons was measured in N. No precision or bias requirements have been established for this test method. The results for control coupons were compared to the results for decontaminant-exposed samples. A statistical analysis of the data was conducted to determine whether the decontaminant-exposed coupon results were statistically different from the control coupon results. A photograph of a wood sample loaded into the Instron is provided in Figure 11.1.

11.3 Results

The coupons were stored for at least 90 days after fumigation. The actual number of storage days was based on the arrival of the Instron fixtures for testing. The coupons for a particular fumigation trial were studied at the same number of days. A photograph of a representative wood sample before and after testing is provided in Figure 11.2. Values for the wood coupon results for the required load and time to break, moisture content, and number of days in storage are provided in Table 11.1. The wood samples vary slightly in knot and grain pattern from sample to sample. The break patterns varied from sample to sample; a photograph of each sample is provided in Appendix C.

Figure 11.1: Wood Coupon Test

Figure 11.2: Representative Wood Coupon Before and After Testing

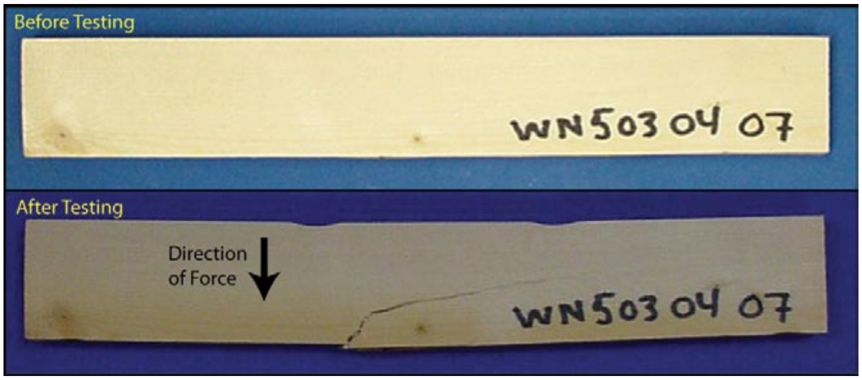


Table 11.1: CD Coupon Test Results for Wood

Maximum Force	Control Samples (0 ppm) Results, N				Half-Target Concentration (1000–1250 ppm) Results, N			Target Concentration (2000–2500 ppm) Results, N		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon set	WN50307	WN50317	WN50502	WN50504	WD60213	WD60207	WD60209	WD51206	WD51207	WD51208
Coupon 1	4945	3903	4516	4600	2360	4163	4712	4683	1906	3037
Coupon 2	2433	3546	5582	3248	3766	4714	4289	4514	3027	3860
Coupon 3	5130	2574	4031	5233	3312	3994	7141	3699	3516	3131
Coupon 4	3592	3494	6370	5170	3702	3651	3779	3741	4427	3095
Coupon 5	4825	4446	3475	4545	4015	4983	6553	4193	2995	3543
Test Average	4185	3593	4795	4559	3431	4301	5295	4166	3174	3333
Standard Deviation	1151	684	1173	798	650	541	1470	444	915	356
Test Set Average ± Standard Deviation	4283 ± 1009				4342 ± 1201			3558 ± 731		
Gross Area Compressive Strength	Control Samples (0 ppm) minutes				Half-Target Concentration (1000–1250 ppm) Results, min.			Target Concentration (2000–2500 ppm) Results, min.		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon set	WN50307	WN50317	WN50502	WN50504	WD60213	WD60207	WD60209	WD51206	WD51207	WD51208
Coupon 1	4.9	3.9	4.3	4.6	2.4	4.1	4.7	4.7	1.9	3.0
Coupon 2	2.4	3.6	5.6	3.2	3.7	4.7	4.3	4.5	3.0	3.9
Coupon 3	5.1	2.6	4.0	5.2	3.3	4.0	4.9	3.7	3.5	3.1
Coupon 4	3.6	3.5	6.1	4.6	3.7	3.6	3.8	3.7	4.3	3.1
Coupon 5	4.8	4.4	3.4	4.4	4.0	5.0	4.5	4.2	2.9	3.5
Test Average	4.2	3.6	4.7	4.4	3.4	4.3	4.4	4.2	3.1	3.3
Standard Deviation	1.1	0.7	1.1	0.7	0.6	0.5	0.4	0.4	0.9	0.4
Test Set Average ± Standard Deviation	4.2 ± 1.0				4.0 ± 0.7			3.5 ± 0.7		
Moisture Content	Control Samples (0 ppm) Results, %				Half-Target Concentration (1000–1250 ppm) Results, %			Target Concentration (2000–2500 ppm) Results, %		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon set	WN50307	WN50317	WN50502	WN50504	WD60213	WD60207	WD60209	WD51206	WD51207	WD51208
Coupon 1		0.12	-0.12	-0.40	-0.18	0.03	0.07	-1.00	-0.93	-0.84
Coupon 2		0.20	-0.17	-0.33	-0.14	0.11	0.00	-1.27	-1.31	-1.27
Coupon 3		0.17	-0.14	-16.61	-0.03	0.13	0.02	-1.29	-1.39	-1.30
Coupon 4		0.12	-0.13	-17.29	-0.12	0.14	0.00	-1.34	-1.39	-1.67
Coupon 5		0.17	-0.13	-0.24	-0.12	0.20	0.00	-1.53	-1.15	-1.00
Test Average		0.15	-0.14	-6.98	-0.12	0.13	0.02	-1.29	-1.24	-1.21
Standard Deviation		0.03	0.02	9.11	0.05	0.06	0.03	0.19	0.20	0.32
Test Set Average ± Standard Deviation	-2.32 ± 5.95				0.01 ± 0.11			-1.24 ± 0.23		
Number of Days in Storage	Control Samples (0 ppm) Days				Half-Target Concentration Days			Target Concentration Days		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon set	WN50307	WN50317	WN50502	WN50504	WD60213	WD60207	WD60209	WD51206	WD51207	WD51208
Coupon 1	135	125	182	372	220	227	225	149	148	147
Coupon 2	135	125	182	372	220	227	225	149	148	147
Coupon 3	135	125	182	372	220	227	225	149	148	147
Coupon 4	135	125	182	372	220	227	225	149	148	147
Coupon 5	135	125	182	372	220	227	225	149	148	147
Test Set Average ± Standard Deviation	204 ± 102				224 ± 3			148 ± 1		

Note: Yellow cells indicate that no data were collected.

11.4 Discussion

Of the 50 coupons tested to destruction in this portion of the program, no coupons could be eliminated as statistical outliers from within their individual test sets or test groups (control, half-target concentration, or full-target concentration samples) at the Q=0.99 level of confidence.

With regard to the data from the test group of coupons, the average maximum load values for the half-target CD exposed coupons increased slightly (~1%) over the value for the control set, while the maximum load dropped by 17% for the full-target concentration coupons; relative standard deviations were on the order of 21–28%. The average maximum force value for the control samples was 4283 ± 1009 N. The half-target concentration samples had an average maximum force value of 4342 ± 1201 N (an increase of 1.4%), while the full-target concentration samples had an average maximum force value of 3558 ± 731 N (a decrease of 17.4% from the control group). The maximum force for the full-target concentration exposed coupons was statistically significantly different from both the controls and from the half-target concentration coupons at a 95% confidence level using the Welch's T-test results.

Average time-to-break value for the control coupons was 4.1 ± 0.9 seconds; the half-target concentration coupons was 4.0 ± 0.7 seconds (a 2% decrease), and the full-target concentration coupons was 3.5 ± 0.7 seconds (a 15% decrease). The time-to-break values for the exposed coupons showed a slight downward trend. Welch's T-Test was again used to compare the time-to-break values of the different groups of coupons. At the 95% confidence level, the full-target concentration coupons were found to be significantly different from the control and the half-target coupons.

The average change in moisture content for the control samples after storage was −2.32 ± 5.95%. For the half-target concentration coupons, the average change in moisture content was +0.01 ± 0.11, and for the full-target concentration coupons the average change in moisture content was −1.24 ± 0.23%. The changes in moisture content were not suggested to be statistically significantly different at the 95% confidence level using the Welch's T-test.

The results suggest that fumigation under the full-target conditions used in this study may impact the force required to break the structural wood, in accordance with the ATSM test method used. At the higher fumigant concentration, the wood samples required less force and time to break than either the controls or half-target samples.

12
Evaluation of Electrical Circuit Breakers

12.1 Introduction

The impact of fumigant and humidity on the performance of electrical circuit breakers was also investigated in this study. This investigation involved circuit breakers prepared as baseline, test, and control. Baseline circuit breakers were the "as-purchased" circuit breakers. The test circuit breakers were prepared in the exposure chambers using fumigant. The control circuit breakers were prepared in the exposure chambers using a temperature and relative humidity profile similar to that of the test breakers.

12.2 Sample Preparation

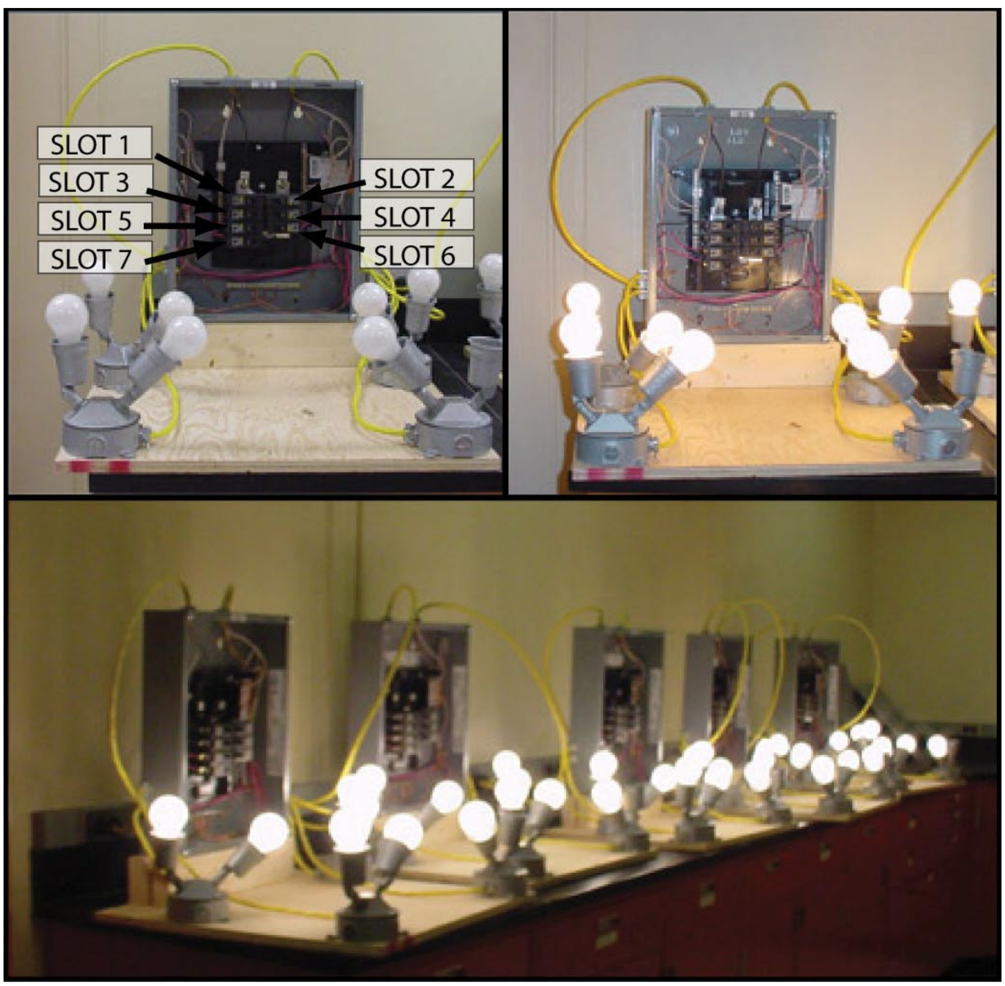
The single-pole, 20-amperes rated circuit breakers were purchased from Home Depot (model HOM120). All of the circuit breakers were installed in the testing stations to confirm that they were operational before exposure testing, and all were removed from the stations, numbered, and chain-of-custody initiated. The baseline circuit breakers were put aside until needed. The test and control exposure testing was discussed in Section 4. Each run used seven circuit breakers.

After a test or control circuit breaker set was prepared in the exposure chamber, the breakers were removed from the exposure chamber and visually inspected.

12.3 Circuit Breaker Testing Stations

After visual inspection, the breakers were installed in the testing station and observed for 90 days under load. A photograph of the testing station is shown in Figure 12.3. The testing station is an electrical box containing 8 spaces, 16 circuits, 100 amp max from square D (Home Depot # 577-340). The circuit breaker box was wired with 12-gauge, 20-amp wire into the 120-V outlet. Each circuit breaker was wired in series with an electrical lamp (s513e) with an outlet box (s110e) manufactured by Thomas & Bretts (Home Depot # c214477 and b214426, respectively). Each lamp contained a Phillips 40-watt light bulb (Home Depot #a356140). The test or control circuit breakers were installed into slots 1 through 7, and the baseline circuit breaker was installed in slot 8 (Figure 12.1, upper left picture). The room temperature and relative humidity were monitored daily.

Figure 12.1: Circuit Breaker Test Station



12.4 Results

The circuit breakers were exposed to fumigant and visually inspected after removal from the exposure chamber. No damage was observed on any of the circuit breakers following fumigation. The circuit breakers were then installed into the testing stations for 90 days. The stations were observed on each work day and light bulbs were replaced as needed. No breakers failed during the 90-day storage under load. Following the 90-day storage, the breakers were tested using current-time measurements done at 150% (30 amp) and 300% (60 amp) of the breakers' rated value. Tests were done using an AVO/multi-amp MS-2, available from Advanced Test Equipment Rentals. The test results are provided in Table 12.1. The circuit breaker data was statistically analyzed to determine whether the breaker was compromised after exposure to decontaminant by comparing the test results obtained with fumigant-exposed circuit breakers to those obtained with control coupons (not exposed to fumigant). Each breaker station contained one control breaker that had not been exposed in the chamber.

Table 12.1: Chlorine Dioxide Circuit Breaker Test Results

6-hour CD Box Test	60-Amp Test Time (sec)	30-Amp Test Time (sec)	6-hour CD Control	60-Amp Test Time (sec)	30-Amp Test Time (sec)
BD5090601	4.65	223.70	BN5022501	5.37	43.21
BD5090602	5.16	82.72	GN5022502	5.81	57.52
BD5090603	4.53	90.65	BN5022503	5.47	55.42
BD5090604	6.59	81.82	BN5022504	5.75	61.22
BD5090605	5.05	115.87	BN5022505	4.85	48.62
BD5090606	3.45	64.91	BN5022506	5.52	50.31
BD5090607	NA	64.87	BN5022507	5.37	48.45
			BN5022506 Retest		55.48
Baseline Breaker	6.08	62.14	Baseline Breaker	4.97	41.47
Test Average	4.91	103.51	Test Average	5.45	52.53
Standard Deviation	1.02	55.75	Standard Deviation	0.32	5.87
12-hour CD Box Test	**60-Amp Test Time (sec)**	**30-Amp Test Time (sec)**	**12-hour CD Control**	**60-Amp Test Time (sec)**	**30-Amp Test Time (sec)**
BD5091501	5.34	61.25	BN5022801	5.54	57.50
BD5091502	4.40	90.23	BN5022802	5.59	51.08
BD5091503	5.14	68.25	BN5022803	6.41	55.60
BD5091504	6.11	60.90	BN5022804	2.99	53.29
BD5091505	3.05	53.95	BN5022805	6.00	51.94
BD5091506	4.81	64.55	BN5022806	5.41	68.59
BD5091507	3.64	76.42	BN5022807	5.98	48.79
Baseline Breaker	3.28	60.99	Baseline Breaker	5.68	63.89
Test Average	4.64	67.94	Test Average	5.42	55.26
Standard Deviation	1.04	12.04	Standard Deviation	1.12	6.55

The measurement for the analysis was the time for the circuit breaker to open (Time-to-Open) when experiencing a current above its rated value. A circuit breaker that trips too quickly will protect personnel and equipment but can represent a significant loss of time and productivity. A circuit breaker that takes too long to trip could result in a heat buildup, and possibly a fire, and might fail to protect equipment, users, and property.

A statistical analysis of the individual test results was conducted to detect potential statistical outliers (Q-test) and determine whether there were any differences between the control and exposed samples (Welch's T-test) in the tests of circuit breakers exposed to CD.

The differing test groups were first checked to determine whether there were any statistical outliers using the Q-test. There were two statistical outliers found in the data at the Q=0.99 level of confidence: Circuit breaker BD5090601 from the 6-hour control tested at the 30-amp challenge and circuit breaker BN5022804 from the 12-hour exposed group tested with the 60-amp challenge. These data points are highlighted in orange in Table 12.1 and were discarded; the remainder of the statistical analysis was conducted without them.

Table 12.2 summarizes the data for the average and standard deviation for the various test groups. The Welch's T-test was used with a 95% confidence level in order to determine whether the changes in the Time-to-Open were significantly different among the different treatment (control, 1000 ppm, or 2000 ppm) and analysis challenge (30-amp or 60-amp) groupings. At the 60-amp challenge, the slight decreases in the Time-to-Open for the circuit breakers exposed to the CD fumigation conditions compared to their respective controls were not statistically significant. In addition, no statistically significant difference was observed due to exposure to the control conditions for 6 compared to 12 hours, nor as a function of fumigation conditions.

However, at the 30-amp challenge, the decrease in the Time-to-Open for the circuit breakers exposed to 2000 ppm of CD for 6 hours was statistically significantly different from the corresponding 6-hour control. At the lower concentration and longer fumigation time, the observed slight increase in Time-to-Open compared to the 12-hour control was not a statistically significant difference. As with the 60-amp challenge, no statistically significant difference was observed due to exposure to the control conditions for 6 compared to 12 hours. In addition, the observed difference in the Time-to-Open for the 6 hour versus 12 hour CD-exposed circuit breakers was also not statistically significant.

The results are somewhat conflicting, i.e., no statistically significant difference between the 6-hour and 12-hour CD exposure for the 60-amp challenge. However, it appears that exposure to the higher concentration of CD may have an effect on the performance of circuit breakers as determined from the Time-to-Open testing based on the comparison to the control. The results suggest that exposure to lower CD concentrations for longer times, rather than higher concentrations for shorter times, may have less deleterious effects on the circuit breakers. No specification was found to determine whether the observed effect at the 2000-ppm CD fumigation condition was within the device failure criteria.

Table 12.2: Summary of Time-to-Open Data

Exposure	30-Amp Challenge Time to Open (sec)	60-Amp Challenge Time to Open (sec)
6-Hour Control	52.53 ± 5.87	5.45 ± 0.32
6-Hour @ 2000 ppm CD	83.47 ± 18.95	4.91 ± 1.02
12-Hour Control	55.26 ± 6.55	5.82 ± 0.38
12-Hour @ 1000 ppm CD	67.94 ± 12.04	4.64 ± 1.04

13
Chlorine Dioxide Fate on Metals

13.1 Introduction

This study characterized the interaction of CD with aluminum, copper, and steel — materials common in electrical systems. Residual chloride by-products on the metals following treatment with CD were analyzed using ion chromatography. Analyses for other possible reaction products such as metal hydroxides and oxides were not attempted. The objective of this study was to identify the anionic chlorine species and concentrations formed on aluminum, steel, and copper after exposure to CD at 1000 and 2000 ppm (parts per million volume) for 12 and 6 hours, respectively.

13.2 Test Procedure

Aluminum (0.5″ W x 0.5″ L), copper (0.5″ D x 0.5″ L), and steel EMT conduit (0.5″ D x 0.5″) coupons were exposed to CD to determine the fate of the decontaminant on the metals. Coupons cut from metals purchased from Home Depot were exposed to chlorine dioxide concentrations of 1000 and 2000 ppm for 12 and 6 h, respectively, to give a total CT of 12000 ppm-hr for each experiment. The metals were exposed at 25 °C, 75–90% RH; control sets were exposed under the same conditions at 0 ppm CD. Fourteen coupons of each metal in small plastic containers were placed on the inside bottom of a PlasLabs model 830-ABC glove box (PlasLabs, Inc., Lansing, MI) during each exposure. The coupons were removed from the glove box after exposure and stored for 90 days in the plastic containers. After storage, each of the 14 coupons was placed in a 2-ounce wide-mouth glass jar with cap. Chloride, chlorite, chlorate, perchlorate, and other anions were removed from each sample by extraction in water (10 mL) with rocking. The extracts were then passed through a 0.22-µm syringe filter and injected directly into the chromatograph to determine retention time and detector response for each analyte present in the sample.[9] Dilution of samples with deionized distilled water was conducted when appropriate. The anions were then identified and quantified by comparison with standard solutions.

13.3 Sample Analyses

Sodium carbonate (analytical-grade) and HPLC-grade methanol used in preparing the mobile phase was purchased from Fisher Scientific (Fair Lawn, NJ). The chloride, chlorate, and perchlorate anion standards were obtained from SCP Scientific, Champlain, NY, and the chlorite anion standard was obtained from HPS Science, Charleston, SC. The analyses were carried out using an ion chromatograph with a Millennium32 Data Workstation equipped with a Rheodyne 77251i Injector, a Model 510 Pump, and a Model 432 Conductivity Detector (Waters Corporation, Milford, MA). Conductivity suppression was carried out using an ERIS 1000HP Autosuppressor (Alltech Corporation, Deerfield, IL).

Analyses for chloride, chlorite, and chlorate in the extracts were performed under the following conditions: column, ION-PAC AS9HC (Dionex Corporation, Sunnyvale, CA); mobile phase, 9 mM Na_2CO_3; flow rate, 1.0 mL/min; injection volume, 20 µL; and detection, suppressed conductivity (1 SFS). Standard solutions of each anion were injected onto the column and retention times of each analyte were determined. Calibration curves were obtained by injecting known concentrations (100, 40, 10, 1, and 0.4 µg/mL) of each anion in deionized water into the chromatograph in duplicate and measuring the conductivity response obtained.

Analysis of the extracts for perchlorate were performed under the following conditions: column, ION-PAC AS 14 (4 mm) (Dionex Corporation, Sunnyvale, CA); mobile phase, 9 mM Na_2CO_3 in 40% methanol; flow rate, 1.0 mL/min., injection volume, 20 µL; detection, suppressed conductivity (1 SFS). Standard solutions of perchlorate were injected onto the column and a retention time was determined. A calibration curve of perchlorate ion was obtained by injecting a known concentration (100, 40, 10, 4, 1, 0.4 µg/mL) of the anion in deionized water into the chromatograph in duplicate and measuring the conductivity response.

The detector response versus concentration for all of the species was determined to be linear. A typical regression line for each was determined using the least square method. The regression curve and linear correlation coefficient (R^2) for each target analyte were determined to be as follows:

- chloride, $y = 118486x - 136041$ ($R^2 = 0.9987$)
- chlorite, $y = 42201x - 53598$ ($R^2 = 0.9979$)
- chlorate, $y = 49100x - 43882$ ($R^2 = 0.9982$
- perchlorate, $y = 151113x - 135662$ ($R^2 = 0.9996$)

The reproducibility was determined to be within ±5 % of the mean.

Analyte standards at the 10 µg/mL concentration level were injected before and after a daily analytical run, with reproducibility having to be within ±10% of the mean. The unknown concentration of each species in the extracts was determined by correlating the detector response to the response versus concentration curve (standard curve) obtained for each target analyte. The detector response was substituted into the appropriate regression equation and the corresponding concentration was calculated.

13.4 Results

A summary of the CD reactions on aluminum, copper, and iron with the corresponding products and concentrations is provided in Table 13.1. A comparison of copper tubing exposed and not exposed to CD is provided in Figures 13.1 and 13.2. A layer of patina is evident on the tubing exposed to chlorine dioxide (Figure 13.2). Ion chromatography analyses of the extraction solvents from the aluminum controls detected chloride ions (1.59 µg), chlorite ions (7.96 µg), and chlorate ions (11.83 µg). Analyses of iron and copper controls identified only chloride ions, 14.58 and 31.78 µg, respectively.

The reaction of CD (6 h at ~ 2000 ppm) with aluminum yielded four metal salts: $AlCl_3$, $Al(ClO_2)_3$, $Al(ClO_3)_3$, and Al(ClO4)3. Decomposition at the lower concentration of chlorine dioxide (12 h at ~ 1000 ppm) produced only chloride, chlorite, and chlorate ions. The amounts of anions, except for chloride, were greater in the reaction with the higher concentration of CD.

The reaction of CD with iron gave three metal salts: $FeCl_3$, $Fe(ClO_3)_3$, and $Fe(ClO_4)_3$. Decomposition in 2000 ppmv (6 h) CD gave about 39% more chloride than at 1000 ppmv (12 h); however, the amounts of the other anions were similar between the two reactions.

Decomposition of CD on copper (12 h at 1000 ppm CD) yielded $CuCl_2$ (most abundant), $Cu(ClO_3)_2$, and $Cu(ClO_4)_2$ complexes. CD (6 h at 2000 ppmv) produced predominantly chloride and a relatively lower concentration of chlorate ions.

Table 13.1: Residual Anions on Metal Coupons After Exposure to Chlorine Dioxide

SAMPLE (TREATMENT TIME AND CONCENTRATION)	**AMOUNT ANALYTE PRESENT (µG)**			
	chloride (Cl^-)	**chlorite (ClO_2^-)**	**chlorate (ClO_3^-)**	**Perchlorate (ClO_4^-)**
Aluminum (control)	1.59	7.96	11.83	BDL*
Aluminum (6 h ~ 2000 ppmv)	933.5	31.48	133.9	45.48
Aluminum (12 h ~ 1000 ppmv)	1,314	19.72	15.76	BDL*
Iron (control)	14.58	BDL*	BDL*	BDL*
Iron (6 h ~ 2000 ppmv)	22,383	BDL*	26.52	39.46
Iron (12 h ~ 1000 ppmv)	16,062	BDL*	21.79	37.60
Copper (control)	31.78	BDL*	BDL*	BDL*
Copper (6 h ~ 2000 ppmv)	942.5	BDL*	40.59	BDL*
Copper (12 h ~ 1000 ppmv)	1,630	BDL*	76.78	37.46

*Below the detection limit (<0.4 µg)

Figure 13.1: Copper Tubing Prior to Exposure to Chlorine Dioxide

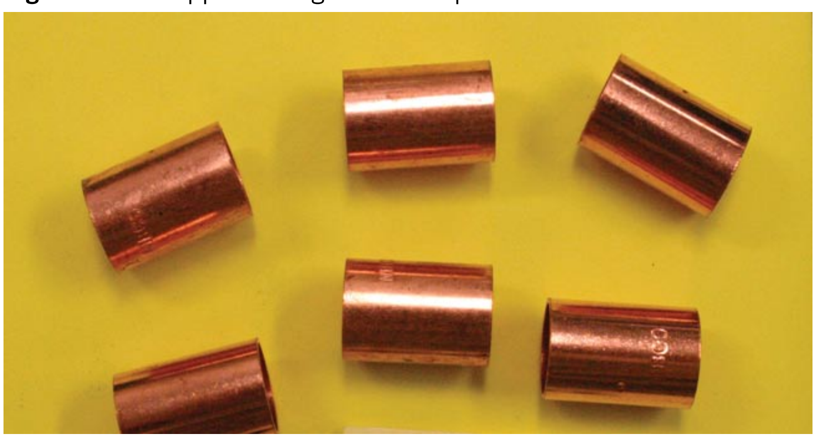
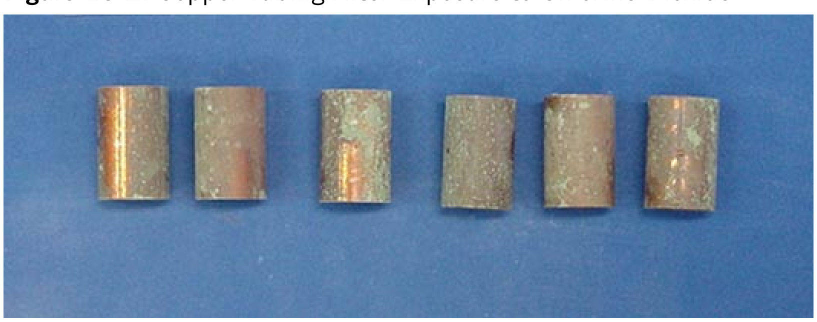


Figure 13.2: Copper Tubing After Exposure to Chlorine Dioxide



14 Quality Assurance Findings

Three technical audits of the Instron destructive testing process on CD-fumigated coupons were conducted over the course of the program. The first technical audit, conducted 6 June 2005, covered steel coupons from a control run in the CD chamber. A second technical audit, conducted on 21 September 2006, involved carpet samples fumigated with CD. A third technical audit, conducted on 22 September 2006, involved wood and gypsum wallboard samples fumigated with CD. All operations were conducted in accordance with the applicable SOPs and IOPs. Data quality audits were conducted on 8 of the 63 CD material compatibility tests (13%). All were found to be acceptable, in accordance with the Quality Assurance Project Plan.

Literature Cited

1. Brickhouse, M.D. Quality "Assurance Project Plan and Work Plan for Deposition Velocity Studies: Materials Sorption of Vaporized Hydrogen Peroxide or Chlorine Dioxide, Doc. No. DSQAPP2004DV," 2004.
2. "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.
3. "Quality Management Plan for Environmental Programs," Edgewood Chemical Biological Center Research, Development and Engineering Command, 2003.
4. "EPA Guidance for Data Quality Assessment, Practical Methods for Data Analysis, EPA QA/G-9," U.S. Environmental Protection Agency, 2000.
5. "EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5," U.S. Environmental Protection Agency, 2001.
6. "EPA Guidance for Quality Assurance Project Plans. EPA QA/G-5," U.S. Environmental Protection Agency, 2002.
7. "EPA Guidance on Environmental Data Verification and Data Validation, EPA QA/G-8," U.S. Environmental Protection Agency, 2002.
8. Brickhouse, M.D. "Quality Assurance Project Plan and Work Plan for Effects of Vaporized Decontamination Systems on Selected Building Interior Materials, Doc. No. DSQAPP2004MC," 2004.

Appendix A:
Coupon Identifier Code

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

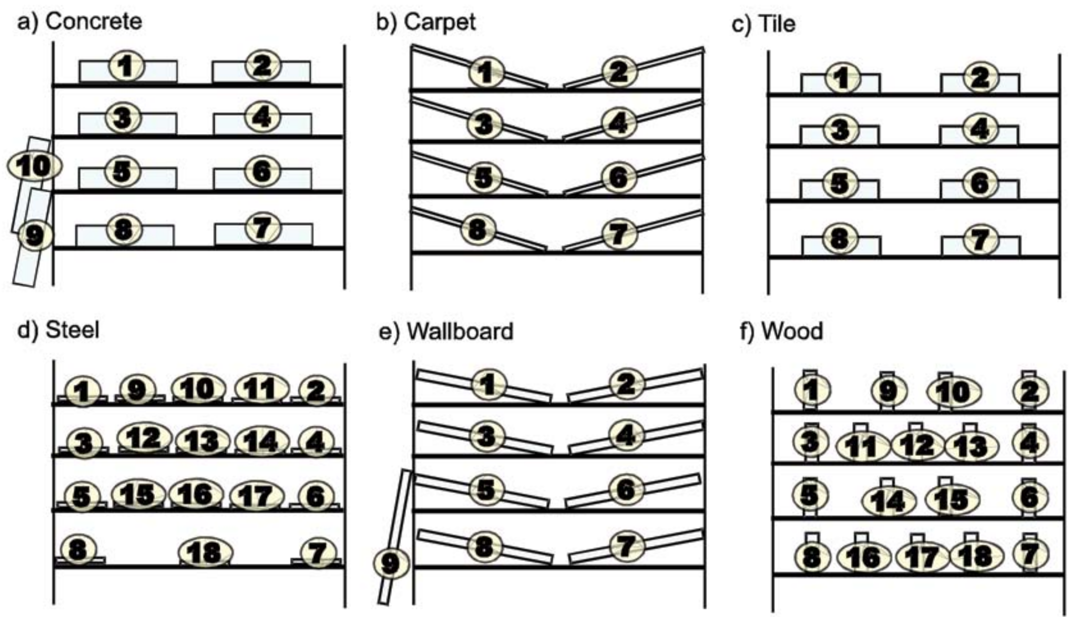
Code Pattern

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

Example GV4101104

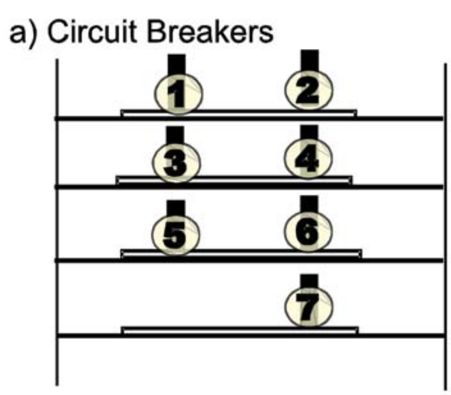
Gypsum Wallboard with test start date of October 11th, 2004, sample number 4.

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



Coupons shown on rack shelves from direction of glove box transfer chamber. Pictoral coupon scaling for length and width is (0.75 * 2 *(cm / 10)).

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



Coupons shown on rack shelves from direction of glove box transfer chamber.

Appendix B:
Detailed Coupon Preparation and Inspection Procedures

COUPON PREPARATION PROCEDURE

The coupon preparation, unless otherwise noted, was 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 in. x 1 ½ in. x ½ in.
- Preparation of Coupon:
  - The machined ends of the stock were discarded by removing > ¼ in. of the machined end. Coupons were cut from stock using a table saw equipped with an 80-tooth crosscut blade.

Latex-Painted Gypsum Wallboard

- Stock Item Description: ½ in. 4 ft. x 8 ft. Drywall
- Supplier/Source: Home Depot; Edgewood, Maryland
- Coupon Dimensions: 6 in. x 6 in. x ½ in.
- Preparation of Coupon:
  - The ASTM method requires that the samples be taken from the interior of the material rather than from the edge (machined edge). The machined edges of the stock were discarded by cutting away > 4 inches from each side.
  - Coupons were cut from stock using a table saw equipped with an 80-tooth crosscut blade.
  - The 6 in. x 6 in. coupons were painted with 1 mil of Glidden PVA Primer and followed by 1–2 mils of Glidden latex topcoat. The primed coupons were allowed to stand for > 24 hours prior to the application of the topcoat.
  - All six sides of the 6 in. x 6 in. ½ in. coupon were painted.

Concrete Cinder Block

- Stock Item Description: 8 in. x 16 in. x 1 ½ in. concrete cinder block cap
- Supplier/Source: York Supply; Aberdeen, Maryland
- Original Coupon Dimensions: 4 in. x 8 in. x 1 ½ in.
- Modified Coupon Dimensions: 4 in. x 8 in. x ½ in.
- Preparation of Coupon:
  - Coupons were cut from stock using a water-jet.
  - Four coupons were cut from each stock piece.
  - Original dimensions, too large for material testing.
    - o Each coupon was cut into three sections.
    - o Two sections were measured at modified coupon dimensions
    - o The third section was discarded.

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 were cut from the stock using a utility knife.
  - The longer direction (8 in.) was cut parallel to the machine edge.
  - The machined edge was discarded by removing > ½ in.

Painted Structural Steel

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

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

Ceiling Suspension Tile

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

COUPON INSPECTION PROCEDURE

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

Mechanically Graded Lumber (Bare Wood)	10 in. ± 1/16 in. x 1.5 in. ± 1/16 in. x 0.5 in. ± 1/32 in.
Latex-Painted Gypsum Wallboard	6 in. ± 1/16 in. x 6 in. ± 1/16 in. x 0.5 in. ± 1/16 in.
Concrete Cinder Block	4 in. ± ½ in. x 8 in. ± ½ in. x 0.5 in. ± 1/16 in.
Carpet	6 in. ± 1/8 in. x 8 in. ± 1/8 in.
Painted Structural Steel	1/4 in. ± 1/128 in. x 12 in. ± 1/16 in. with 2 in. ± 1/16 in. wide at ends, ¾ in. ± 1/16 in. wide at center
Ceiling Suspension Tile	12 in. ± 1/8 in. x 3 in. ± 1/16 in. x 9/16 in. ± 1/16 in.

Appendix C:
Wood Coupon Location of Break

The ASTM test method requires reporting the location of the break for each wood sample. The purpose of this appendix is to provide this information in pictorial form. Yellow arrows are used on samples where the photograph contrast may not clearly show the location of the break.

Figure C-1: Location of Break, Wood Coupons – Chlorine Dioxide Control Set

Figure C-2: Location of Break, Wood Coupons – Chlorine Dioxide 1000-ppm Set

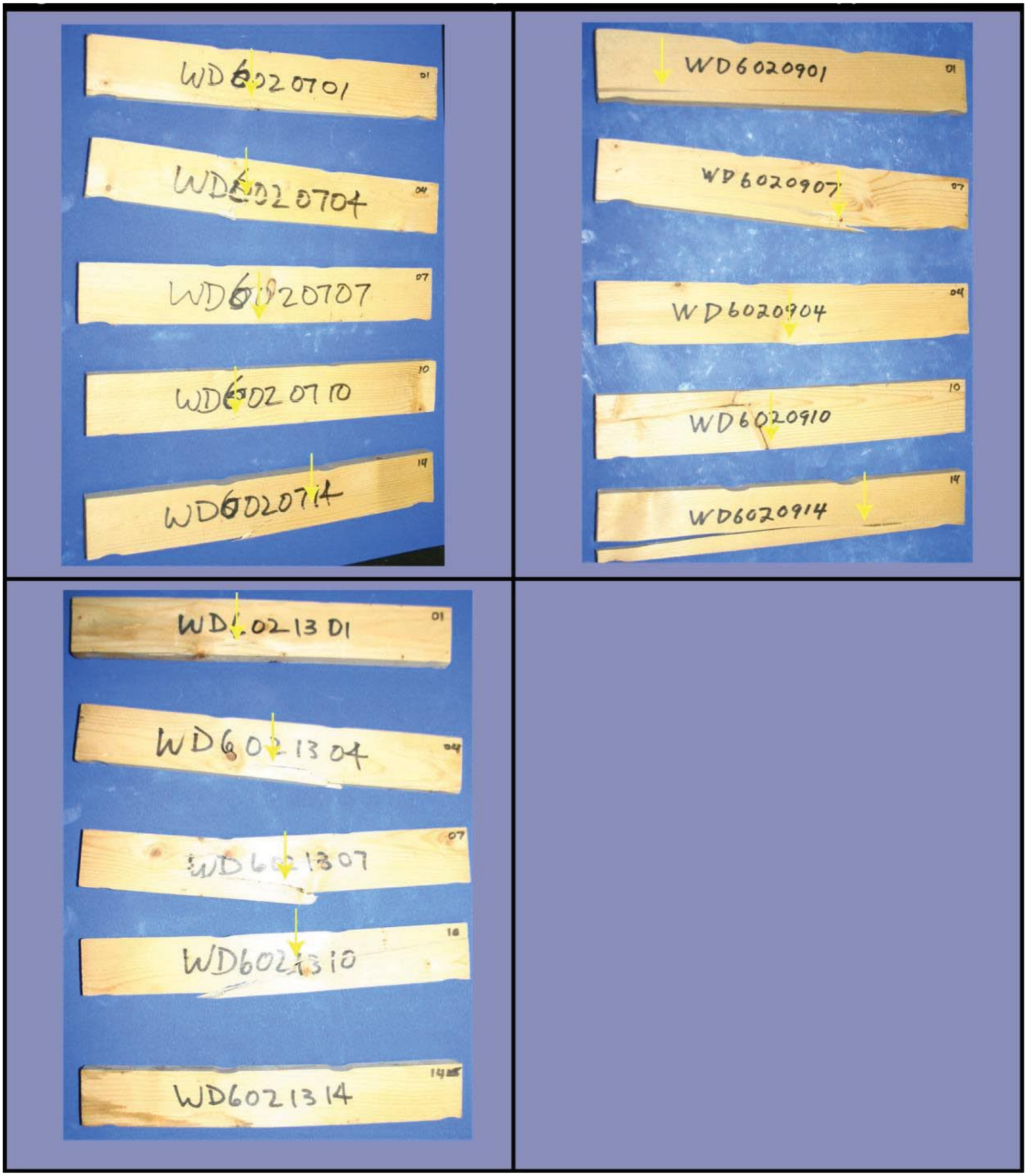


Figure C-3: Location of Break, Wood Coupons – Chlorine Dioxide 2000-ppm Set

Appendix D:
Concrete Cinder Block Coupon Location of Break

Figure D-1: Location of Break, Block Coupons – CD Control Set

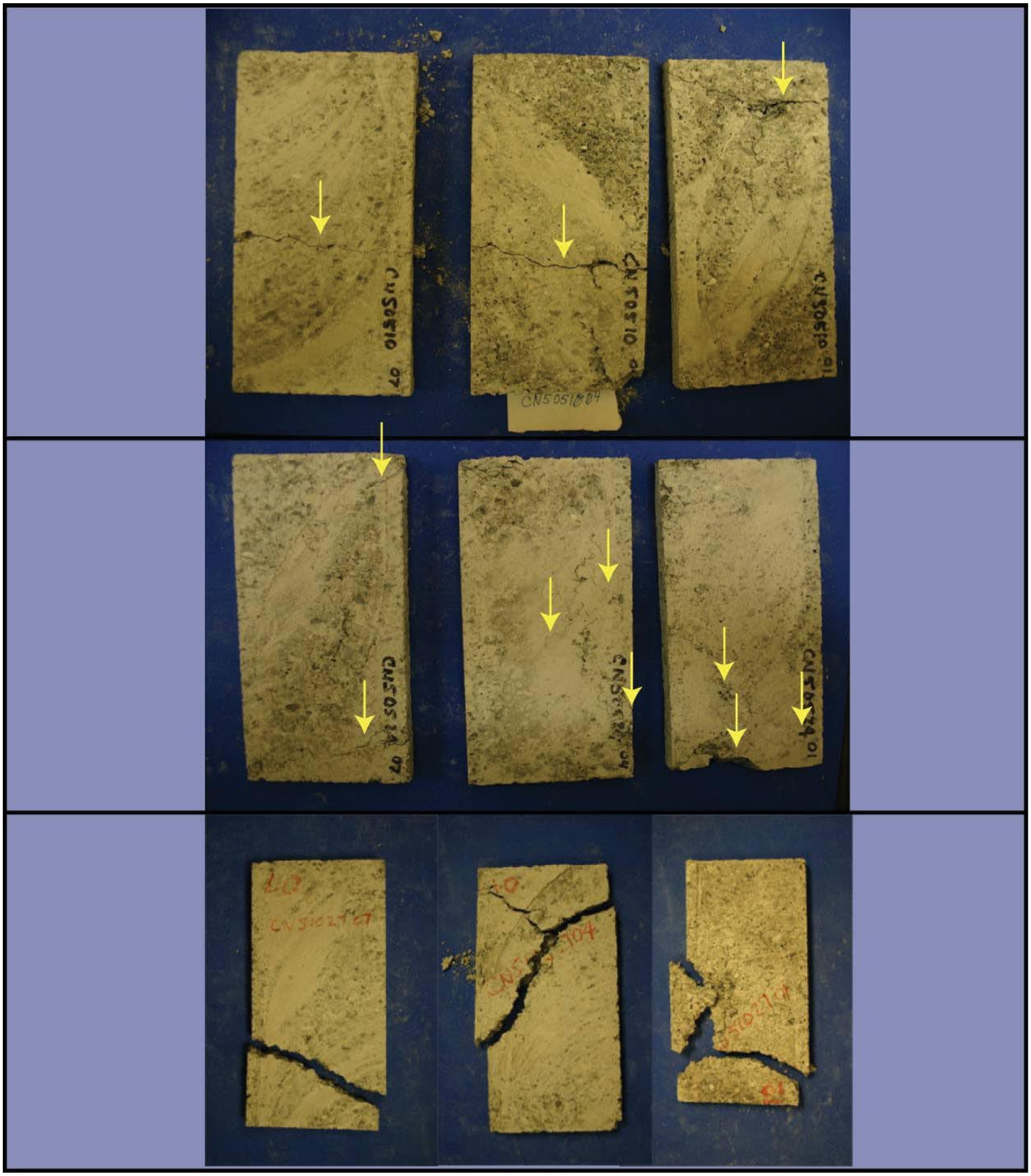


Figure D-2: Location of Break, Block Coupons – CD 1000-ppm Set

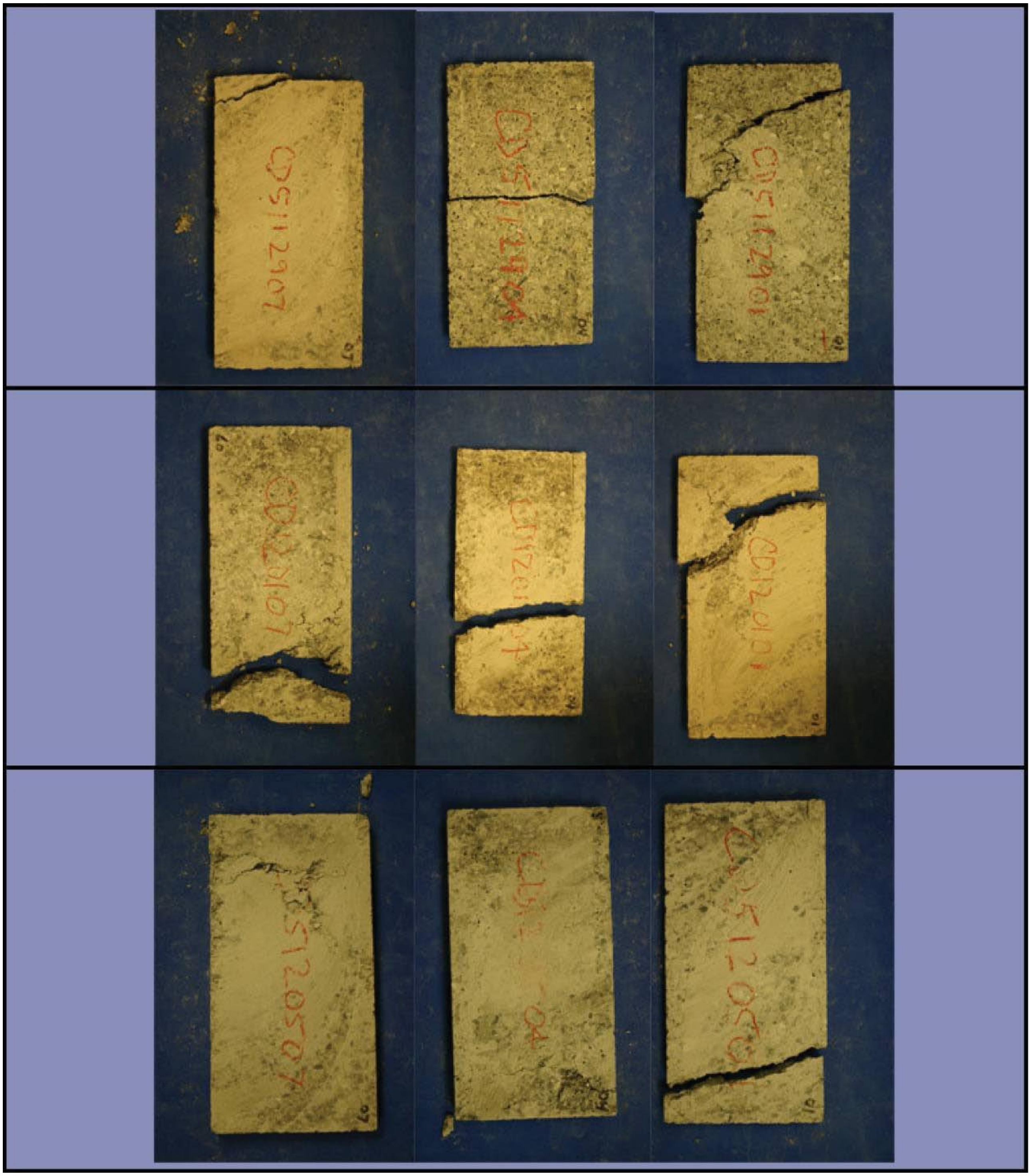
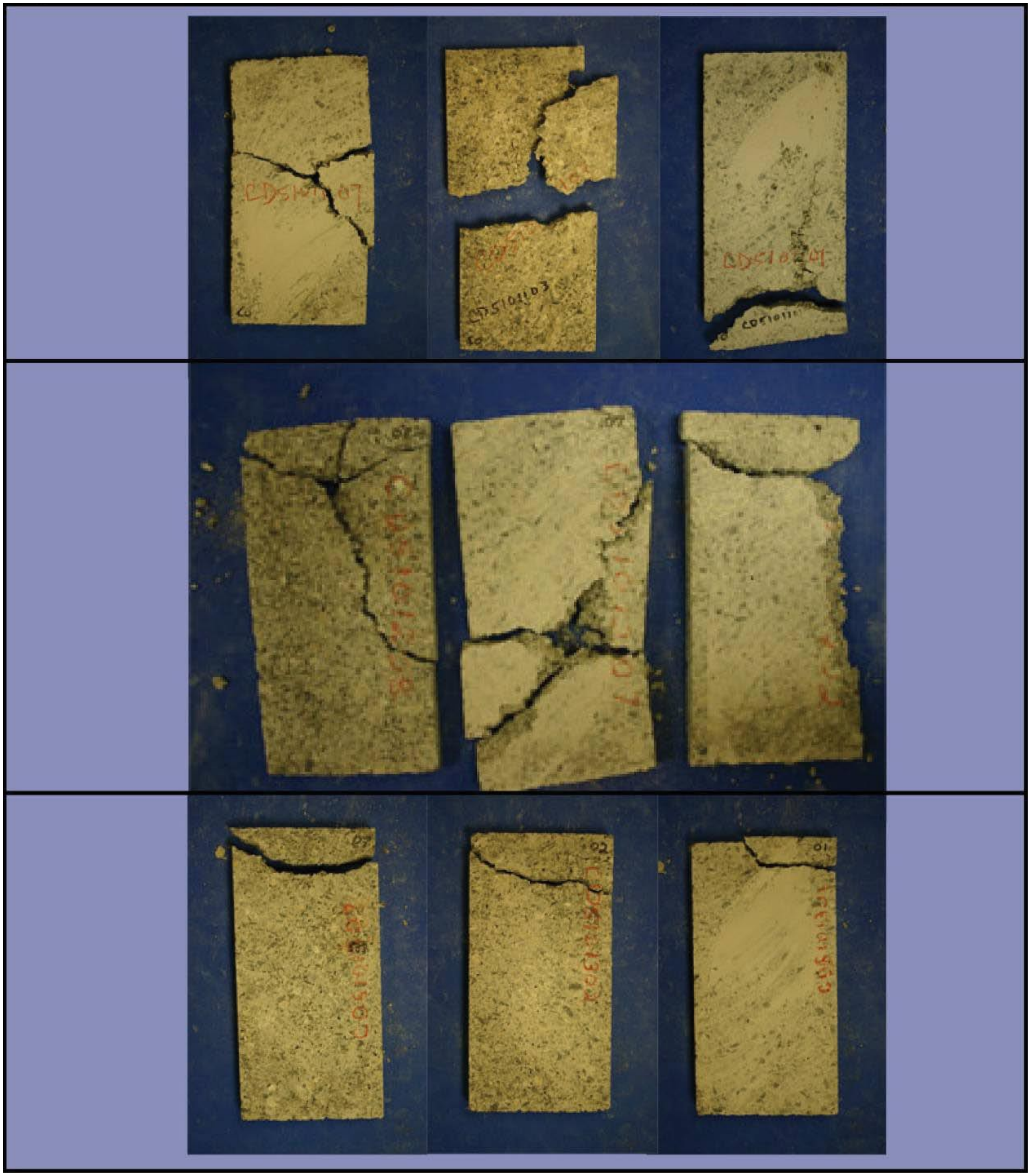


Figure D-3: Location of Break, Block Coupons – CD 2000-ppm Set



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