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**Evaluation of the Effectiveness of  
Coatings in Reducing Dislodgeable  
Arsenic, Chromium, and Copper  
from CCA Treated Wood**

Draft Final Report

18 October 2006



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## List of Acronyms

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As	Arsenic
Av	Average
AWPA	American Wood Preservers' Association
BL	Baseline
CCA	Chromated Copper Arsenate
CD-Rom	Compact Disk – Read Only Memory
CL	Confidence Limits
CLP	Contract Laboratory Program
CPSC	Consumer Product Safety Commission
DCCA	Dislodgeable CCA Wood Analytes
DAs	Dislodgeable Arsenic
DCr	Dislodgeable Chromium
DCu	Dislodgeable Copper
DI	Deionized Water
DPD	Dew Point Depression
DQI	Data Quality Indicator
EPA	United States Environmental Protection Agency
ERC	Environmental Research Center (old building)
FIFRA	Federal Insecticide Fungicide and Rodenticide Act
H&S	Health and Safety
HF	Hydrofluoric Acid
ICP	Inductively Coupled Plasma
ICP-MS	Inductively Coupled Plasma—Mass Spectrometry
ID	Identification
Ln	Natural Logarithm
Mph	Miles Per Hour
MS	Matrix Spikes
MS/MSD	Matrix Spikes and Matrix Spike Duplicates
MSD	Matrix Spike Duplicates
MSDs	Mean of the Squared Differences
NCDC	National Climate Data Center
NE	Northeast

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NFG	National Functional Guidelines
NOAA	National Oceanic and Atmospheric Administration
NOPW	Number of Previous Wipes
NW	Northwest
NWS	National Weather Station
OLS	On-Site Laboratory Support
OPP	Office of Pesticide Programs
Pcf	Pounds per Cubic Foot
PEA	Performance Evaluation Audit
PFA	Perfluoroalkoxy
PM	Project Manager
PSA	Primary Sampling Area
PTFE	Polytetrafluoroethylene
QA/QC	Quality Assurance and Quality Control
QAM	QA Manager
QAO	Quality Assurance and Quality Control (QA/QC) Officer
QAPP	Quality Assurance Project Plan
RDU	Raleigh—Durham International Airport
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
RSD/RPD	Relative Standard Deviation or Relative Percent Deviation
RTP	Research Triangle Park
SAP	Scientific Advisory Panel
Std. Dev.	Standard Deviation
STL	Severn Trent Laboratory
SYP	Southern Yellow Pine
TFE	Tetrafluoroethylene
TSPW	Time (Months) Since the Previous Wipe
U.S.	United States
WA	Work Assignment
WAL	Work Assignment Leader
WAM	Work Assignment Manager
UV	Ultraviolet

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**Evaluation of the Effectiveness  
of Coatings in Reducing  
Dislodgeable Arsenic,  
Chromium, and Copper from  
CCA Treated Wood**

October 2006

## **Executive Summary**

EPA conducted a study to evaluate the effect of coatings on dislodgeable arsenic, chromium, and copper residues on the surfaces of chromated copper arsenate (CCA) treated wood. Dislodgeable CCA (DCCA), determined by wipe sampling the wood surfaces, was the primary evaluation criterion for the coatings tested in this study, due to the potential for ingestion of CCA chemicals by hand-to-mouth activities of young children that contact CCA treated wood. The study was conducted by EPA's Office of Research and Development in Research Triangle Park, North Carolina, in support of the Office of Pesticide Programs (OPP) and in collaboration with the staff of the Consumer Product Safety Commission (CPSC), who used a similar protocol to evaluate several of the same products at a site in Gaithersburg, Maryland.

EPA risk assessors, the coatings and wood treating industries, and citizens who may be concerned about contact with CCA treated wood need sound information regarding the impact of coatings on dislodgeable arsenic residues because, although no longer produced or available for most consumer residential uses, CCA has been used extensively for construction of decks and play structures that may have a long service life and therefore, may pose a potential exposure route for CCA residues for many years to come.

Two sources of weathered CCA-treated southern yellow pine (SYP) were harvested from in-service decks and used to construct a series of miniature decks (minidecks), onto which one of twelve coatings was applied per manufacturer's instructions. Products included oil and water-based sealants and stains, two porch and deck paints, and two products that were advertised to encapsulate CCA treated wood. Instructions for ten of the products recommended surface preparation with a cleaning agent followed by a rinse prior to application of the coating, instructions for one product recommended only a water rinse, and instructions for one product recommended neither cleaning nor rinsing prior to application. Instructions for six of the products recommended two applications of the product. Each minideck contained four CCA-treated boards: two from a relatively old source deck and two from a relatively new source deck. Each coating was applied to three minidecks per manufacturer's instructions. There were also three positive control (CCA treated, uncoated) minidecks and one negative control (untreated, uncoated) minideck for a total of 40 minidecks. After coating, the minidecks were subjected to outdoor weathering at a controlled site in North Carolina where climate measurements were recorded on a near-continuous basis.

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Figure E-1. Minideck Test Site at EPA's Research Triangle Park, North Carolina, Campus

Dislodgeable arsenic (DAs), chromium (DCr), and copper (DCu) were determined at specified intervals: precoat (baseline), and after 1, 3, 7, 11, 15, 20 and 24 months. The DCCA residues were determined by wiping deck surfaces with a polyester wipe attached to a 1.1 kg weight that slides between rails of a wipe apparatus, developed by the staff of the CPSC. The arsenic, chromium and copper were extracted from the wipe in a nitric acid solution and the total amount of each metal in the extract was determined by inductively coupled plasma-mass spectrometry (ICP-MS). Baseline samples were taken before preparing the minideck surfaces for coating by mild pressure washing. Thus, the baseline values provide insight into the impact of rinsing CCA-treated decking. The statistical analysis used to evaluate coating performance compares dislodgeable CCA residues determined on the uncoated CCA controls with CCA residues determined for the coated minidecks at each time period. For completeness, the data were analyzed with and without inclusion of baseline values as a covariate in the model.

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**Figure E-2. Sampling to Determine Dislodgeable Arsenic (DAs) Residues on CCA-Treated Wood Using the CPSC Wipe Sampling Apparatus**

The amount of DAs versus time for each board on each minideck exhibited some immediately recognizable trends: Each coating, as well as the positive controls (uncoated CCA-treated minidecks), showed a significant decrease in DAs between baseline (prerinse and precoat) and samples taken 1 month after coating. The rinsing preparation step for the uncoated CCA controls initially reduced the DAs by about 75 percent. Thus, the rinse step provides some reduction in DAs; however, the reduction is relatively small compared to the reduction attributable to the coatings. The results indicate that coating (using any of the coatings tested) mitigates DAs to some degree, although not always at a statistically significant level. Given sufficient time, DAs goes up after coating as boards weather. However, the trend over time may appear uneven, because the amount determined at any wipe sampling event is an imprecise snapshot in time of processes that increase and processes that decrease the amount of dislodgeable residues—wipe sampling and precipitation remove residues whereas weathering and time increase residues. The log-scale plots, Figures E3 and E4 below,

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illustrate changes in DAs with time for two minidecks, deck 8C, coated with a water-based stain, and Deck 13A, an uncoated CCA-treated positive control deck. Lines above the data in each plot indicate the baseline DAs determined prior to rinsing and coating. The colors are keyed to individual boards, source deck, A (older) or C (newer), and end grain orientation, up or down. The difference between the lines at the top of the graph and the time period 1 data point in Figure E3 demonstrates the impact of rinsing and coating whereas the difference in Figure E4 is due to the impact of rinsing the uncoated CCA-treated control deck.

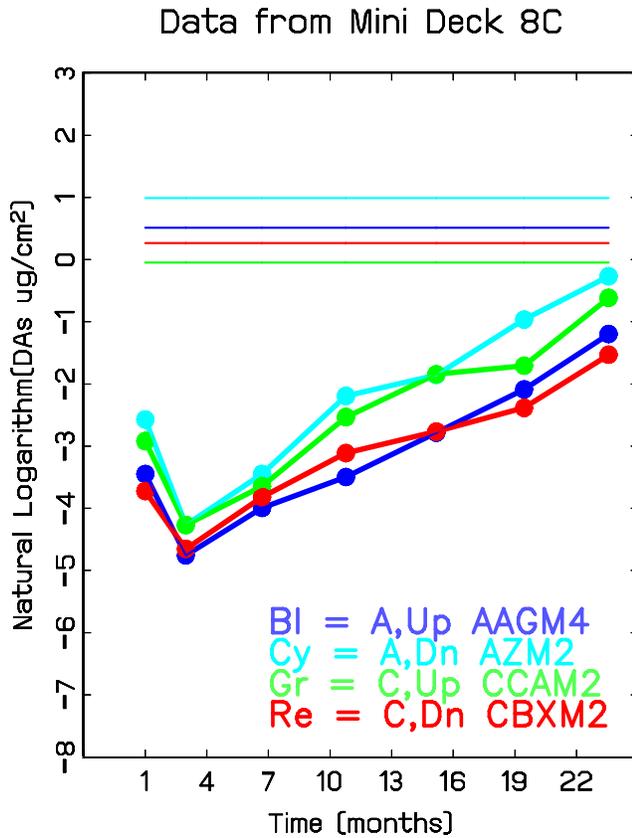


Figure E-3. DAs vs. Time Determined on Four Boards of Minideck 8C, Coated with a Water-based Stain

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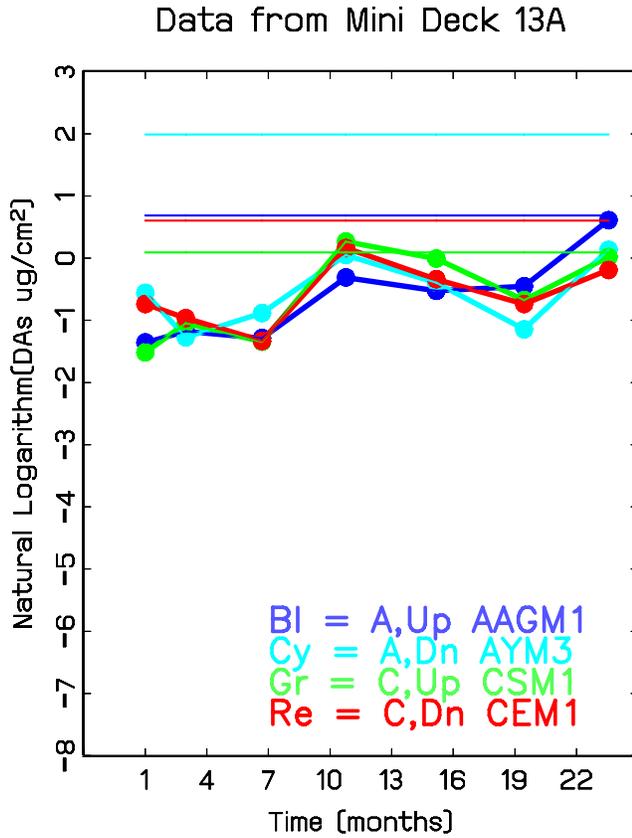


Figure E-4. DAs vs. Time Determined on Four Boards of One of the Positive Control Minidecks (no coating applied)

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The key findings of the study are captured in the pair-wise analysis of difference between each coating and the uncoated CCA treated minidecks at each of the seven sampling events over the two-year study. The pair-wise analyses demonstrate that (1) all of the coatings tested reduced DCCA, however, the efficacy of each product decreased over the period of the study, and by 24 months only two of the products, the paints, had DAs levels that were significantly less than the positive controls (p-value<sup>1</sup> < 0.05), and (2) the reductions of DAs for one product, a water-based sealant, were not statistically different from the positive controls over the entire course of the study (p-value ranged from 0.7966 to 1).

Exploratory statistical tests that investigated differences among identifiable subgroups of products tested in this study (as determined from information on the label and information found in material data safety sheets obtained for each product) indicate that, in terms of DAs reduction: (1) stains performed better than sealants (p-value = 0.0007), (2) products with acrylic performed better than products without acrylic (p-value = 0.0018), (3) non-alkyd products performed better than products with alkyd (p-value = 0.0002), (4) film-forming products performed better than penetrating products (p-value < 0.0001), and (5) multi-coat products performed better than single-coat products (p-value < 0.0001). The difference between products with clear or semi-transparent cover was not significant (p-value = 0.5579) and the difference between oil and water-based products was not significant at the 0.05 level (p-value = 0.0885) when paints are excluded from the analysis. These results should be interpreted in light of the fact the study was not designed to make such comparisons and thus the number of representatives from each coating subgroup was limited; furthermore because the coatings often contain several ingredients, the results for coating groups tested in our study may not apply to similarly defined subgroups of other untested coatings, because such coatings could contain a different mix of ingredients. Additionally, these subgroups were often overlapping, therefore the statistical tests are not independent and the conclusions are somewhat redundant. Our results are suggestive that substantial differences in performance among subgroups of coatings exists, and thus their greatest value may be to guide future research. Because only testing of a broader selection of products with more representatives within product classifications,

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<sup>1</sup>A p-value is a measure of the amount of statistical evidence supporting an alternative hypothesis relative to a particular null hypothesis, with smaller p-values corresponding to greater evidence. The null hypothesis is usually the hypothesis of "no difference" or "no effect"—no difference in mean DAs between Coating 1 and the Coating 13 at time period 1. Whereas the alternative hypothesis asserts that a difference (or effect) exists—Coating 1 and Coating 13 have different mean DAs at time period 1. P-values always lie between 0 and 1 and thus are probabilities. In fact, the p-value is the probability of finding a difference (or an effect) as large as the one observed in the data, when the null hypothesis is true. A p-value less than 0.05 is generally considered to be "statistically significant" although the choice of 0.05 is somewhat arbitrary and sometimes other cutoff values—0.1 or 0.01, are used to classify finding as statistically significant or not.

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supported by analyses that clearly define product constituents, will be able to convincingly reveal differences with respect to product classification, constituents, and performance.

Although the products for which application of more than one coat was recommended (and applied) performed significantly better than the products for which a single coat was recommended, one cannot infer that multiple coats necessarily provide improved reduction of DAs because the study design did not examine this question directly by testing multiple application levels for all products. Also, the lack of statistical significance between oil-based and water-based products does not imply that they perform equivalently; rather, only that differences in performance, if they exist, were not great enough to be detected in this experiment. However, a water-base stain was one of the two most effective non-film-forming products tested. This observation is of interest because the availability of oil-based products will likely decrease after 2006 in response to implementation of the Clean Air Act amendments restricting the atmospheric release of volatile organic compounds from architectural coatings.

Taken as a whole, the results of this study suggest that after the first application, typical deck coatings (sealants and stains) need be re-applied periodically in order to maintain significant levels of DAs mitigation compared to an uncoated board. For the conditions experienced in this study, which notably did not include a significant abrasion component, it appears that a one-year recoat schedule would be appropriate; however, this could be different under different conditions, including in a different geographic area. The differences between efficacy of products suggests a need for broad-based testing to enable consumers to make informed choices with regard to efficacy of DAs reduction by deck sealants and coatings. Note that elevated levels of arsenic were determined in peeling paint chips (and possibly attached wood fibers) recovered from the weathered painted decks indicating a need to characterize the potential exposures due to preparation steps, particularly for film forming products.

Additionally it was found that there were significant differences in dislodgeable arsenic, pre and post-coat, between the two source decks (p-value with baseline covariate < 0.0001) with Source A DAs greater than Source C DAs. Thus, source deck is an important variable in this type of test. Grain orientation, up or down, was not a significant variable (p-value of 0.416) in this experiment. Dislodgeable arsenic concentrations on the cross-contamination control boards (untreated boards between each CCA-treated board) averaged about 2 to 8 percent of the averages for each minideck at each sampling interval, implying measurable but limited movement of dislodgeable arsenic to adjacent surfaces.

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As with any study that endeavors to address a complex question, there are limitations that impact the extent to which the results may be generalized. The principle limitations of this study are that a limited number of products were tested on two sources of wood in one climatic region, in the absence of the normal stresses that a full-scale, in-use deck may experience. Evaluation of a broader range of coatings, evaluation of the impact of surface preparation steps, evaluation of periodic reapplication, and testing under conditions of normal wear and tear in representative climatic regions is needed to determine how broadly the results of this study may be applied. Note that except for the two products designed to encapsulate CCA treated wood, these tests evaluated the ability of consumer products to perform a function for which they are neither designed nor marketed.

This report supersedes the previously issued "Interim Data Report." In addition to covering the complete dataset through 24 months of testing, this final report also utilizes a more sophisticated data analysis model. The draft project plan (QAPP) was posted for public and stakeholder comment and the QAPP as well as the Interim Data Report were the subjects of letter peer review. This draft report is posted for public and stakeholder comment. Comments received on this draft will be included as part of the background materials for an open peer review by OPPTS's Scientific Advisory Panel, in the fall of 2006. Records of the letter peer reviews and the supporting documents for the upcoming peer panel review can be found in EPA's Science Inventory ([www.epa.gov/si/](http://www.epa.gov/si/)) at Record 150970.

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## 1. Project Description

### 1.1 Background

CCA is a wood preservative that is impregnated under pressure into wood to protect it from decay and insect damage. CCA is registered under the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA) by EPA's Office of Pesticide Programs (OPP). In October 2001, EPA-OPP prepared a preliminary deterministic exposure assessment for selective internal and external peer review comments as an interim report intended to address child residential "playground" exposures. In addition, EPA requested guidance from the FIFRA Scientific Advisory Panel (SAP) for risk mitigation measures such as sealants and coating processes. The SAP Panel made "recommendations regarding the need for additional studies in this area..." because the "weight-of-evidence from available studies indicates that certain coatings can substantially reduce dislodgeable and leachable CCA chemicals." The Panel also recommended that "EPA inform the public of the ability of certain coatings to substantially reduce leachable and dislodgeable CCA chemicals..."

In March 2003, the registrants of CCA wood preservatives signed an agreement with EPA for voluntary cancellation of CCA-treated wood for residential uses (such as playsets and decks) effective beginning January 1, 2004. However, existing decks and playsets made of CCA-treated wood will still be in use. Therefore, the potential remains for dermal contact with arsenic, chromium, and copper residues on treated surfaces, , , and this may be a concern for infants and small children, due to their close contact with surfaces and hand-to-mouth activities (see Kwon, 2004, Ursitti, 2004, Zartarian, 2006).

In support of OPP's need for information, the Office of Research and Development (ORD) conducted a study that evaluates the ability of selected coatings to reduce the amount of dislodgeable CCA (DCCA) chemicals on the surfaces of CCA-treated wood. The Consumer Product Safety Commission (CPSC) was a collaborator on this project via an interagency agreement (CPSC-I-03-1235) between the United States Environmental Protection Agency (EPA) and CPSC. The test data will support EPA and CPSC efforts to inform the public regarding the use and maintenance of existing CCA-treated wood products, such as decks and playground equipment.

In previous studies, the CPSC staff determined the relationship between amount of arsenic transferred to a hand by contact with CCA-treated wood and the amount determined by the CPSC wipe technique (See Thomas, T. et al., 2004, Levenson, M. et al., 2004, and Hatlelid, K. et al., 2004). EPA modified the CPSC wipe technique in efforts to increase sensitivity. Appendix D reports experiments conducted by EPA and

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CPSC staff to determine the relationship between dislodged CCA residues determined using each approach. Although calculation of intake is considered beyond the scope of this coatings study, the wipe comparison experiments established a relationship between the two surrogate wipe methods. However, further analysis or experimentation may be needed to reduce uncertainty and/or estimate potential intake due to contact based upon the data presented in this report.

This report supersedes the previously issued "Interim Data Report" (U.S. EPA 2005). In addition to covering the complete dataset through 2 years of testing, this final report also utilizes a more sophisticated data analysis model. This report covers all baseline measurements taken, wood preparation and coating application data, and mitigation sampling at 1, 3, 7, 11, 15, 20 and 24 months after coating application, in addition to other supporting data collected as part of the study. The draft QAPP and Interim Data Report were externally peer-reviewed through letter review and public input was received for the draft QAPP and the revised version of the Interim Data Report which was posted after letter peer review. This draft report will be posted for public comment and submitted for peer panel review through OPPTS's Science Advisory Panel in the fall of 2006.

## **1.2 Project Objectives**

The objective of this project was to evaluate the ability of typical deck coating products to reduce dislodgeable CCA chemicals on pressure treated wood.

Environmental variables that impact coating performance include ultraviolet (UV) radiation, condensation, precipitation, and thermal shock. Efficacy of coatings may also be impacted by the level and fixation of CCA treatment, age and condition of the wood at the time of coating, and type and dimensions of the treated wood, among other variables. Due to the large number of variables, and EPA's desire to provide guidance quickly for in-service wood, the scope of this evaluation was limited to selected coatings applied to aged CCA-treated southern yellow pine (SYP) wood exposed to natural outdoor weathering at a site in North Carolina.

## **1.3 Experimental Design**

Two sources of weathered CCA-treated southern yellow pine (SYP) were harvested from in-service decks and used to construct a series of miniature decks (minidecks), onto which selected coatings were applied. Dislodgeable arsenic (DAs), chromium (DCr), and copper (DCu) were measured at specified intervals. Weathering tests were conducted by exposing minidecks to natural outdoor conditions at a site in Research

Triangle Park, North Carolina. The ability of the coatings to reduce DCCA (dislodgeable wood analytes) as the wood and coatings weather was evaluated by periodically determining the amount of DCCA removed from the surface of the wood specimens using a specific wipe technique.

For the purposes of this study, DCCA was defined as the amount of CCA analyte removed from the surface of the test specimen using a dermal wipe procedure initially developed and demonstrated by the staff of the CPSC. This method was slightly modified for the EPA study. The different wipe sampling methods and all modifications implemented by EPA at each stage of the project are fully described in Section 2.8.3 of this report. A side study was conducted to determine the comparability of the two methods and the results of that study are presented in Appendix D.

Wood nomenclature used in this report is defined in Figure 1-1. A “board” is defined as the unit of wood removed from an existing structure (the “source”), while “specimen” refers to the pieces of each board cut for this project. (Specimens are sometimes called coupons in weathering testing jargon). Furthermore, areas on specimens that were wipe-sampled during each sampling interval are termed “primary sampling areas” (PSA), in contrast to adjacent areas which were not sampled at each interval. Each specimen used in this project contained one PSA and one adjacent area. The PSAs were wipe-sampled during each sampling event (i.e., at 1, 3, 7, 11, 15, 20 and 24 months after coating). Areas on the minidecks adjacent to the PSAs were termed “baseline areas” (BL) because they were used to calculate specimen-specific baseline DCCA values, even though these baseline DCCA values were not ultimately used in the statistical model used to analyze the time-series data (refer to Section 2.8.2.1 for further explanation).

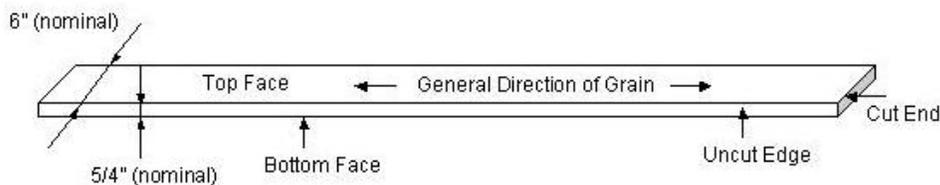


Figure 1-1. Wood Nomenclature

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All sampling was done on the top faces of the boards. A “grain-up” or “bark side up” board is defined as one where the tree growth rings, evident on the cut end of the board, form a convex pattern (a “hill”) when observed with the face of the board that was exposed on the source deck facing up. Likewise, a “grain-down” or “bark side down” board is defined as one where these rings form a concave (a “valley”) pattern when the exposed face is facing up. Because wood tends to deform along these ring lines, grain orientation may be an important variable in the measurement and mitigation of DCCA on surfaces of CCA-treated wood. Grain-down boards tend to deform in a manner which “cups” and holds water and moisture, while grain-up boards tend to deform in a manner which sheds water from the surface of the board.

Each minideck had nine decking specimens: two specimens from each of the aged wood sources (one specimen with bark side up grain orientation and one with bark side down orientation), separated by specimens of new untreated wood (all positioned bark side up) to prevent cross-contamination and to serve as blank controls to assess cross-contamination potential as a result of splash-over of rain water, for example. The minidecks were constructed with each of the aged wood specimens having the same top face as the specimen had during its exposure on its source structure. Prior to coating the minidecks, baseline DCCA concentrations were determined by averaging the results of wipe samples from areas adjacent to the PSAs. A total of twelve different coatings were applied to three minidecks each. Additionally, three identical, but uncoated minidecks and one untreated, uncoated minideck were included as controls. The minidecks were prepared for coating according to the specific coating manufacturer’s recommendations. Coating was then applied to each minideck per coating manufacturer’s instructions. After allowing the coatings to dry and cure, the minidecks were subjected to natural weathering for a period of two years. During the two-year weathering period, at specified intervals (1 month, 3 months, 7 months, 11 months, 15 months, 20 months and 24 months after coating application) each specimen was wipe-sampled for DCCA and the results from each sampling event were statistically compared with corresponding DCCA results from uncoated control minidecks. Supporting samples included wood core samples, liquid samples of the coatings applied and paint chip samples.

The experiment was laid out as a split-split-plot design. In the language of split-plot designs: each minideck was a whole plot, and the whole-plot treatment was coating; the split-plot treatment was determined by the two-by-two factorial arrangement of the combinations of source decks (A, C) and grain orientation (up, down); each board constituted a split-split-plot and time was the split-split-plot treatment.

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The coatings were randomized to minidecks with the only restriction being that neighboring minidecks were not assigned the same coatings. In particular, the replication was not blocked, and this is the primary difference between the experiment and a typical split-split-plot. Two versions of the standard split-split-plot analysis-of-variance model were used to analyze the data, with and without baseline covariate adjustment.

This split-split-plot models allow for correlations among measurements from the same minideck, and also for correlations among the repeated time measurements made from the same specimen within a minideck. The data were analyzed with SAS's Proc Mixed with model statement,

model Y = coat

```

bbf sdeck bbf*sdeck coat*bbf coat*sdeck coat*bbf*sdeck
time coat*time bbf*time sdeck*time bbf*sdeck*time
coat*bbf*time coat*sdeck*time coat*bbf*sdeck*time (X);
random mdeck(coat1) mdeck(coat1*bbf*sdeck);

```

where:

Y = natural logarithm of DAs in  $\mu\text{g}/\text{cm}^2$  (DCr, DCu)

coat = coating (1...13) 13=no coating

bbf = board bark face (grain orientation; up, down)

sdeck = source deck (A, C)

time = time period (not continuous)

X = natural logarithm of the baseline measurement (included for one analysis, excluded for the second)

#### 1.4 Data Quality Objectives

The critical measurements for the natural weathering tests are total arsenic, and to a lesser extent, total chromium, and total copper concentrations, which are subsequently converted to dislodgeable arsenic, chromium, and copper, reported on a mass per unit area basis ( $\mu\text{g}/\text{cm}^2$ ). Data quality indicator (DQI) goals for concentration in terms of accuracy, precision, and completeness, as established in the QAPP for this project, are shown in Table 1-1. The method detection limit (MDL) for the analytical method is also shown. The 0.1  $\mu\text{g}/\text{L}$  MDL corresponds to 0.000032  $\mu\text{g}/\text{cm}^2$  DAs. An assessment of accuracy, precision and completeness goals is presented in Section 4.

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**Table 1-1. Data Quality Indicator Goals for Critical Measurements**

Analyte	Method	Accuracy (Percent Recovery)	Precision (Percent RSD/RPD)	Completeness (Percent)	Method Detection Limit (ug/L)
Arsenic (total)	SW-846 Method 6020 (modified)	90-110	10	90	0.1
Chromium (total)	SW-846 Method 6020 (modified)	90-110	10	90	0.1
Copper (total)	SW-846 Method 6020 (modified)	90-110	10	90	0.1

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## 2. Sampling and Analysis Protocol

Many protocol elements were considered in detail because no standardized protocol existed to evaluate the ability of coatings to reduce DCCA from CCA-treated wood.

### 2.1 Selection of Wood Sources

Criteria established to select from candidate aged wood source structures included: age and condition of deck with sufficient deck material of grain up and down treated to 0.40 pound per cubic foot (pcf) with Ground Contact CCA-C to meet project needs. First, sources needed to be of the Southern yellow pine species, as it is the most widely-used CCA treated wood species in the U.S. According to the Southern Pine Council (SPC 2006), SYP is preferred “because of its ease of treatability.” The unique cellular structure of Southern pine permits deep, uniform penetration of preservatives, rendering the wood useless as a food source for fungi, termites and micro-organisms. Some 85 percent of all pressure-treated wood is Southern pine.” In 2004, the SPC estimated that 76 percent of all CCA treated lumber and timbers were SYP with the next most common species group being the Hemlock-Fir group at 9 percent (Lebow 2006, personal communication). Regional differences are likely, with SYP overwhelmingly predominant in the Southeast U.S., a greater percentage of hemlock-fir, Douglas-fir and spruce-pine-fir on the west coast, and red pine and ponderosa pine also being used in the north and mid-west (Lebow 2006, personal communication).

Age, general condition and treatment history were additional source deck selection criteria as it was recognized that these variables might impact coating performance. Given the almost infinite combinations of age, treatment history, and condition, and the need for a relatively large amount of material, two locally available in-service decks were selected for testing from approximately a dozen candidate decks that were offered for use in the project. Details of the selected decks are provided below.

Environmental Research Center (ERC) Deck: This structure was located outside at EPA’s old (leased) Research Triangle Park facility. It was a stand-alone deck with generally full weather exposure and only moderate shading by adjacent buildings during low sun positions. Given its open, stand-alone nature, abrasion patterns appeared very consistent and the boards were visually similar to one another. Additional information on this source was gathered as it was being dismantled. The deck was constructed of SYP, treated to 0.40 pound per cubic foot (pcf) with Ground Contact CCA-C. This source was approximately 7 years old and was believed to have received one application of a standard deck sealant near the beginning of its use (over 5 years ago). The overall condition of the wood was considered fair: the coloration was

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gray and there was slight-to-moderate splintering. Specific locations and orientations of individual boards were documented during dismantling of the source structure; a map of the structure showing the location of each specimen tested was prepared. This map is shown in Figure 2-1. Photos are provided in Figure 2-2. This deck is referenced as the "A" source.

New Hill Deck: This source, donated for use during this project, was taken from an outdoor deck on a private residence. It represents an ideal source of relatively new, good-condition, aged CCA-treated wood. The coloration of the wood was light brown and relatively bright and there was minimal splintering. The New Hill Deck was an exposed, attached structure. There was no noticeable biological growth or other dampness-related defects. The deck was constructed of SYP, treated to 0.40 pcf with Ground Contact CCA-C, had been in service for just over one year, and had never been cleaned or treated. Specific locations and orientations of individual boards were documented during dismantling of the source structure; a map of the structure showing the location of each specimen tested was prepared. This map is shown in Figure 2-3. Photos are provided in Figure 2-4. This deck is referenced as the "C" source.

## 2.2 Preparation and Characterization of Wood Sources

### 2.2.1 Source Wood Harvesting and Preliminary Characterization

Wood specimens were prepared using nominal 5/4" x 6" cross-sectional dimensions, taken from the source structures described in Section 2.1. Care was taken to minimize handling and abrasion of the primary (i.e., 6" width) faces of the treated source boards, with the short edges of the board preferentially held during transport and cutting. New 5/4" x 6" SYP that was not CCA treated was used for the blank control specimens and the cross-contamination control specimens that were located at the ends of each minideck and between each of the four CCA-treated boards on each minideck.

For each aged CCA-treated board, the total board length was recorded along with visually-observable source wood characteristics the most important being predominant grain orientation (up versus down). Each board was also characterized as to predominant grain type (percent flat versus percent edge grain), predominant ring spacing (tight, medium, wide), predominant wood season (percent early versus percent late wood), and predominant wood type (percent heartwood versus percent sapwood). The percentages of the various grain characteristics defined below were visually observed, estimated and documented.

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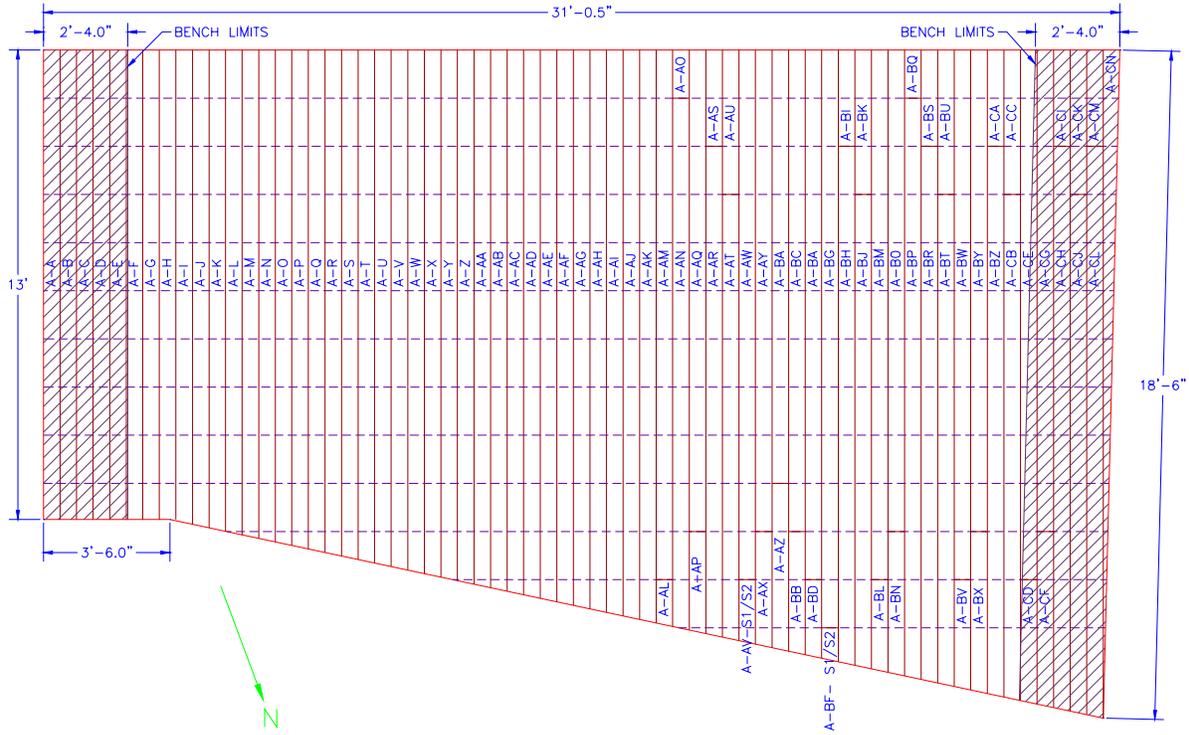


Figure 2-1. ERC Deck Map



Figure 2-2. Views of ERC Deck

Note that moisture stains were temporary and that boards under benches were not used to construct minidecks.

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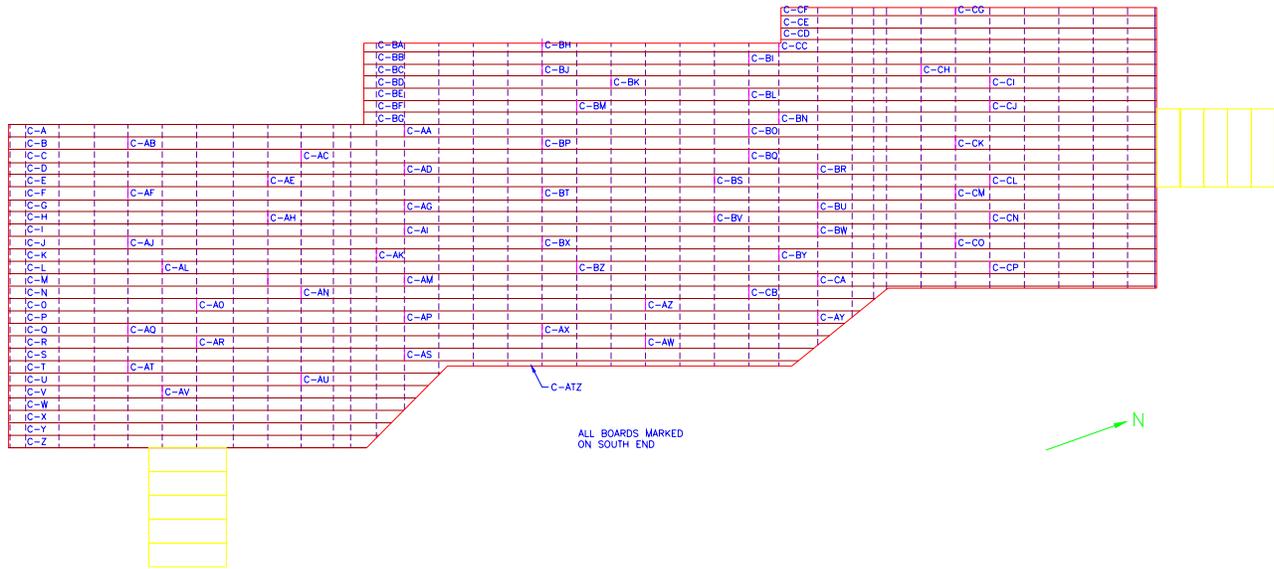


Figure 2-3. New Hill Deck Map



Figure 2-4. Views of New Hill Deck

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Aged boards were cut using a circular table saw in to specimens of approximately 86 cm (34") lengths. These lengths were cut in such a manner as to capture three sets of existing nail holes on each aged wood specimen, and required that the nail holes were spaced on 16-inch centers as typical. Of utmost concern was that the PSAs were segments of the specimen with a 38-cm (15-in) or more clear distance between adjacent nail holes, to allow for the full wipe length without crossing nail holes. Nail holes were not wiped during either the baseline or routine wipe sampling events. To prevent cross-contamination of samples, the saw was decontaminated between cutting the different types of wood and the untreated wood was cut separately (after installation of a new blade). Decontamination of the saw was done by wiping down the blade with a deionized water moistened cloth.

Where possible, the ends of each board were removed and archived and segments between each 86-cm test specimen were removed and archived. Some of these interior segments were used to characterize the source wood via moisture content measurement and wood core sampling for total arsenic, chromium, and copper analyses. The 86-cm wood specimens were visually inspected to ensure that they did not have excessive amounts of deformities, presence of heartwood, knots, resin pockets, or other defects. Each segment was identified with a unique alphanumeric code as follows:

- Aged board codes were prefixed by the letter "A" for source A, the ERC Deck source, and "C" for source C, the New Hill Deck.
- Each aged board from the two sources was identified with a unique letter (A, B, C, and so forth).
- Each space between adjacent nail holes was identified with an alphanumeric code, where the prefix "BL" refers to segments used for establishing baseline characteristics, while the prefix "M" refers to segments that were to be regularly wiped; that is, the PSAs. These codes were suffixed with sequential numbering (1, 2, 3, etc.) along the length of each source board.
- Unused, unwiped segments were designated with the prefix "X."

The specimen identification criteria presented above is illustrated by the example schematic in Figure 2-5. In this example, BL1, BL2, BL3, BL4, and BL5 would be wipe-sampled before cutting the board. These results would be used to establish baseline DCCA values for PSAs M1, M2, and M3. After cutting the boards to harvest 86-cm

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specimens (illustrated in the figure by the dashed boxes) for minideck construction, BL2 and BL5 would be subsequently used for taking one core sample each for total arsenic, chromium, and copper analyses, as well as moisture content. M1, M2, and M3 would be wipe-sampled during routine sampling events to determine coating efficacy. BL1, BL3, and BL4 would be wipe-sampled only periodically in an effort to determine the effects of abrasion and rewiping on coating efficacy and DCCA. For example, if Figure 2-5 was Board A from Source C and cut as shown by the dashed boxes, this board would generate specimens C-A-BL1, C-A-BL3, C-A-BL4, C-A-M1, C-A-M2 and C-A-M3.

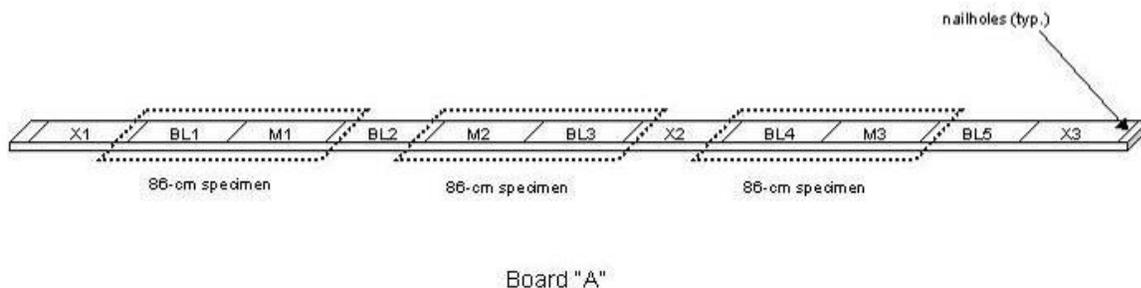


Figure 2-5. Specimen Identification and Baseline Sampling Scheme Example

All cut specimens were identified on one cut end or uncut edge with its identification code, as well as with its "top" side using permanent marker. All numbered specimens were qualitatively and semi-quantitatively characterized for visually-observable wood condition characteristics, with data recorded on a standardized wood characterization data sheet. The characteristics recorded included number of knots for that specimen, splintering, cracking, and rotting (for these last three, a rating of 1 to 5, with 5 being like new wood and 1 being complete failure, was assigned). All of this source characterization data is included in full in Appendix B (a summary table can be found in Appendix A, Table A-1). Additionally a photo record was made of all specimens which includes photographs taken at the beginning of the test (i.e., precoating), as well as at each sampling event after coating. Remaining segments of wood were retained and archived.

### 2.2.2 Baseline Sampling

Each source board used in the construction of the minidecks had at least two time = 0, baseline wipe samples taken from it prior to coating. The wipe method used to collect these samples is described in Section 2.8.3.1. Samples were then digested and

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analyzed for CCA content. The baseline wipe sample results were used to establish baseline DCCA concentrations for the PSAs on the minidecks. Furthermore, the baseline data were used to assess the variability of DCCA across each board (intraboard) and between boards (interboard) and source decks. The full dataset showing tables with summarized data and the individual specimen baseline values is provided in Appendix B. Summary tables are included in Appendix A, Tables A-2 and A-3. Data summary includes averages, standard deviations, and RSD for each board and include summary statistics at the end of each table for each source (A and C). Note that RSDs should not be interpreted as indicators of data quality, but rather as indicators of natural variability within and between boards.

The results of this sampling are presented in Section 3.1.1.

The baseline measurements were made using a different sampling method than subsequent sampling events. The A2 wipe preparation technique was used for baseline measurements and is described in Section 2.8.3.1. The 2X wipe method was used for subsequent sampling and is described in Section 2.8.3.2. Conversion factors were developed for calibrating the baseline method (A2) measurements to the main-study method (2X) measurements, taking the form  $cX$ , as indicated below. However, these have no impact on the analysis of variance because  $\ln(cX) = \ln(c) + \ln(X)$  and thus only the intercept in the ANOVA model is affected by the use of the calibrated measurements and not comparisons among coatings.

As mentioned in the previous paragraph, a separate wipe comparison study was conducted to compare the baseline (precoat) wipe sample results taken using acid washed wipes (the "A2" method) with the 2X method (as used for the subsequent sampling events) wipe preparation results. The study evaluated the need for calibration equations to convert between results obtained using different wipe preparation techniques.

Additional variables that were considered in the wipe comparison study included: grain orientation (up, down), source deck (A, C), sample date (1 month, 3 months, 7 months, 11 months), rinse (rinsed, unrinsed), and prep lab (EPA, CPSC). "Unrinsed" boards in this context refers to boards that were taken directly from storage and wipe-sampled, while "rinsed" boards were thoroughly hosed down with tap water and allowed to dry for several days before wipe sampling. "Prep lab" refers to which laboratory digested or extracted the wipes and subsequently either analyzed the samples in-house (CPSC) or sent them out to a subcontract laboratory for analysis (EPA). The full wipe comparison report is included as Appendix D.

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Statistical model selection was used to identify calibration equations for predicting method 2X DCCA measurements from method A2 DCCA measurements and the other factors, including grain orientation (up, down), source deck (A, C), sample date (1 month, 3 months, 7 months, 11 months), rinse (rinsed, unrinsed), and prep lab (EPA, CPSC). Based on these analyses, separate calibration equations are suggested for rinsed and unrinsed boards, but not for any of the other factors. In other words, when models for predicting DCCA using 2X wipes from DCCA using A2 wipes, grain, source deck, sample date, rinse, and prep lab were considered, the identified prediction model depends only on DCCA using A2 wipes and rinse.

The wipe method correction factors are summarized as follows:

- For arsenic:

Rinsed Specimens:  $As-2X = 1.42 (As-A2)$ , 95 percent Confidence Interval: (1.18, 1.66)

Unrinsed Specimens:  $As-2X = 0.80 (As-A2)$ , 95 percent Confidence Interval: (0.72, 0.88)

The R-square value for the combined models is 0.78

- For chromium:

Rinsed Specimens:  $Cr-2X = 1.31 (Cr-A2)$ , 95 percent Confidence Interval: (1.05, 1.57)

Unrinsed Specimens:  $Cr-2X = 0.81 (Cr-A2)$ , 95 percent Confidence Interval: (0.73, 0.89)

The R-square value for the combined models is 0.62

- For copper:

Rinsed Specimens:  $Cu-2X = 1.18 (Cu-A2)$ , 95 percent Confidence Interval: (0.94, 1.42)

Unrinsed Specimens:  $Cu-2X = 0.83 (Cu-A2)$ , 95 percent Confidence Interval: (0.75, 0.91)

The R-square value for the combined models is 0.81

Because the baseline analyses for this study were done on unrinsed boards, the unrinsed specimen equations were used to adjust the baseline results. In this report, only "corrected" baseline (samples taken before coating) DCCA is reported. Likewise, reported values which are calculated using baseline DCCA in the calculation (e.g., the percent reduction values) always use the corrected baseline values. DCCA from all subsequent sampling events are reported uncorrected, as they were conducted using the 2X wipe method.

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### 2.2.3 Wood Core Sampling and Analysis

Up to four wood core samples were taken from each CCA treated board used to construct minidecks for this study. Professional judgment was used to select areas from which to take core samples to ensure that their average would be representative of the board in question. Individual core samples were taken by advancing a 1/4-inch diameter drill bit through the entire 1-inch (5/4" nominal) thickness of the board and collecting the wood shavings.

The wood shavings were then dried to constant weight in a drying oven at approximately 105 °C. The dry weight of the sample was recorded. The wood shavings were then digested in concentrated nitric acid using a similar protocol to that defined in Section 2.8.4 for the wipe samples. This procedure is consistent with American Wood Preservers Association (AWPA) Standard A7-93 (microwave assisted nitric acid digestion). Digestates were analyzed by inductively coupled plasma - mass spectrometry (ICP-MS). This is consistent with AWPA Standard A21-00.

AWPA standards allow the actives composition of CCA-C – the formulation used to treat the wood used in this study – to vary between 44.5 – 50.5 percent for CrO<sub>3</sub>, 17.0 – 21.0 percent for CuO, and 30.0 – 38.0 percent for As<sub>2</sub>O<sub>5</sub> in a specific assay zone, the outer 0.6 in (15 mm) (Lebow, 1996). Knowing that the source wood for this project was treated to target retentions of 0.40 pounds per cubic foot (pcf), hypothetical, ideal actives composition (analyte concentrations) can be calculated for each CCA analyte: 0.190 pcf (86.1 g/cf) CrO<sub>3</sub>, 0.074 pcf (33.6 g/cf) CuO, and 0.136 pcf (61.7 g/cf) As<sub>2</sub>O<sub>5</sub>. Furthermore, the average dry, pretreatment density of SYP is 32 pcf, or 14.5 kg/cf. Using these values, predicted levels of CCA analytes in the study wood core samples can be approximated as:

$$\begin{aligned} \text{CrO}_3 & \quad (86.1 \text{ g/cf}) / (14.5 \text{ kg/cf}) \times (1000 \text{ mg/g}) = \mathbf{5,938 \text{ mg/kg}} \\ \text{CuO} & \quad (33.6 \text{ g/cf}) / (14.5 \text{ kg/cf}) \times (1000 \text{ mg/g}) = \mathbf{2,317 \text{ mg/kg}} \\ \text{As}_2\text{O}_5 & \quad (61.7 \text{ g/cf}) / (14.5 \text{ kg/cf}) \times (1000 \text{ mg/g}) = \mathbf{4,255 \text{ mg/kg}} \end{aligned}$$

Wood core sample data is presented in Section 3.1.2.

### 2.3 Minideck Construction

After cutting and marking specimens with their identification codes, source wood specimens were transported to the minideck host site. The minidecks were constructed on site in accordance with Figure 2-6. The minideck surfaces were initially constructed without leaving spaces between the boards. When this mistake was discovered, the

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three internal untreated boards per deck were removed and planed sufficiently on either edge enough to be spaced using 16p nails from adjacent boards. They were then reattached and the deck tops were rinsed with tap water and allowed to dry before coating. Following construction, the minideck surfaces were prepared (e.g., washed, rinsed, etc.) in strict accordance with the particular coating manufacturer's recommendations for coating aged wood as described in Section 2.5.

Three minidecks were constructed for each of the 12 selected coatings and an uncoated positive control. Minidecks were identified as 1-A, 1-B, 1-C, 2-A, and so on. Each minideck contained two 86-cm aged source "A" specimens, two 86-cm aged source "C" specimens, and five 86-cm untreated wood specimens. In addition to these 12 minidecks, a 13<sup>th</sup> (uncoated) minideck was constructed using 5 boards all of untreated SYP. The three inside boards of this deck were wipe-sampled at the prespecified regular sampling event intervals as blanks (negative controls).

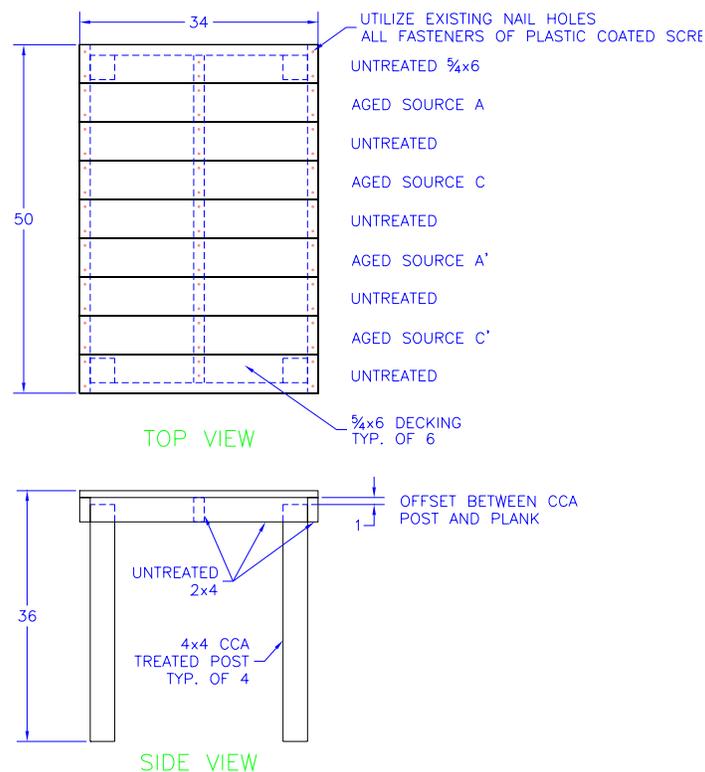


Figure 2-6. Schematic of Minideck Construction

Note that untreated 34" specimens were planed so that 1/8" of space was provided between each pair of specimens

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New 4" x 4" CCA-C treated wood posts were used in the construction of the minidecks. Specimens were screwed directly into a grid of 2" x 4" untreated SYP supports. To avoid contamination, these supports were slightly offset above the tops of the posts to ensure that the treated posts did not have the opportunity to directly contact the wood specimens. Plastic-coated screws were advanced through existing nail holes on the treated specimens in order to secure decking specimens to the minideck frames. For the untreated specimens, which were new at the time of construction, the same coated screws were used to attach them to the supports. The minidecks were free-standing (i.e., posts are not set into the ground) and were leveled after setting them on the test site. A photograph of a typical minideck is provided as Figure 2-7. Which treated wood specimens were used on each minideck is shown in Table 2-1. Specimens were matched with minidecks randomly.



**Figure 2-7. Example Minideck**

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Table 2-1. Wood Specimens Used to Construct Minidecks

Coating #	Deck ID	A – up	A – down	C – up	C–down
1	1-A	A-AE-M1	A-Z-M1	C-N-M1	C-BO-M2
	1-B	A-V-M3	A-AT-M3	C-BE-M2	C-CC-M1
	1-C	A-AJ-M1	A-BW-M4	C-S-M2	C-AA-M2
2	2-A	A-O-M3	A-BY-M2	C-BZ-M3	C-E-M3
	2-B	A-BC-M2	A-AH-M4	C-BI-M1	C-AN-M1
	2-C	A-AR-M1	A-P-M1	C-BY-M2	C-BX-M3
3	3-A	A-T-M1	A-L-M3	C-N-M3	C-CE-M2
	3-B	A-AG-M3	A-AF-M1	C-BJ-M2	C-AN-M3
	3-C	A-AD-M2	A-BW-M2	C-CD-M1	C-AA-M1
4	4-A	A-T-M2	A-BG-M4	C-CD-M2	C-AD-M2
	4-B	A-BC-M1	A-AH-M1	C-BM-M2	C-AM-M2
	4-C	A-I-M3	A-Q-M2	C-AC-M1	C-BT-M4
5	5-A	A-U-M2	A-L-M2	C-AC-M2	C-CE-M1
	5-B	A-AD-M1	A-Z-M3	C-BM-M3	C-BO-M1
	5-C	A-AR-M3	A-BG-M3	C-CA-M1	C-AD-M3
6	6-A	A-U-M1	A-BY-M1	C-BZ-M2	C-AA-M3
	6-B	A-AC-M2	A-AN-M3	C-AJ-M1	C-AI-M1
	6-C	A-BC-M3	A-P-M2	C-S-M3	C-CC-M2
7	7-A	A-O-M2	A-Y-M2	C-N-M2	C-AM-M3
	7-B	A-V-M1	A-AH-M3	C-BY-M1	C-BX-M1
	7-C	A-AJ-M3	A-BW-M1	C-BZ-M4	C-E-M2
8	8-A	A-AR-M2	A-BY-M3	C-BE-M1	C-AE-M3
	8-B	A-I-M1	A-AT-M1	C-AC-M3	C-AM-M1
	8-C	A-AG-M4	A-Z-M2	C-CA-M2	C-BX-M2
9	9-A	A-T-M3	A-P-M3	C-AP-M1	C-BW-M1
	9-B	A-AC-M1	A-AE-M2	C-BI-M2	C-AN-M2
	9-C	A-AG-M2	A-AN-M1	C-BZ-M1	C-AE-M2
10	10-A	A-AD-M3	A-BG-M2	C-AP-M3	C-AD-M1
	10-B	A-X-M1	A-Y-M1	C-BJ-M1	C-AK-M4
	10-C	A-AJ-M2	A-Q-M3	C-BU-M2	C-BT-M2
11	11-A	A-U-M3	A-Q-M1	C-AP-M2	C-AI-M3
	11-B	A-X-M2	A-AH-M2	C-BE-M3	C-BW-M2
	11-C	A-AJ-M4	A-BW-M3	C-BJ-M3	C-AE-M1
12	12-A	A-O-M1	A-AN-M2	C-AJ-M2	C-AM-M4
	12-B	A-AC-M3	A-AE-M3	C-BI-M3	C-AD-M4
	12-C	A-V-M2	A-L-M1	C-BM-M1	C-BT-M1
13 (uncoated)	13-A	A-AG-M1	A-Y-M3	C-S-M1	C-E-M1
	13-B	A-I-M2	A-AT-M2	C-AJ-M3	C-AI-M2
	13-C	A-X-M3	A-BG-M1	C-BU-M1	C-BT-M3

## 2.4 Selection of Coatings

Basic formulation and application information for a large number of products was obtained primarily by using Internet searches and visits to local retail hardware and home improvement stores and compiled into a master list of 125 products. Products selected for testing are listed in Table 2-2 and include oil and water-based sealants and stains, paints, and products marketed to encapsulate CCA-treated wood.

For preservation of appearance of CCA-treated decks, experts generally recommend use of penetrating finishes such as oil and water-based sealants rather than film-forming products such as paints due to the tendency of film-forming products to crack and peel as the substrate shrinks and swells with uptake and release of moisture. (See for example, Williams and Feist, 1993.) For this reason, eight of the twelve products selected were oil and water-based sealants and stains that fall into the category of penetrating finishes. The oil and water-based paints were selected because porch and deck paints are widely available to consumers and may be applied to CCA decking. The two products that were marketed to encapsulate CCA treated wood were selected because they were marketed as products that would reduce exposure to CCA residues and could be readily applied by consumers.

Products were classified by base (oil vs. water), cover (clear, semi-transparent, opaque), and product type. Product type was broken out into the following: paints, primers, sealants, stains, and other. Additional classification descriptors include ingredients (primarily alkyd or acrylic), surface (penetrating vs. film-forming) and color.

Table 2-2 generically lists and characterizes the 12 products selected for the study. The list includes two paints, two products specifically marketed to prevent DCCA exposure, eight representatives of the combined stains and sealants category (four oil-based products and four water-based products, one of which was alkyd-based, one acrylic, one both alkyd and acrylic, and one specifying neither).

Table 2-2. Selected Products for Evaluation

#	Product Type	Base	Cover	Main Ingredients	Comments
1	Sealant	Oil	Semi	Aliphatics, Napthalene	"Cedar" with UV blocker
2	Sealant	Oil	Clear	Acrylic, alkyd, urethane	"Clear"
3	Stain	Oil	Clear	Acrylic	"Deep tone base"
4	Stain	Oil	Clear	Alkyd	"Clear stain"
5	Sealant	Water	Clear	Unknown <sup>1</sup>	"Clear"
6	Sealant	Water	Clear	Acrylic, alkyd	"Clear"
7	Stain	Water	Semi	Alkyd	"Cedar" with UV blocker
8	Stain	Water	Clear	Acrylic	"Tint base, solid" with no tint added <sup>2</sup>
9	Paint	Water	Opaque	Acrylic	"Gray". Latex, designed for porches and floors
10	Paint	Oil	Opaque	Alkyd, polyurethane	"Gray". Designed for porches and floors
11	Other	Water	Clear	Elastic vinyl	Designed for CCA encapsulation
12	Other	Water	Clear	Polymer	Designed for CCA encapsulation
13	No coating	N/A	N/A	N/A	Uncoated control minidecks

<sup>1</sup> MSDS for coating listed no ingredients >1 percent.

<sup>2</sup> Note that the labeling for product #8 specifically states that it must be tinted before use.

## 2.5 Coating Application

After baseline characterization of source wood, construction of the minidecks, and preparation of the minidecks for coating, all exposed surfaces of the decks were coated in accordance with coating manufacturers' recommendations. Coatings were applied to fully cover the top faces, exposed uncut edges, and cut ends of CCA-treated wood specimens. For the paints, products #9 and 10, a common latex primer was first applied in accordance with the paint and primer manufacturers' instructions. Top faces were coated first, followed by the exposed edges and the cut ends. Because the coatings' manufacturers generally recommended that application not be done during periods of direct sunlight, a tent was temporarily set up on site to allow for coating minidecks in the shade. After 24-hours of initial coating drying in the shade, minidecks were manually relocated to allow for additional drying in exposed conditions.

Flow sheets generically detailing the wood preparation procedures employed for each coating are provided as Appendix C. The surfaces of the minidecks for each coating

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(except coating #7, which did not call for a rinse prior to coating) were rinsed with a pressure washer at a 1,000-3,000 psi setting. Ten of the coatings also had a deck cleaning product applied, as specified in their instructions. Note that specific products used to prepare the minidecks for coating are not mentioned in order to maintain coating confidentiality. The surfaces of the positive [coating #13 (uncoated)] and negative control minidecks were rinsed with a pressure washer at a 1,000-3,000 psi setting.

Two types of 2" chip brushes were used to apply coatings to the minidecks. One brush with natural bristles and the other with polyolefin bristles (to apply coatings that recommended a synthetic bristled brush). Prior to coating application, both brush types were analyzed in order to ensure that they did not contribute significant amounts of arsenic, chromium, or copper to the wood surfaces. Each type of brush used was prequalified for use per a set of two control samples whereby two brushes of each type were agitated in a 250 mL vessel containing 40 mL of deionized water for 30 seconds. The liquid samples were then transferred to Digtubes for digestion. Four milliliters of nitric acid were added to each tube and the samples were then digested at 95 °C for two hours. Digested samples were sent to STL, the subcontract analytical laboratory used for this project, for analysis. Arsenic levels were found to be below the reporting limit of 0.1 µg/L, chromium levels were <1 µg/L and copper levels were lower than levels seen on blank, untreated boards. A summary of the data is tabulated in Appendix A, Table A-4.

Three brushes were used for each minideck, a new brush for coating each of the aged wood sources and to the new untreated wood surfaces. Untreated surfaces were coated first, followed by the aged CCA surfaces. Brushes were prepared for initial coating application in accordance with brush manufacturer's recommendations. After a particular coating was applied to a given group of triplicate minidecks, used brushes were archived.

Separate aliquots of coating were used for each minideck in order to prevent cross-contamination of coating by re-dipping the brush applicator. Three aliquots of coating were used for each minideck: one for the "A" specimens, one for the "C" specimens, and one for the untreated (termed "N") specimens. Separate aliquots of coating were poured into disposable plastic graduated volumetric beakers. The disposable beakers were acid-washed using a procedure similar to that specified in Section 2.8.4.1 prior to use. Poured but unused coating was composited so that one sample was retained for each coating and wood type (new and untreated and the two, aged, CCA-treated sources). These leftover coating samples were stored in sealed, unused paint

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containers and archived for possible future analyses. Application procedures and any notable observations were documented for each coating.

The amount of coating, measured as both volume and mass, applied to each substrate on each minideck was determined by transferring 200 to 300-mL of coating directly from the original coating container into a 400-mL graduated beaker. The starting volume of coating in the beaker and the final volume (after squeezing out excess coating from the brush used) were recorded and, from these, a calculation of the volume of coating applied was made. Additionally, the container of unused coating and the brush to be used for application was preweighed. After the coating was applied, the final weight of the beaker and brush was measured and recorded. The weight applied was calculated as the difference between the initial and the final weights. Unused aliquots of each coating tested were sampled in duplicate, prepared, and analyzed in accordance with methods specified in Section 2.9.

A coating application data form was completed for each coating. A sample form is provided as Appendix F.

The volume and mass of coating applied to the A, C, and N boards (the untreated boards) on each minideck were determined. No coating mass data are provided for coating #1, because the decision to measure mass was made after the coating #1 decks were coated. Measured volumes were rounded to the nearest increment of 5 mL. The method for determining mass applied was more accurate and mass measurements are reported to the nearest 0.1 g.

Results of volume and mass measurements are presented in Section 3.1.4.

## 2.6 Outdoor Weather Site Setup

The objective of the natural weathering tests was to evaluate the effects of weathering on the efficacy of the selected coating products in reducing DCCA from aged, in-service CCA-treated wood. Minidecks were randomly assigned to gridded blocks, with the qualifier that minidecks featuring the same coating were not allowed in the same row, column, or diagonally immediately adjacent to one another. The site layout showing sequentially numbered minideck locations is provided as Appendix G. The location of each minideck is summarized in Table 2-3 – the blocks listed in Table 2-3 correspond to those shown on the site plan in Appendix G.

**Table 2-3. Minideck Block Assignments (blocks correspond to those identified in Appendix G)**

Minideck	Block	Minideck	Block
1-A	6	9-A	46
1-B	29	9-B	3
1-C	22	9-C	26
2-A	39	10-A	42
2-B	7	10-B	27
2-C	33	10-C	24
3-A	36	11-A	30
3-B	10	11-B	4
3-C	14	11-C	20
4-A	37	12-A	1
4-B	23	12-B	35
4-C	5	12-C	21
5-A	25	13-A	19
5-B	43	13-B	40
5-C	9	13-C	12
6-A	13	BC	8
6-B	28	NC	47
6-C	45	LH	15
7-A	2	SC1	31
7-B	41	SC2	38
7-C	34	SC3	16
8-A	18	LC1	44
8-B	32	LC2	11
8-C	48	LC3	17

- BC = the blank, negative control, minideck
- NC = 1 minideck with no CCA wood used (for a related bioavailability study being conducted)
- LH = 1 uncoated CCA minideck for leachate collection (for a related bioavailability study being conducted)
- SC1, SC2, SC3 = 3 soil controls (for a related bioavailability study, no minidecks are located in these blocks)
- LC1, LC2, LC3 = 3 leachate controls (for a related bioavailability study, no minidecks are located in these blocks)

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The site was prepared for testing by:

- Setting up deposition samplers (constructed of new untreated wood) on-site and periodically wipe sampling them to assess the potential for atmospheric deposition of CCA analytes, in order to qualify the site. The results of the deposition samples showed that background levels of CCA analytes were negligible.
- Delineating a currently grassed, relatively remote area for testing minidecks, and preparing the area by tilling the ground to 6" total depth, leveling it to remove potholes, and lightly rolling it to prevent dust and erosion and prepare for graveling, but not overly compacting it. This area was then gridded using landscaping fabric and crushed stone to prevent vegetative growth, which would require maintenance, such as mowing, that might result in unacceptable impacts to the decks (e.g., dust and grass clippings). Note that the crushed stone and landscape fabric that were used were tested for arsenic content, which was determined to be negligible. Note that the space underneath the minidecks was not covered with landscape fabric or gravel. Vegetation in these areas is controlled manually, by hand, to accommodate bioavailability testing being conducted by other researchers.
- Delineating the perimeter of minideck test area to alert landscape maintenance staff to avoid the area. The entrance road to the site has a gate that is locked every evening at 6:00 pm until the next morning and vandalism was not a problem.
- Clearance of saplings from the area to prevent shading.
- After placement within their assigned gridded spots, minidecks were leveled in both directions. Level placement was confirmed using an engineer's level, with untreated 2" x 4" spacer blocks to prevent direct contact between the level and the untreated end pieces of the minidecks.

## 2.7 Weather Data Collection

Weather data were collected for the outdoor weathering tests using a Davis Instruments Vantage ProPlus weather monitoring station. The station is located as shown in Figure 2-8. Through the use of available software (WeatherLink for VantagePro), data from the weather station was compiled and downloaded to a Microsoft Excel file, although there were several periods during the second year of operation when the data logger malfunctioned and on-site data was missing. The WeatherLink software allows the user to store data in the VantageProPlus console and download to a computer at their convenience. Data that was routinely collected via the Vantage ProPlus are listed in Table 2-4.



**Figure 2-8. Minideck Site**

*Note weather monitoring station on the right.*

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**Table 2-4. Vantage ProPlus Weather Station Data**

	Units			
Barometric Pressure	in Hg	Mm Hg	hPa (Tor)	Mb
Inside Humidity	percent			
Outside Humidity	percent			
Dew Point	°F	°C		
Rainfall	In	Mm		
Rate of Rainfall	in/hr	Mm/hr		
Solar Radiation	W/m2			
UV Index & Dose	index	Meds		
Inside Temperature	°F	°C		
Outside Temperature	°F	°C		
Apparent Temperature	°F	°C		
Wind Speed	Mph	m/s	km/h	
Predominant Wind Direction	N, NNE, NE, ENE, E, ESE, SE, SSE, S, SSW, SW, WSW, W, WNW, NW, NNW			
Wind Chill	°F	°C		

The data can be archived at 1 min, 5 min, 10 min, 15 min, 30 min, 1 h, or 2 h.

Data was archived at 30-minute intervals.

All data points are discrete except for Rate of Rainfall and UV Dose.

The National Oceanic and Atmospheric Administration (NOAA) in RTP, North Carolina, collected data on wind speed and direction, temperature, precipitation amount, direct solar radiation, and total solar radiation at the site used for minideck weathering through the first year of this testing. A summary of the data collected by NOAA at the site are listed in Table 2-5. The weather data available from NOAA at the test site, however, had generally been collected on strip charts requiring conversion to allow comparison with data obtained from the Vantage ProPlus weather station.

Table 2-5. NOAA-Generated Weather Data

Parameter	Unit	Remarks
Required		
Irradiance (UV)	W/m <sup>2</sup>	Direct and total radiation is available.
Temperature	°F	
Precipitation, Duration	hours	Can be determined from strip chart, although certain losses may occur due to evaporation.
Precipitation, Amount	inches	Automated rain gage.
Dew Point (Measure of dew formation)	°F	Dew point could be used to calculate dew point depression (diff. with temp.) If DPD is small, there is likely to be dew overnight.
Wind direction + speed		

The test site NOAA metrology instrumentation was calibrated against working standards traced to world standards at Eppley Laboratories. This calibration was conducted periodically based on the stability of the instrument and the judgment of the instrument operators who were not otherwise associated with this project. The temperature system was checked against certified data from NOAA’s Raleigh—Durham International Airport (RDU) weather station on stable days and also with a sling psychrometer. The weighing rain gage was calibrated with weights and also against a manual rain gage with each precipitation event. The Aerovane wind system recorded wind speed in miles per hour (mph) and only begins to register at 3 mph. It was also checked against RDU on stable windy days.

These and other parameters are additionally collected by the NOAA’s National Climate Data Center (NCDC), at their Raleigh-Durham International Airport (RDU) weather station, and are available in monthly summaries, detailing specified conditions on a daily and, for some parameters, an hourly basis. The RDU station does not monitor solar radiation. Supplemental solar radiation measurements were obtained from the State Climate Office of North Carolina for their Reedy Creek Field Laboratory monitoring site, also in close proximity to the EPA test site.

NOAA-generated data from the test site were compared to data from the Vantage ProPlus weather monitoring station dedicated for use during this project. Spot checks of all parameters common to both the NOAA and Vantage ProPlus unit were conducted and determined to be well within reasonable tolerances (± 5-10 percent

depending on parameter). Because the on-site NOAA weather data became unavailable several months into the study, certified data from NOAA's RDU station was also used to confirm the on-site weather monitoring station data. Details of these spot check results are provided in Appendix H. Finally, the solar radiation data from the Reedy Creek site was compared with data from the weather station at the test site and showed agreement within 1 percent on a monthly average basis. All sets of comparisons are in agreement, thus confirming the accuracy of the on-site weather monitoring station data and justifying the use of select data from alternative sources as necessary.

During the second year of the study, the performance of the on-site weather station's data logger became unstable and for several periods, data was not properly logged, so the weather data was supplemented by NOAA-RDU data and State Climate Office of North Carolina, Reedy Creek Station data.

## 2.8 DCCA Measurement Methods

### 2.8.1 Sampling and Extraction Methods

Prior to beginning wipe sampling events, testing was performed to determine characteristics of the wipes and their ability to retain levels of target compounds. These tests were fully described in the Interim Report and are summarized here.

#### 2.8.1.1 Wipe Blank Study

A number of blank evaluations were performed to evaluate the amounts of target compounds inherent in the extraction process and the wipes themselves. Initially, wipes directly out of the bag were acid extracted. Blank results for arsenic and chromium are shown in Table 2-6. Initial wipe blank analysis did not include an analysis for copper.

Table 2-6. Wipe Blank Analyses

Sample Number	As ( $\mu\text{g/L}$ )	Cr ( $\mu\text{g/L}$ )
AQS-54	1.2	0.8
AQS-55	1.3	1.1

To investigate the potential to reduce the background levels of As and Cr seen in the wipe blanks, nine wipes were pretreated by acid extraction, rinsing thoroughly with de-ionized water, and allowing them to dry in a clean environment. Results comparing the pretreated wipe blank to the out-of-the-bag wipes are shown in Table 2-7.

**Table 2-7. Results from September 2003 Wipe Comparison Study**

Average	As (µg/L)	Cr (µg/L)	Cu (µg/L)
Wipe Blank (Out of Bag)	0.41	0.93	2.1
Digested Nitric Blank	<0.10	<0.50	0.3
Acid Wipe Blank	0.2	<0.50	1.3

Results for the initial out of bag wipe blanks experiments were performed using a whole 12" x 12" wipe. The data collected from the September 2003 Wipe Comparison Study were performed using half of a wipe, thus the concentrations for the half a wipe should be lower than from a whole wipe.

*2.8.1.2 Spiking Study*

A number of spike studies were done to ensure that the analytes of interest could be captured with the wipes, extracted and analyzed. Three samples were prepared by spiking known amounts of arsenic and chromium standard stock onto a clean glass plate and allowing the liquid to evaporate. Each glass plate was then wiped using the CPSC technique and the wipes were extracted and analyzed. Recovery results are shown in Table 2-8.

**Table 2-8. Results of Spiking onto Glass**

	As	Cr	Units
Spiked Amount	50	49.75	µg
Sample AQS-56	33	38	µg
Sample AQS-57	41	46	µg
Sample AQS-58	41	46	µg
Average	38.3	43.3	µg
SD	4.6	4.6	µg
Percent recovery (av)	77 percent	87 percent	
Percent RSD	12 percent	11 percent	

Less than 100 percent recovery of the metals could be expected due to the drying of the spike solutions on the glass. A small amount of residue was seen left on the glass after wiping with the wiping apparatus. The residue could be removed with further cleaning which indicated that the stain was metal salts and not etching.

Next, pretreated wipes were directly spiked with 1 µg/L, 50 µg/L and 1000 µg/L of arsenic, chromium and copper standard. These samples were extracted and analyzed by the laboratory. Results are shown in Table 2-9.

Table 2-9. Results of Spiking Wipes Directly

Sample ID	Arsenic (µg/L)	Recovery (Percent)	Chromium (µg/L)	Recovery (Percent)	Copper (µg/L)	Recovery (Percent)
SS-562	1.0	100	1.0	100	1.2	120
SS-563	47	94	51	102	47	94
SS-564	1100	110	1000	100	970	97

In addition to liquid standards, CPSC also provided ARCADIS/EPA with a standard CCA Dust Material that contained a known amount of arsenic. A known weight of this material was placed directly into extraction vessels containing the acid-rinsed wipes and the samples were extracted and analyzed. This spiking was done in duplicate and recoveries for arsenic were 98 percent and 102 percent. As a result of the spike studies, it was determined that arsenic, chromium and copper could be adequately recovered from wipe samples.

#### 2.8.2 Wipe Sampling Events

Wipe samples were taken directly from the top faces of the specimens on the minidecks. Each specimen had three sets of nail holes and two possible sampling areas: a BL area and a PSA area. The length of all wipe samples was 15 inches to avoid contact with nail holes which are typically spaced 16 inches on-center.

The wipe sampling device (see Figure 2-9) used for the tests was based on a design and construction by CPSC, who also developed the associated method (CPSC staff 2003b). The CPSC staff wipe sampling device utilizes a 1.1 kg disc that is approximately 8.65 cm in diameter as the wiping block (note that the actual width of 5/4" x 6" decking is approximately 5.5" or 14 cm). The sampler allows for the use of variable wipe lengths; for this study, 38 cm was used, yielding a sampling area of

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approximately 314 cm<sup>2</sup>. The EPA developed wipe method incorporates relatively minor modifications from the CPSC method, as illustrated in Section 2.8.5



Figure 2-9. CPSC Wipe Sampling Apparatus

#### 2.8.2.1 Baseline Sampling of Boards and Minidecks

The baseline areas were wipe-sampled prior to coating, using wipes that had been prewashed in nitric acid, in an attempt to remove trace contaminants from the wipes. It was later determined that subsequent DI water rinsing steps were not sufficient in removing the nitric acid from the wipe and it was decided to not continue to use this method of prewashing wipes for the routine wipe samples taken 1, 3, 7, 11, 15, 20 and 24 months after coating due to concerns about the unnatural and potentially detrimental effect of the acid on the coating.

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Individual baseline DCCA values were determined for each PSA on each specimen to be coated and tested. The baseline DCCA of a PSA was determined by averaging the precoating baseline DAs from the two adjacent baseline (BL) specimens on either side of the PSA. However these BL sample results were ultimately not used in the statistical model used to analyze the time series coating performance data, in part to avoid complications arising from the acid-washing method used for the BL samples, as mentioned above.

The other sampling area, the PSA, was not wipe-sampled prior to coating to avoid artificially removing DCCA from the PSAs before coating them. These areas were wipe-sampled at specified intervals using wipes that were straight out of the bag and wetted, as described in Section 2.8.3.2, to avoid any potential detrimental effects of residual acid from the wipe washing procedure on the performance of the coatings.

#### 2.8.2.2 Subsequent Wipe Sampling Events

Subsequent sampling was done with “out of the bag” wipes, simply wetted with DI water. The wipe methods employed are described in Section 2.8.3.3. Several more qualitative measures were taken to qualify and document wipe-sampling events:

- Wipe sampling events were only conducted when specimens appeared dry and when weather forecasts indicated that consistent, dry weather would prevail for the entire sampling event. Actual climatic conditions were recorded and well-documented throughout the entire study, including sampling events.
- During each sampling event, each minideck was digitally photographed, with wiped and unwiped areas identified, in a running photolog.

Routine control samples that were taken included:

- Three negative control wipe samples taken; one from each of the three interior untreated, uncoated specimens on the blank minideck. These control measurements provided an indication of whether there is significant atmospheric deposition of CCA analytes at the site.
- One untreated (but coated, for minidecks prefixed 1 through 12) specimen from each minideck was wipe-sampled. Because there are five untreated specimens on each minideck, there are a total of 10 such potential sampling areas. The specific

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areas sampled during each routine sampling event were randomly selected for each minideck and were different for each sampling event.

### 2.8.3 Wipe Sampling Methods

#### 2.8.3.1 EPA Acid-Wash, Rinse, and Saturate with DI Water Wipe Preparation Technique (A2 Method)

For the precoat and baseline sampling events, TexWipes TX1009 clean room wipes (100 percent continuous filament polyester), were cut in half using a new razor blade that had been cleaned using acetone and a lint-free wipe on a acetone cleaned lab bench. After cutting, the half-wipes were placed in a wide mouth glass bottle and soaked in a 10 percent solution of Trace Metals Grade Nitric Acid. The bottle was placed in an oven at 85 °C overnight. When the bottle was removed from the oven, the nitric acid solution was decanted and wipes were rinsed in the bottle five times with deionized H<sub>2</sub>O. After the final rinse, each wipe was removed and squeezed by hand so that they were damp but no more water could be removed. The damp wipes were placed into individual Digitubes until used for wipe sampling. This technique was determined to yield moisture contents of  $2.1 \pm 0.1$  (1 standard deviation) times the dry wipe weight.

#### 2.8.3.2 EPA 2X DI Water Wipe Preparation Technique (2X Method)

The EPA wipe preparation procedure for subsequent sampling events (samples taken 1, 3, 7, 11, 15, 20 and 24 months after coating) TexWipe TX1009 clean room wipes were cut in half using a new razor blade or scissors cleaned using acetone and a lint-free wipe on a acetone cleaned lab bench. After cutting, the half-wipes were inserted into PTFE tubes, into which two times the wipe weight in DI water was added to be soaked up by the wipe. Therefore the wet wipe was three times its dry weight. Wetted wipes were stored in their sealed PTFE tubes until use. Sampling staff, while cutting, transferring, and wetting the wipes, wore nitrile or latex gloves at all times.

#### 2.8.3.3 Standard EPA Wipe Method (Adaptation of CPSC Staff Method)

The adaptation of the CPSC wipe method employed by EPA for all sampling events is described below. The differences between this method and the CPSC method are listed in Section 2.8.5.

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Prior to starting a new wipe sample, the sampling staff put on a new pair of disposable nitrile or latex gloves. These were worn over a second pair of gloves that are not changed between wipes (i.e., double gloved). The rubber-coated side of the steel rubbing disk was covered with plastic wrap. The wetted wipe was then removed from the PTFE tube, folded in half, and placed over the plastic wrap and secured with a plastic tie-wrap strap. The disk was lowered so that it was in contact with the wood. Sampling staff slide the disc along the tracks of the sampling apparatus forward and backward for five 38-cm (15-inch) strokes between nail holes while another person held the end of the wiping device in place. A stroke consisted of one forward and back movement. The speed of sampling was variable depending on the quality of the area being wiped, with rougher wipe areas required longer sampling times (slower average speeds). Splinters and sampling area imperfections could “hold up” the sampler requiring the person doing the wipe to adjust the horizontal force they exerted on the weight to continue moving it forward. The wipe was rotated 90° on the rubbing disk, which was then slid forward and back for five more strokes, for a total of 10 front-and-back strokes. Sampling staff then removed the wipe from the disk and placed it back into its PTFE extraction tube. Wood splinters larger than a grain of rice were removed prior to placing the wipe in the extraction vessel.

After the sample was taken, the plastic wrap was discarded and the wiping apparatus was decontaminated by wiping the rails of the apparatus which were in contact with the wood surfaces with lint-free wipes wetted with DI water. The apparatus was then checked for structural integrity and any loose bolts are tightened. Finally, sampling staff removed and discarded their gloves and if more samples were required the procedure was repeated.

#### 2.8.4 Wipe Extraction and Analysis Techniques

##### 2.8.4.1 EPA Laboratory Technique

Wipe samples were prepared for analysis using techniques employed by CPSC staff (2003) and Stilwell, et al. (2003), with adaptations for use with laboratory equipment available for this project. As such, a microwave- or heat-assisted extraction and digestion procedure was employed comparable to that used in prior studies, and similar to SW-846 Methods 3051 and 3052.

Precleaned disposable digestion vessels were used for sample collection and digestion. All volumetric glassware was prepared by acid cleaning by leaching with hot 1:1 nitric acid for a minimum of two hours, then rinsed with deionized water and dried in

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a clean environment.  $30 \pm 0.1$  mL 10 percent nitric acid (trace metal grade  $\text{HNO}_3$ , DI  $\text{H}_2\text{O}$ ) was added slowly to the digestion vessel containing the wipe sample to allow for preextraction. Once any initial reaction had ceased, the sample was capped and introduced into an Environmental Express HotBlock metals digestion system. The vessels were placed into the digestion system and heated for 1 hour at  $95^\circ\text{C}$ . After digestion system extraction, sample vessels were allowed to cool for a minimum of 5 min. prior to removing them from the system. The liquid was poured off into a 100 mL volumetric flask. In addition, as much extraction liquid as possible was squeezed by hand from each wipe. The gloves, funnels, and flask necks were rinsed with DI  $\text{H}_2\text{O}$ . The extracted wipe was then placed back into the extraction flask with an additional 30 mL of 10 percent  $\text{HNO}_3$ .

Again, the vessels were placed into the digestion system and heated for 1 hour at  $95^\circ\text{C}$ . After extraction, the liquid was poured off into the aforementioned 100 mL volumetric flask. As much extraction liquid as possible was squeezed by hand from each wipe and the gloves, funnels, and flask necks are rinsed with DI  $\text{H}_2\text{O}$ . The wipe was placed back into the extraction vessel and 20 mL of 10 percent  $\text{HNO}_3$  was added to each extraction vessel before the digestion system cycle was repeated. The extract was then poured into the 100 mL volumetric flask. Deionized water was used to rinse the extraction vessel; rinsate was added to the 100mL volumetric flask. If necessary, deionized water was added to take the contents to the 100 mL level. The contents of the 100 mL flasks were then transferred to and stored in two plastic tubes (duplicate or split samples) with plastic caps. One was sent to a contract laboratory for analysis, while the other was archived, under refrigeration or freezer storage. These tubes were manufactured by SCP Science made of virgin polypropylene and are certified contaminant-free.

Per the specified analytical method, the hold time for all metals other than mercury is 6 months.

Analyses for total arsenic, chromium, and copper were conducted by STL in Savannah, Georgia, using a modification of SW-846 Method 6020 (ICP-MS). STL utilizes ICP-MS for arsenic analysis, modifying the technique to utilize hydrogen plasma, rather than argon as classically performed. This modification eliminates concerns over the formation of  $\text{Ar}^{40}\text{Cl}^{35}$ , which can create a positive bias when measuring As. STL-Savannah's analytical method has reporting limits of  $0.10 \mu\text{g/L}$  for all three CCA analytes (this corresponds to a DAs of  $0.000032 \mu\text{g/cm}^2$ )

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STL is an accredited laboratory, participating in the Contract Laboratory Program (CLP), as well as numerous state programs. In addition to prequalifying the laboratory for use in the minideck study, each set of samples submitted included blind blanks and spiked samples, allowing for continued monitoring of laboratory performance during the project.

#### 2.8.5 Differences Between EPA and CPSC Procedures

Differences between the CPSC staff and EPA 2X methods for collection and analysis of surrogate wipes on CCA-treated wood include:

- ARCADIS/EPA uses plastic wrap to cover the rubber-coated side of the rubbing disk rather than Parafilm.
- C-clamps are not used by EPA to secure the horizontal wiper (because the boards being wiped are part of a deck structure). An assistant holds the wiper in place.
- In the EPA method, poly wipes are immediately placed directly into the vessels in which extraction will take place.
- A three-step extraction and digestion procedure is used by EPA rather than the CPSC staff one-step water bath extraction and digestion.
- EPA uses a 2X DI water spike (wetted wipe weight is three times the dry wipe weight) to prewet the wipes while CPSC staff uses a 1x 0.9 percent saline solution spike (wetted wipe weight is two times the dry wipe weight).
- EPA uses a 38-cm (15-in) wipe length (nominal 314 cm<sup>2</sup> sampling area) and samples between nail holes of boards supported 16 inches on-center, while CPSC staff uses a 50-cm (19.7-in) wipe length (nominal 386 cm<sup>2</sup> sampling area).

#### 2.8.6 Calculation of DCCA from Extraction Fluid Concentrations

Raw data from the subcontract analytical laboratory were reported in units of µg/L and represent the mass of analyte per unit volume of extraction solution sent to the laboratory. For standard wipe sample results, data were reduced in order to characterize the mass of analyte per unit surface area wipe-sampled, in units of µg/cm<sup>2</sup>, using the following equation:

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$$C_{DA} = \frac{C_{DF} \times \frac{V}{1000}}{A} \quad (\text{Equation 2-1})$$

Where:

$C_{DCCA}$  = DCCA of a sample ( $\mu\text{g}/\text{cm}^2$ )

$C_{DF}$  = Concentration of analyte in extraction fluid ( $\mu\text{g}/\text{L}$ )

$V$  = Total volume of extraction fluid (mL)

$A$  = Area of wiped surface ( $\text{cm}^2$ ) =  $314 \text{ cm}^2$

Using the reporting limits of  $0.1 \mu\text{g}/\text{L}$  from the laboratory, this correlates to a sampling detection limit of  $0.000032 \mu\text{g}/\text{cm}^2$ .

## 2.9 Preparation and Analysis of Coating Samples

Total arsenic, chromium, and copper in the coatings themselves were determined in the same manner used to analyze the wipe samples (acid digestion and extraction followed by ICP-MS). The coating was thoroughly shaken to ensure homogeneity and then an aliquot was transferred to a tared PTFE digestion vessel and allowed to dry. Following loss of volatiles through drying, the residue was digested using concentrated nitric acid as described in EPA SW-846 Method 3052. Additionally, hydrofluoric acid (HF) was added as necessary to ensure complete digestion in accordance with the method. Specifically, 9 ml of concentrated nitric acid and 3 ml of concentrated hydrofluoric acid were added and the samples were microwaved for 25 minutes at 50 psi followed by 45 minutes at 80 psi. The HF has to be neutralized before analysis. To do this, 30 ml of a 4 percent boric acid solution was added and the sample was digested a second time in the microwave, for 30 minutes at 50 psi. In this process, the HF is converted to  $\text{BF}_3$ , which is a gas and leaves the sample. The digestate was quantitatively transferred to a volumetric flask and brought to 100 ml with 10 percent nitric acid before submission to the contract laboratory for ICP-MS analysis (SW-846 Method 6020).

These results are presented in Section 3.5.

## 2.10 Archiving of ICP-MS Samples

Analysis of the samples by ICP-MS consumed only a fraction of the submitted sample. ARCADIS/EPA archived an aliquot of each digestate, to be held until the completion of the project. Samples were archived by storing them in TFE or PFA containers under refrigeration. Additionally, any remaining sample volume at the contract analytical laboratory was archived until the analytical results were confirmed.

## 2.11 Moisture Analysis of Wood Specimens

Initial precoat wood moisture content was measured using a hand-held meter, after the technique had been qualified and calibrated via side-by-side testing with the drying oven technique, ASTM D4442 (Primary Oven Drying). Per ASTM D4442, a small representative wood sample was weighed prior to drying overnight at 103 °C in a forced air oven. After 24 hours, the sample was cooled in a desiccator, weighed and returned to the oven. The process was repeated until weight changes between weighings were within ± 5 percent.

The results of the moisture analyses are included in the Specimen Characterization Data contained in Appendix B.

## 2.12 Miscellaneous Samples

Other samples that were collected during the study and archived or analyzed are summarized in Table 2-10.

Table 2-10. Miscellaneous Samples Collected

Sample Description	# Samples Analyzed	# Samples to be Archived
Unaltered coating	2 for each coating	Leftover coating to be stored
Leftover brush-applied coating	N/A	1 for each coating and wood type
Brush wash water	2 for each brush type	Brushes are retained
Wood	Up to 4 cores per board	Leftover wood is stored

## 2.13 Quality Control Samples

The following types of quality control samples were included: (1) positive (CCA-treated, uncoated) controls, (2) negative (untreated, uncoated) controls, (3) cross-

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contamination controls, (4) wipe frequency (rewipe, abrasion) controls. Each is discussed briefly below.

#### 2.13.1 Positive (CCA-Treated, Uncoated) Controls

The three minidecks prefixed by the number 13 were constructed in exactly the same way as the minidecks for coatings #1 through #12, except that they were not coated. The results from these minidecks were used to determine how the DCCA values vary over time without considering the effect of coating. As such, control deck DCCA values could be determined at each sampling event for comparison with DCCA from the coated decks. These positive control results were used to assess coating performance in the statistical data analysis.

The advantage to using the positive control minidecks for this purpose was that other potentially important factors were incorporated in the minideck 13 (uncoated) results, including: the effect of rinsing the boards precoating (via comparison of precoat and samples taken 1 month after coating for the positive control minidecks), the effect of weathering between subsequent sampling events, and the effect of climatic conditions during the sampling itself. The use of the positive control minideck DCCA results thus allowed for these and other potential sources of bias to be considered in the data analysis.

These results are represented by the data presented for Deck #13 (uncoated).

#### 2.13.2 Negative (Untreated, Uncoated) Controls

The single uncoated minideck, labeled BC (for "blank control"), consisting of five untreated specimens, was used to routinely take blank samples to measure the background levels or atmospheric deposition of analytes. Wipe samples were taken from the same areas of the middle three boards on this deck during each monitoring event, similar to samples taken from the other minidecks.

These results are presented in Section 3.1.5.

#### 2.13.3 Wipe Frequency (Rewipe, Abrasion) Controls

Each CCA-treated test specimen on each minideck included two sampling areas: a PSA (suffixed "M") and an adjacent baseline sampled area (suffixed "BL"). BL areas were those that were initially wiped prior to coating to establish baseline DCCA. A

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subset of the BL areas were resampled during each sampling event. All of the BL areas on one of the three minidecks per coating (as well as the positive control minidecks) were sampled during each sampling event. Because there were three minidecks per coating, a given BL area was resampled every third sampling event. As such, the coatings on these sections of lumber were not abraded by wiping to the same extent as the coatings on the PSAs, so the effect that wiping has on coating efficacy could be investigated.

These results are presented in Section 3.1.6.

#### 2.13.4 Analytical (Contract Laboratory) Control Samples

A series of laboratory control samples were sent with each batch of samples tested by the subcontract analytical laboratory. Each set of digested wipe samples submitted to the subcontract analytical laboratory typically included 5 percent additional blind field blanks (extracted unused wet wipes), one blind blank (extraction fluid only), one set of three different concentration-spiked samples, and duplicates (split samples) for 5 percent of the wipe sample digestates that were analyzed to assess laboratory performance. Control samples were not identified as such to the contract laboratory performing the analyses. For example, assuming that a total of 200 wipe samples were collected and shipped to the subcontract laboratory in a single batch, the following additional samples were to be included:

- Ten (10) field blank samples prepared by taking unused wetted wipes and extracting them in accordance with the procedures previously specified
- One (1) blank consisting of extraction fluid only
- One (1) digestion fluid sample spiked to 1.0 µg/L (0.015 µg in 15 mL digestion fluid) with As, Cr, and Cu
- One (1) digestion fluid samples spiked to 50 µg/L (0.75 µg in 15 mL digestion fluid) with As, Cr, and Cu
- One (1) digestion fluid samples spiked to 1000 µg/L (15 µg in 15 mL digestion fluid) with As, Cr, and Cu
- Ten (10) duplicates (selected split samples of digested wipes from actual samples generated)

Furthermore, the subcontract laboratory analyzed project-specific postdigestion spiked samples for each analyte, as well as equipment blanks run on each batch of samples. Results of the laboratory control samples are discussed in Section 4.2.

#### 2.14 Paint Chip Sampling

The chemical composition of paint fragments from the painted minidecks was analyzed to determine whether exposure to paint chips, dust, etc. may present another exposure pathway to CCA. For example, when consumers are preparing their decks for recoating with paintings, they may be exposed to paint chips and dust resulting from the sanding and scraping of old weathered paint. This could be more of a health risk than dermal exposure to DCCA analytes. A separate QAPP was prepared for the paint chip sampling and analysis.

Testing involved collecting samples of chipping/peeling paint from the minidecks coated with paint using cleaned tweezers. These decks were identified with the prefixes 9- and 10-, as they correspond to coatings #9, a water-based (latex) paint, and #10, an oil-based (enamel) paint. For both paints, the wood surfaces were coated with a commonly-available primer prior to coating with the paints themselves. Samples were taken from each of the CCA-treated boards of each of the three minidecks coated with the given paint and composited into a single sample, homogenized, subsampled, weighed, digested and analyzed. The same general procedure was used for the untreated boards from the three minidecks coated with the given paint. For each composite sample, the extraction fluid was split so that duplicate analyses for total arsenic, total chromium, and total copper could be run by the subcontract analytical laboratory. Table 2-11 lists the samples that were analyzed.

**Table 2-11. Composite Samples Taken for Paint Chip Sampling/Analysis**

Coating #	Boards to Sample	# of Minidecks	# of Boards (total)	Type of Sample	Number of Samples
9	CCA-Treated	3	12	Composite	Duplicate
9	Untreated	3	15	Composite	Duplicate
10	CCA-Treated	3	12	Composite	Duplicate
10	Untreated	3	15	Composite	Duplicate

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**Evaluation of the Effectiveness  
of Coatings in Reducing  
Dislodgeable Arsenic,  
Chromium, and Copper from  
CCA Treated Wood**

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Plastic tweezers were decontaminated prior to sampling and in between each sampling event. Equal amounts of paint fragments were removed from each of the five untreated wood boards on each of the three coating #9 minidecks. Individual paint fragment samples were taken from various places on each board to ensure a representative sample. All paint fragments for a given sample were added to a single digitube which had been preweighed in the laboratory prior to sampling. A total paint mass of 1.5 grams was targeted. Sampling was repeated accordingly for the five untreated wood boards on each of the three coating #10 minidecks, then the four CCA-treated wood boards on each of the three coating #9 minidecks and lastly, the four CCA-treated wood boards on each of the three coating #10 minidecks. From each digitube, 0.5 gram of paint fragments was removed and placed in another digitube. The balance of the paint fragments (~1.0 gram) was left in each of the original digitubes. The Digitubes were weighed/reweighed to determine the mass of paint in each sample. The contents of each of the eight digitubes were then digested in their entirety, in accordance with the procedures specified in Section 2.9. Each of the 1.0 gram composite sample digestates were split into two equal duplicate samples and shipped to the subcontract analytical laboratory for determination of total arsenic, chromium, and copper in accordance with the procedures in Section 2.9.

Comparison of the 0.5 and 1.0 gram paint fragment digestate analysis results were used to help determine if there was any impact on analysis by the paint matrix itself.

These results are presented in Section 3.5.

### 3. Study Results

#### 3.1 Source Characterization and Sampling Events

##### 3.1.1 Distribution of Baseline Data

Overall baseline wipe sample variability is relatively high (around 50 percent RSD). Intraboard variability (that is, variability between sampling area DAs along a given board) likewise vary: some are relatively low (5-20 percent RSD) while others are quite high (>50 percent RSD). While it's overall (interboard) variability was relatively high, the newer C deck appears to have significantly lower intraboard variability than the A source deck.

Figures 3-1 through 3-6 provide simple box plots of the distribution of baseline data. These figures are grouped by analyte and source, and plot coating on the x-axis versus baseline DCCA on the y-axis. Appendix I includes similar plots grouped by board instead of by coating.

In all of the box plots presented, the box stretches from the lower hinge (defined as the 25<sup>th</sup> percentile) to the upper hinge (defined as the 75<sup>th</sup> percentile) and therefore contains the middle half of the scores in the distribution. The median is shown as a line across the box. Therefore, ¼ of the distribution is between this line and the top of the box and ¼ of the distribution is between this line and the bottom of the box. The plus symbol (+) shows the mean. In these plots, the bars on either side of the box define the minimum and maximum.

The baseline data were visually inspected for positional trends and correlations in an effort to gain insight into the validity and utility of the method by which precoat baseline values were determined for this study. Positional trend meaning the tendency for baseline measurements to be related to position along the board (position was measured from one end of the board). For example, do baseline measurements from the center of a board differ systematically from those near the ends of the board. Positional correlation is the tendency for baseline measurements close in position on the same board to have stronger correlation than baseline measurements from distant positions.

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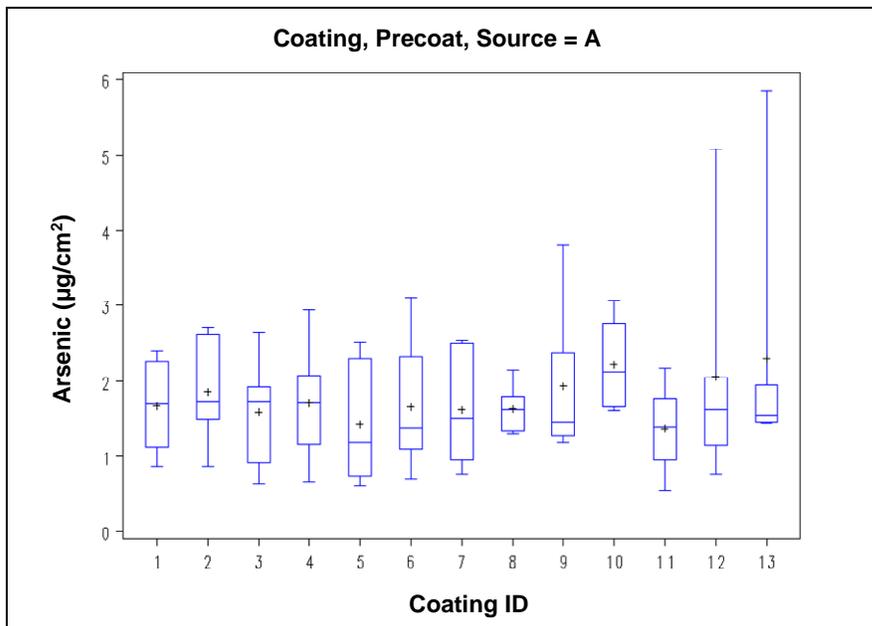


Figure 3-1. Box Plot, Baseline DAs, by Coating, Source A

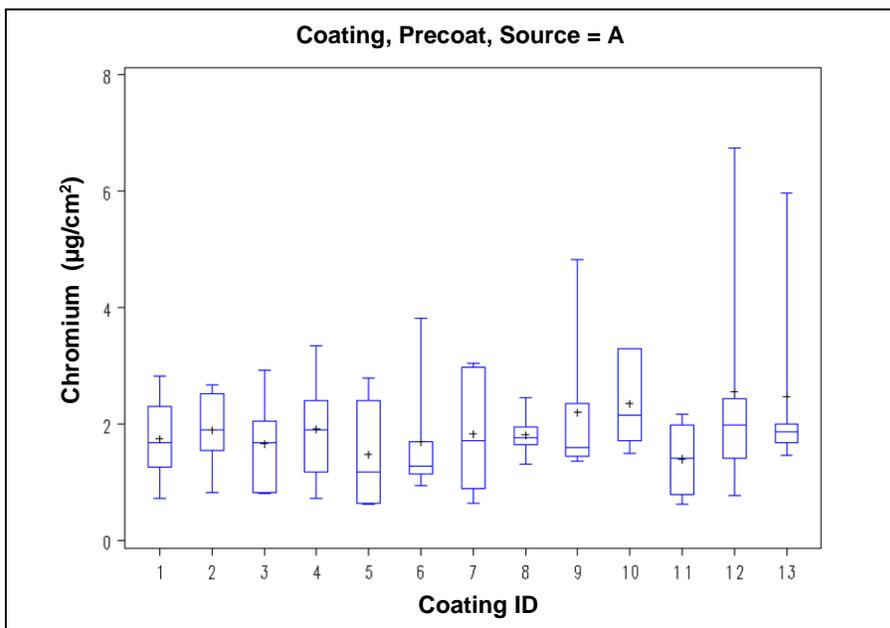


Figure 3-2. Box Plot, Baseline DCr, by Coating, Source A

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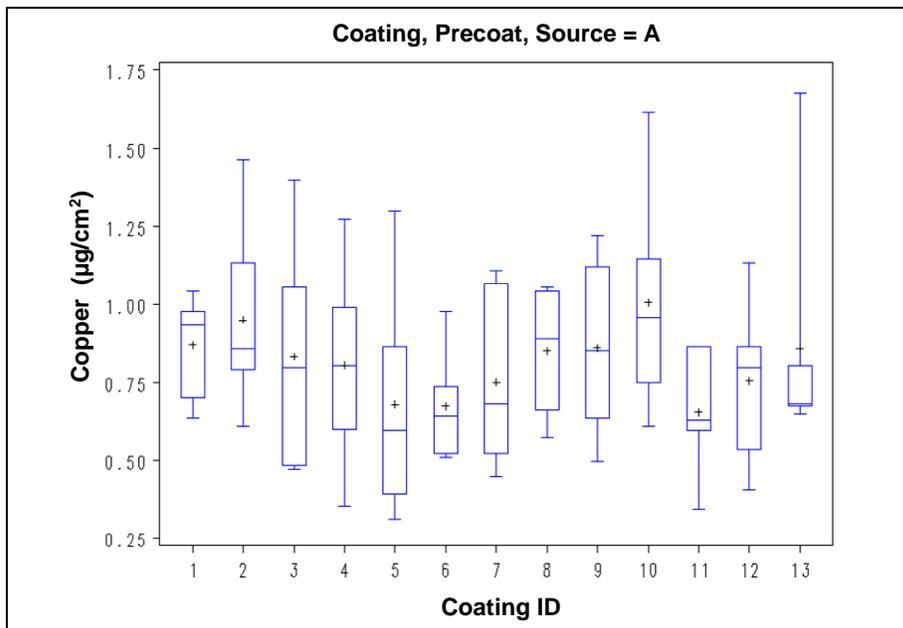


Figure 3-3. Box Plot, Baseline DCu, by Coating, Source A

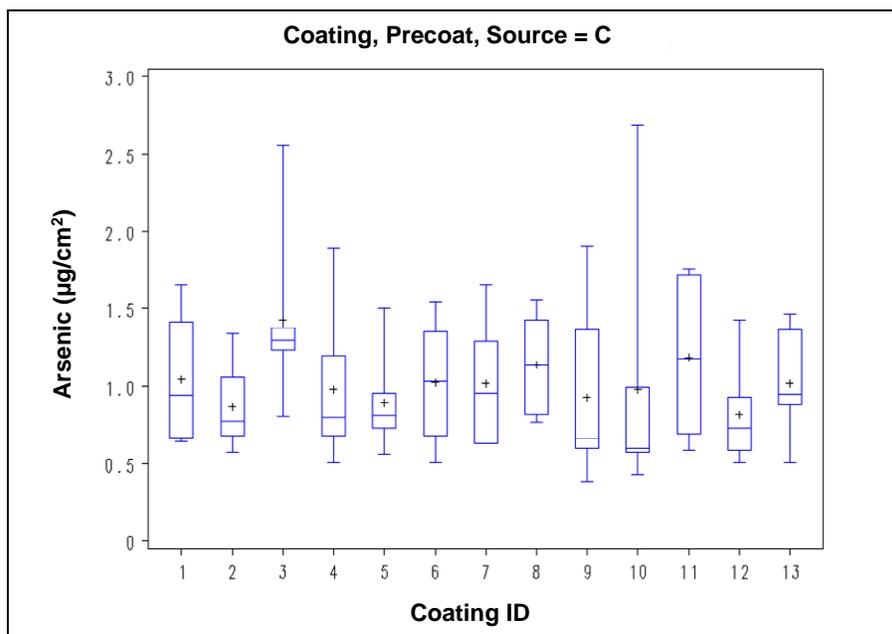


Figure 3-4. Box Plot, Baseline DAs, by Coating, Source C

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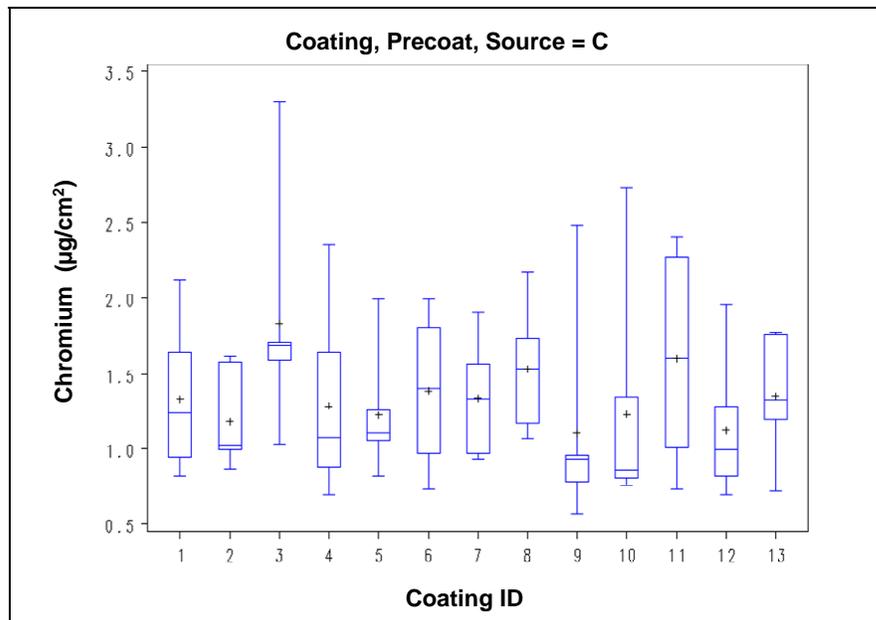


Figure 3-5. Box Plot, Baseline DCr, by Coating, Source C

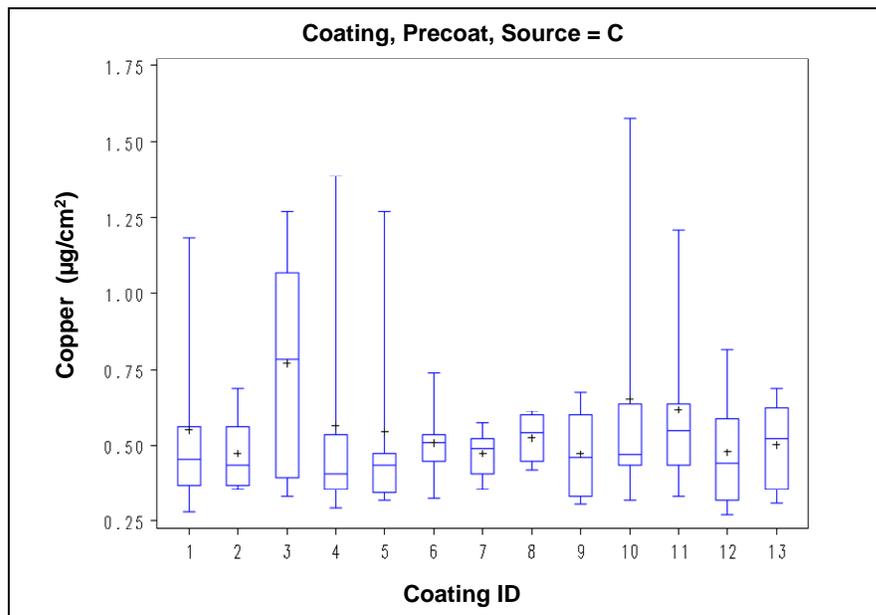


Figure 3-6. Box Plot, Baseline DCu, by Coating, Source C

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### 3.1.2 Wood Core Sample Data

The wood core data for source A and source C are summarized in Tables A-5 and A-6 in Appendix A. The tables show averages, standard deviations, and RSD for each board and include summary statistics at the end of each table for each source (A and C). RSDs should not be interpreted as indicators of data quality, but rather as indicators of natural variability in CCA retention within and between boards. Note that some of the boards listed in the tables below were not actually used in the construction of the minidecks; however, they are included here for completeness. Complete data showing the results of each individual measurement are provided in Appendix J.

The overall average results reported as elemental solid-phase concentrations, generally compare favorably with the expected ratio of concentrations of CCA analytes calculated in Section 2.2.3. There are some wood core sample data points that are clear outliers and overall variability is relatively high at about 50 percent RSD. Additionally, because of the way that boards are cut from the tree, taking core samples from the wide face – as done in this study – increased the likelihood that heartwood will be sampled. If the narrow faces had been sampled, sapwood would have more consistently been sampled and the values would have likely been more consistently high and less variable. A summary of the nominal (ideal), source A and source C CCA actives composition is provided in Table 3-1.

### 3.1.3 Core Sample-Baseline DCCA Correlation Analysis

Average baseline DCCA was plotted versus average wood core sample concentration for each board for which both averages were available. The plots are presented as Figures 3-7 and 3-8 for the A and C source boards, respectively. There may be some correlation between high DCCA and high core concentrations, particularly at the higher concentrations and for source A. Note that CCA analyte concentrations are plotted as elemental concentrations in Figures 3-7 and 3-8.

Table 3-1. Comparison of Nominal, Source A, and Source C CCA Actives Composition

Table 3-1			
	Nominal CCA	Source A	Source C
As (mg/kg)		1,645	2,075
As as As <sub>2</sub> O <sub>5</sub> (mg/kg)	4,255	2,522	3,182
As <sub>2</sub> O <sub>5</sub> (percent)	34.0 (30.0 – 38.0)	31.7	35.2
Cr (mg/kg)		2,045	2,095
Cr as CrO <sub>3</sub> (mg/kg)	5,938	3,933	4,029
CrO <sub>3</sub> (percent)	47.5 (44.5–50.5)	49.4	44.5
Cu (mg/kg)		1,203	1,465
Cu as CuO (mg/kg)	2,317	1,506	1,834
CuO (percent)	18.5 (17.0–21.0)	18.9	20.3

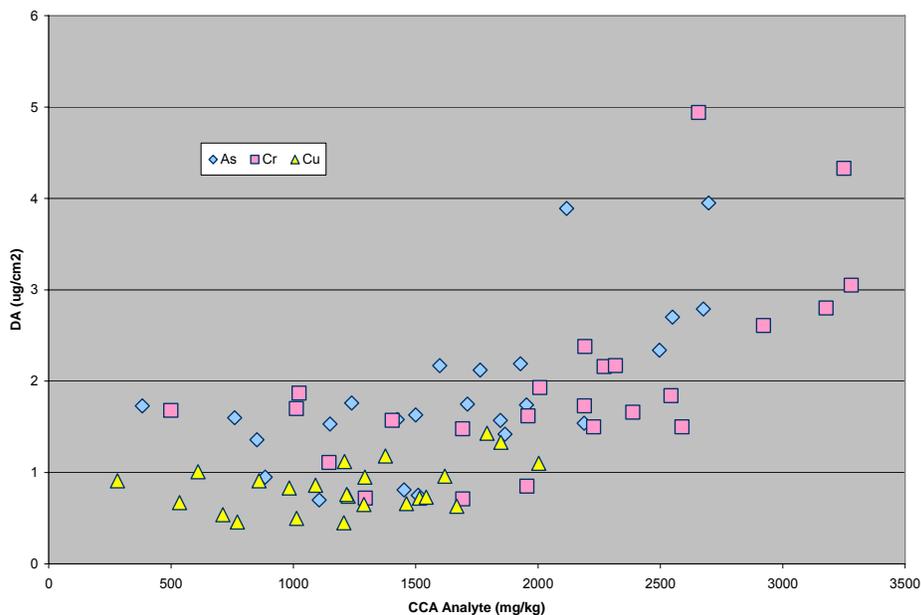


Figure 3-7. Wood Core Concentration vs. Baseline DCCA for Source A Boards

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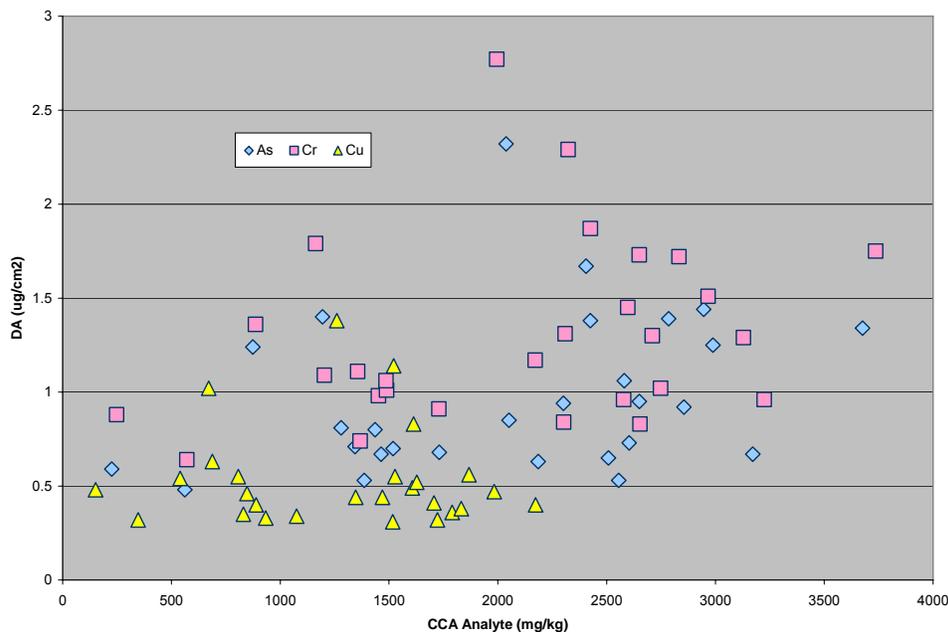


Figure 3-8. Wood Core Concentration vs. Baseline DCCA for Source C Boards

### 3.1.4 Cross-Contamination Control and Negative Control Deck Sample Results Data

The untreated specimens separating the CCA-treated test specimens on each deck served as sources for cross-contamination control samples. During each wipe sampling event, one untreated (but coated, for minidecks 1 - 12) specimen from each minideck was wipe-sampled. Because there were five untreated specimens on each minideck, there were a total of ten potential sampling areas. The specific areas sampled during each routine sampling event were randomly selected for each minideck and were different for each sampling event. The results of these samples were used to assess the level of cross-contamination for adjacent samples as a result of splash-over of rainwater from one specimen to the next and possibly other transfer mechanisms.

Cross-contamination potential was assessed by comparing the cross-contamination results for the thirteen minidecks (the uncoated control boards) versus the results from the non-CCA control minideck. These results are summarized in Table 3-2, while the full dataset is provided in Appendix K.

**Table 3-2. Summary of Cross-Contamination and Blank Control Minideck Results**

Non-CCA Minideck	DAs ( $\mu\text{g}/\text{cm}^2$ )	DCr ( $\mu\text{g}/\text{cm}^2$ )	DCu ( $\mu\text{g}/\text{cm}^2$ )
Average	0.001	0.003	0.034
Std. Dev.	0.0006	0.004	0.011
<b>Non-CCA Boards on Minidecks</b>			
Average	0.007	0.009	0.056
Std. Dev.	0.021	0.028	0.042

The DAs results of all of the untreated wood wipe samples are very low in comparison with those from the treated wood specimens, although there appears to be more DAs from the cross-contamination controls versus the blank minideck controls. The cross-contamination data for all of the coatings are summarized in Appendix A (Table A-7), while the full dataset is provided in Appendix K: Coating Volume and Mass Data.

Figure 3-9 shows the total volume and mass of each coating applied sorted by coating ID. A complete tabulated summary of the coating application data is included in Appendix A (Table A-8). The summary includes average, standard deviation, and RSD for each combination of board type and coating. RSDs should not be interpreted as indicators of data quality, but rather as indicators of the variability inherent in the coating methods utilized. Any variability in coating volume or mass among decks is part of the natural variability one would see in practice, on real decks being coated by homeowners. Film thickness proved to be quite difficult to measure on wood substrates and was not appropriate for non-film forming coatings; as such, film thicknesses were only measured on some minidecks and these data are not presented.

### 3.1.5 Negative Control Minideck Results

Atmospheric deposition of CCA analytes were insignificant in relation to the DCCA measurements used in the coating efficacy data analysis. The full dataset is provided in Appendix L.

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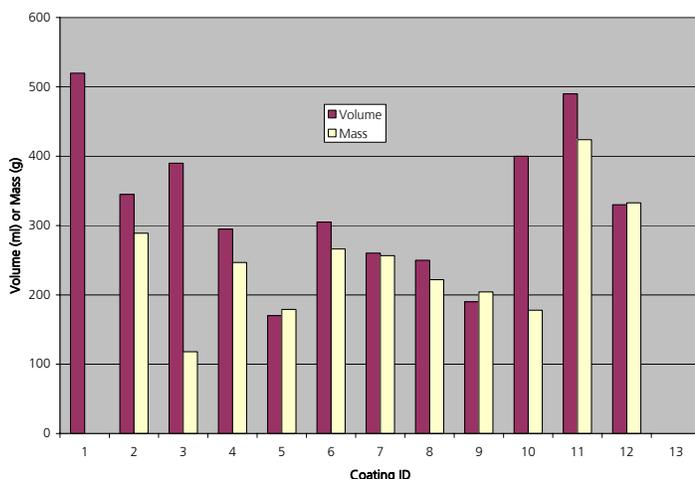


Figure 3-9. Coating Application (total of triplicate minidecks on both A and C sources)

### 3.1.6 Wipe Frequency and Abrasion Results

Although the experiment was not specifically designed to provide detailed information about the effects of wipe frequency (rewipe effect) and the number of previous wipes (abrasion), the data collected provide a limited opportunity to investigate these factors. To this end two predictive variables were defined for the purpose of quantifying the amount of previous wiping and the elapsed time since the previous wipe:

NOPW = number of previous wipes

TSPW = time (months) since the previous wipe

NOPW is a surrogate for total prior postcoat abrasion; and TSPW measures wipe frequency, or more specifically, the time interval between wipe samples on a given sampling area. Possible effects of these two factors were investigated using the combined dataset [i.e., both the sample (M) and baseline (BL) DCCA results], though it must be noted that the BL samples were only taken through 11 months postcoating. An earlier statistical model that utilized standard analysis-of-variance methods and was employed for preliminary data analysis in an interim data report for this project was augmented with the linear variables NOPW and TSPW. The model facilitated the investigation of trends (either increasing or decreasing) in NOPW and TSPW after adjusting for all of the other factors. Because the experiment was not designed specifically for the purpose of assessing abrasion and wipe-frequency effects,

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however, the information in the data for assessing these factors is limited and the analysis was intended primarily for exploratory purposes.

The statistical analysis indicated that there were no significant trends with the TSPW, but that there is evidence of a downward trend with NOPW. That is, the greater the NOPW, the lower the DCCA level. The direction of the trend is opposite what would be expected if wiping had abraded coatings and reduced their effectiveness (i.e., the hypothesis that more wiping would wear down the coating and allow more CCA analytes to permeate the worn-down, thinner coating).

This finding should be viewed with some skepticism. Because the experiment was not designed explicitly for assessing abrasion and wipe frequency, there is not much relevant information in the data, and it is difficult to claim with any certainty that the method of analysis used is the best way to use the data available for teasing out abrasion and rewipe information (from each other and from the other effects). Consequently the significant effect associated with NOPW could be due to confounding with other effects. Alternatively, it is possible that the amount of abrasion induced by rubbing the coating with a pad is overshadowed by the amount of cleaning (of "built-up" DCCA) of the surface. Thus the number of rewipes might be a surrogate for prior cleaning; the greater the prior cleaning, the less the measured DCCA.

### 3.2 Coating Performance Data

Tabulated summaries of project data for DAs, DCr and DCu is included in Appendix A (Tables A-9, A-10 and A-11, respectively). A complete set of wipe sampling data, including data for both the PSA (M) samples and baseline (BL) samples at each sampling event, is provided in Appendix M.

#### 3.2.1 DCCA vs. Time

Baseline and time series DCCA values for each CCA analyte, sorted by coating, and averaged over the combined A and C sources are shown graphically using a transformed DCCA scale on the y-axis in Figures 3-10 through 3-12. The tabulated data used to create these graphs is included Appendix A (Table A-9). Several observations can be made from these data. First, each coating, as well as the positive controls (uncoated minidecks), generally show a significant decrease in DCCA between baseline (precoat) and samples taken 1 month after coating. This suggests an impact on DCCA from rinsing or washing the minidecks. Second, the coated minidecks all have lower DCCA than the positive controls, which indicates that coating (using any

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of the coatings tested) mitigates DCCA to some degree. Third, over the course of this study, DCCA increases with time after coating, most likely due to the effects of weathering and possibly abrasion on the coating, although it should be noted that the uncoated positive controls show similar, though generally less pronounced, trends.

Note that confidence intervals are not shown on Figures 3-10 through 3-12 to avoid obscuring the general trends. Refer to Figures 3-30 and 3-31 for graphical representations that capture and show statistically significant differences between coatings by time and also Tables 3-7 and 3-8 that present p-values for differences between means at each time point and present estimated 95% confidence intervals for estimated percent reductions between coatings and the positive controls at each time period.

### 3.2.2 Graphical Data Analysis

The statistical methods used in this section are described in Section 1.3.

The total number of valid observations in the dataset is 1,090. The effects of interest are coating, grain orientation, source deck, and time, and thus there are up to four-way interactions that need to be considered.

The split-split-plot analysis of variance is very useful for identifying significant factor effects, but is less informative with regard to understanding the nature of the factor effects, especially those involving higher-order interactions. Therefore, graphical displays are extensively used to supplement the standard split-split-plot statistical analysis. The intent is to provide a graphical representation of the patterns and trends in the data that are indicated by the numerical analysis of variance.

The statistical analysis is started with a comprehensive graphical presentation and summary of the data. The intent is to provide an objective and transparent “look” at the experimental data unadulterated by any statistical model imposed on the data, and to aid the interpretation of the formal statistical analysis of variance results presented later in this section.

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Evaluation of the Effectiveness of Coatings in Reducing Dislodgeable Arsenic, Chromium, and Copper from CCA Treated Wood

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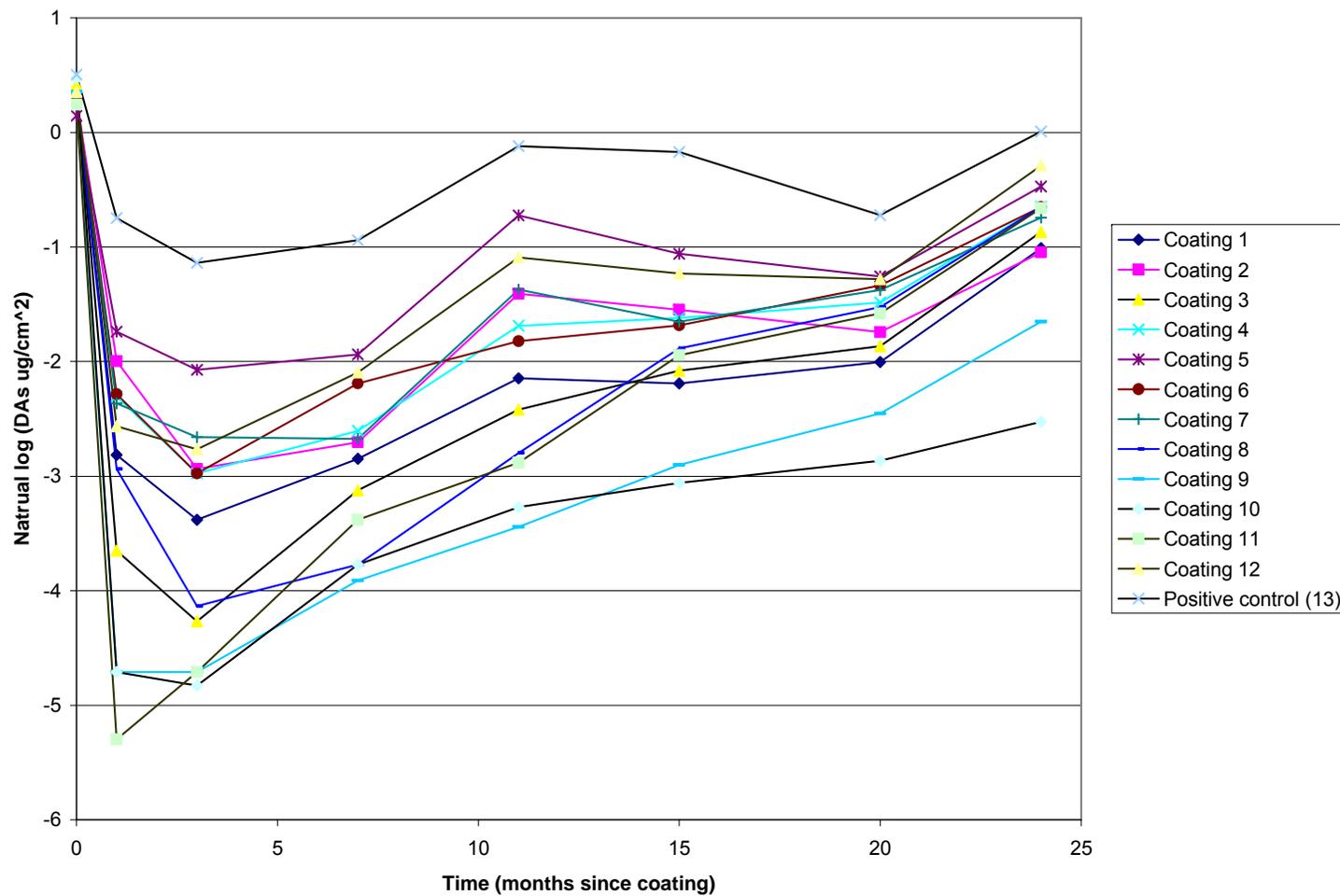


Figure 3-10. Average DAs vs. Time for All Coatings

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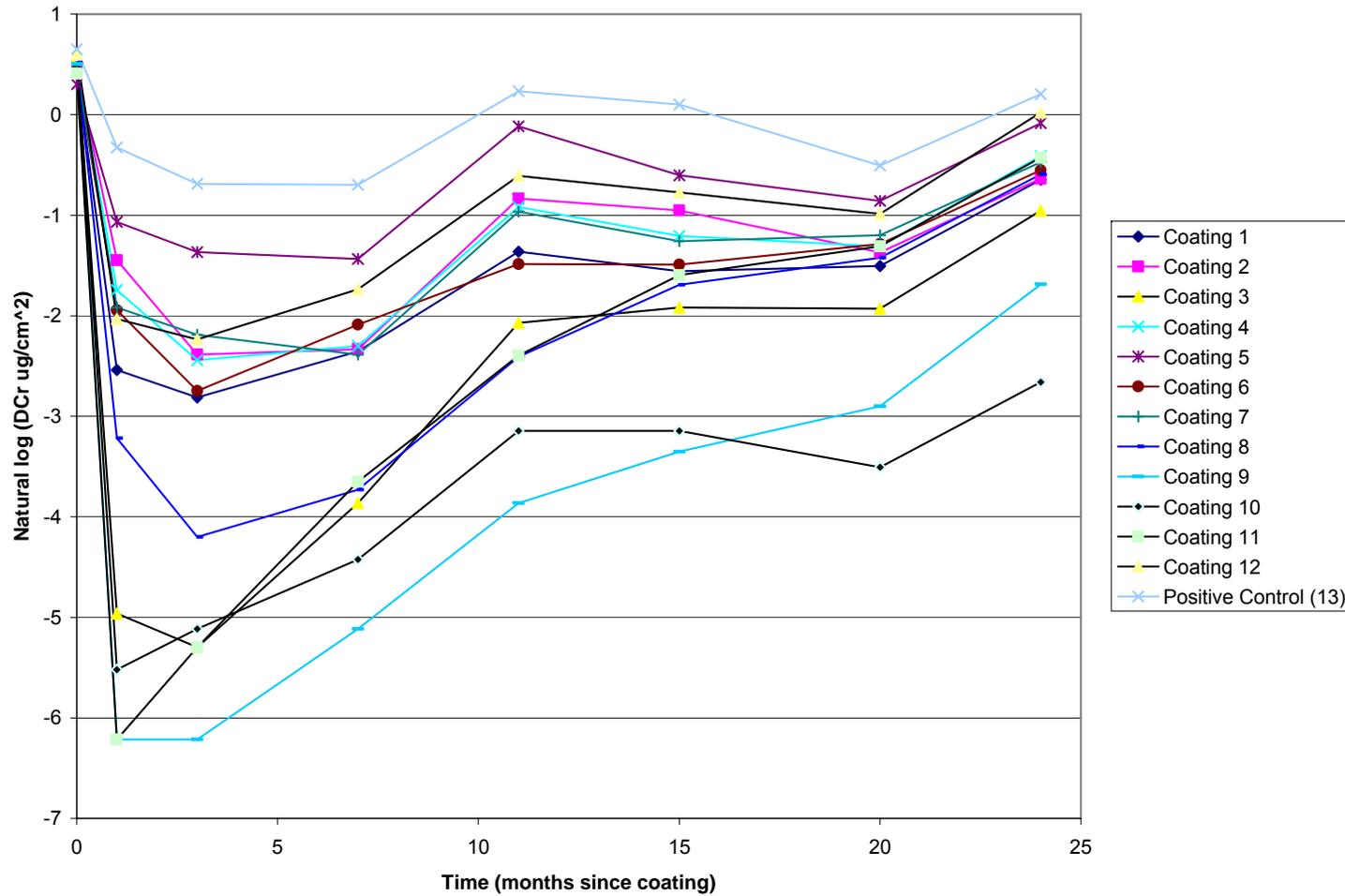


Figure 3-11. Average DCr vs. Time for All Coatings

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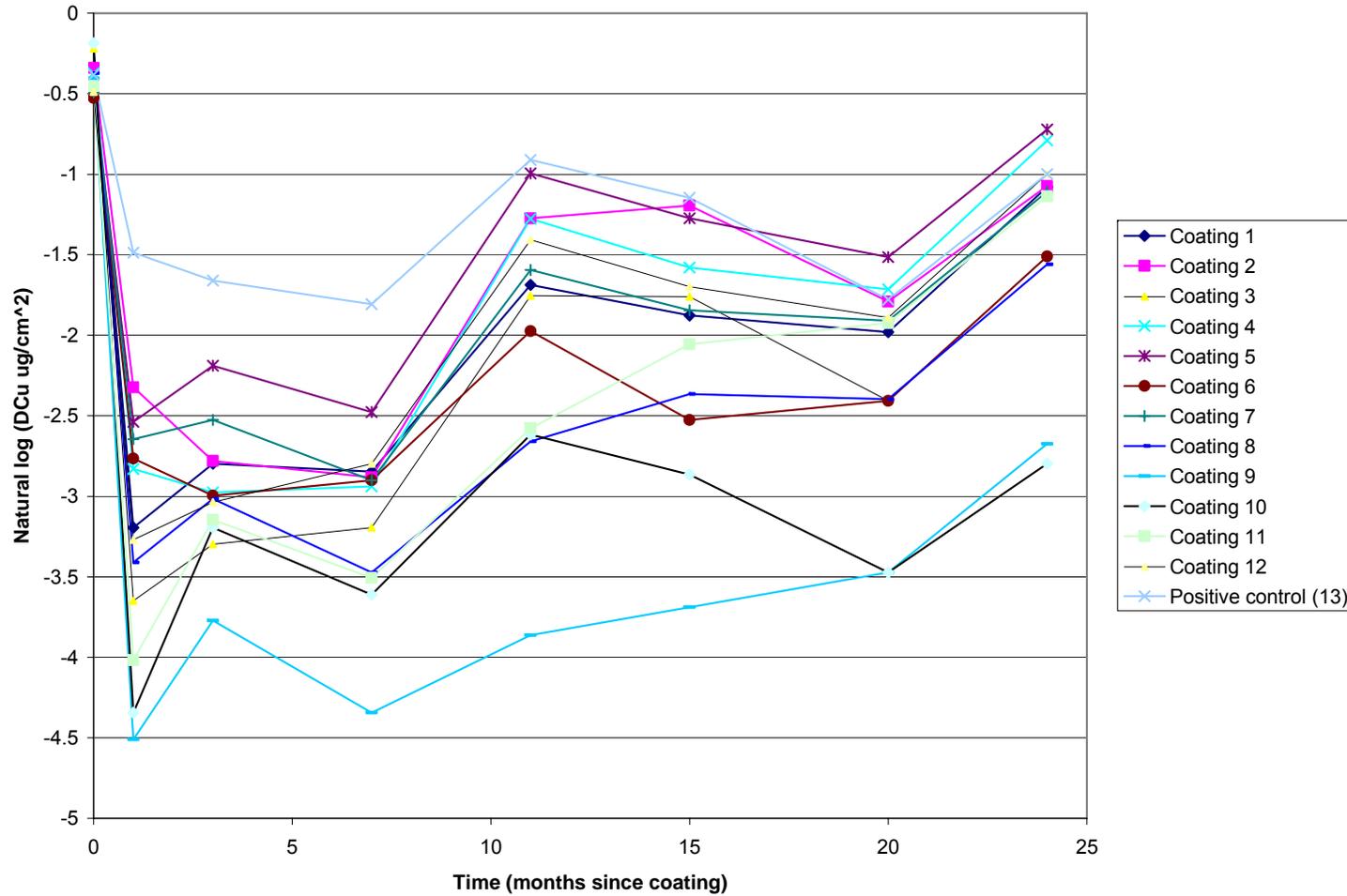


Figure 3-12. Average DCu vs. Time for All Coatings

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The experiment has several factors and a comprehensive graphical summary of the data, while very enlightening, requires an understanding of why and how certain graphical elements are constructed. First the data and the graphical display for a single minideck is described. A composite data display in which all of the data from the thirty-nine minidecks is displayed on a single page is then presented. This graphic and other composite data displays facilitate comparison among coatings. Next, plots of various averages designed to illustrate the effects of source deck, grain orientation, time and coating type are presented. Finally, a grand composite display that includes all of the relevant graphical elements on a single page is presented.

3.2.2.1 *Single Minideck Data Plot*

The data from Minideck 9-A is used as an illustration; refer to the accompanying figure and data tables in Figure 3-13. Minideck 9-A was chosen for illustration because individual data points are well spread out and easily identified; and because it is “typical” in the sense of manifesting certain patterns and trends that are common to several other minidecks.

Each minideck contains four CCA-treated boards, with each board having different source and grain characteristics: Source Deck A, Grain Up; Source Deck A, Grain Down; Source Deck C, Grain Up; and Source Deck C, Grain Down. The plots are color coded according to board source deck and grain orientation as shown in Table 3-3.

**Table 3-3. Legend for Individual Minideck Data Plots**

Color	Source	Grain Orientation
Dark Blue	A	Up
Green	C	Up
Cyan (Light Blue)	A	Down
Red	C	Down

Each board contributes eight measurements of DAs to the study: one baseline measurement and seven post baseline measurements at successive time points after treatment with one of the thirteen coatings (actually twelve coatings plus control). Thus each minideck plot displays thirty-two DAs measurements. However, the manner in which the measurements are plotted is different for baseline and post baseline measurements.

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Baseline (time = 0) measurements are plotted as horizontal lines extending the full width of the plot. Note that even though the baseline measurement is plotted over the entire length of the time axis, there is only one baseline measurement per board (at time = 0). The baseline values are plotted in this manner for two reasons. First, is that a purpose of the baseline measurements is to serve as the “before treatment” measurement for each of the “after treatment” measurements.

The post baseline measurements are plotted in time-order sequence with filled-circles of the color matching the board characteristics, as indicated in Table 3-4. The lines connecting the points are for clarity, and should not be misconstrued as to infer that DAs values necessarily changed linearly (or even smoothly) between successive time periods. Note, from Table 3-4, that the log-transformed baseline values (column Time = 0) are 1.090, 0.402, -0.291, and -0.740 for the A-Up, A-Down, C-Up, and C-Down boards respectively. These values are plotted in Figure 3-13 as the dark blue (Bl), cyan (Cy), green (Gr), and red (Re) horizontal lines respectively. The same color coding is used in plotting the post baseline measurements versus time. The log-transformed DAs values plotted in the graph are from the data table columns with headings Time = 1 through Time = 7. For example, for Board A-T-M3 (Source Deck A, Grain Up), the seven time-ordered, post baseline, log-transformed As values (-4.065, -3.976, -3.043, -2.917, -2.067, -1.967, -1.407) are plotted versus time with dark blue circles and connecting lines.

One purpose of the baseline measurements is to explain some of the variability in the post baseline measurements. A board with higher than average initial DAs measurements could reasonably be expected to have higher-than-average post baseline DAs measurements as well. Thus, in Figure 3-13, for Minideck 9-A, it can be seen that the Source Deck A boards had both higher baseline, and post baseline, measurements than did the Source Deck C boards, regardless of grain orientation or time period.

Note that the dark blue and cyan time plots (Source Deck A) overlap considerably and that the red and green time plots (Source Deck C) also overlap to a lesser extent. However, the blue and cyan curves lie entirely above the red and green curves. Thus, for this minideck, the observed differences in DAs measurements between grain orientation from the same source deck are less pronounced than differences between boards from different source decks.

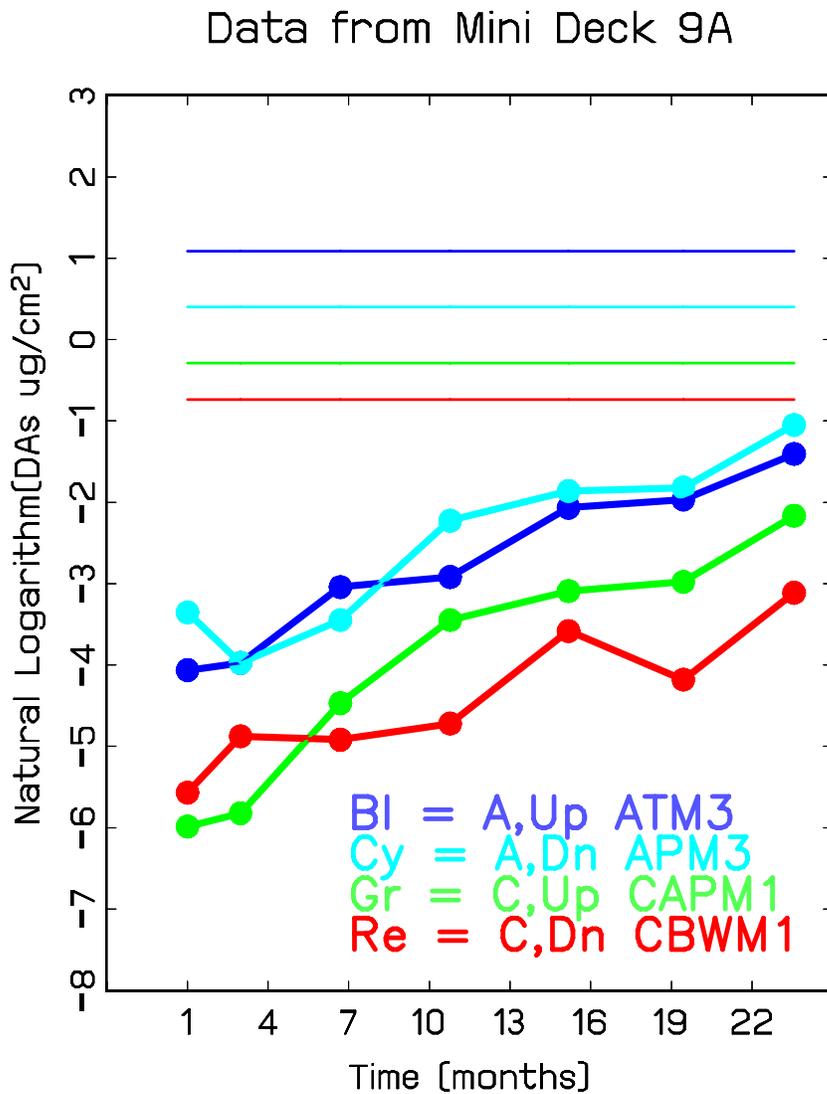


Figure 3-13. Data Plot for Minideck 9A

Table 3-4. DAs Data for Minideck 9-A, as used in Figure 3-13

Color	Spec ID	Source	Grain	Time = 0	Time = 1	Time = 2	Time = 3	Time = 4	Time = 5	Time = 6	Time = 7
<b>Raw (Not Natural Log Transformed) Data</b>											
Blue	A-T-M3	A	Up	2.974	0.017	0.019	0.048	0.054	0.127	0.140	0.245
Cyan	A-P-M3	A	Down	1.495	0.035	0.019	0.032	0.108	0.156	0.162	0.350
Green	C-AP-M1	C	Up	0.747	0.003	0.003	0.012	0.032	0.046	0.051	0.115
Red	C-BW-M1	C	Down	0.477	0.004	0.008	0.007	0.009	0.028	0.015	0.045
<b>Natural Log Transformed Data</b>											
Blue	A-T-M3	A	Up	1.090	-4.065	-3.976	-3.043	-2.917	-2.067	-1.967	-1.407
Cyan	A-P-M3	A	Down	0.402	-3.353	-3.976	-3.448	-2.224	-1.861	-1.819	-1.050
Green	C-AP-M1	C	Up	-0.291	-5.987	-5.823	-4.470	-3.448	-3.090	-2.978	-2.167
Red	C-BW-M1	C	Down	-0.740	-5.568	-4.876	-4.919	-4.721	-3.583	-4.182	-3.112

Finally, the most noticeable feature of this graph is the increasing trend in DAs measurements over time, regardless of board type. With the exception of one point from the “A-Down” board (Cy = cyan-colored line), the time plots are strictly increasing with time. DAs measurements are lowest at the first post baseline measurement and highest at the final post baseline measurement for each board type. Yet the final post baseline measurement is still less than the corresponding baseline DAs measurement.

### 3.2.2.2 Data Plots for Other Minidecks

Plots were constructed for all thirty-nine minidecks in the same fashion as Figure 3-13. These plots are shown as Figures 3-14 through 3-26. These enable detailed visual inspection of the data for individual minidecks. Full page figures of each minideck can be found in Appendix N.

### 3.2.2.3 All Thirty-nine Minidecks Plotted Together

A composite graphic displaying all of the data facilitates comparisons among coatings and also illustrates the repeatability of the experimental method which bears on the experimental method’s validity.

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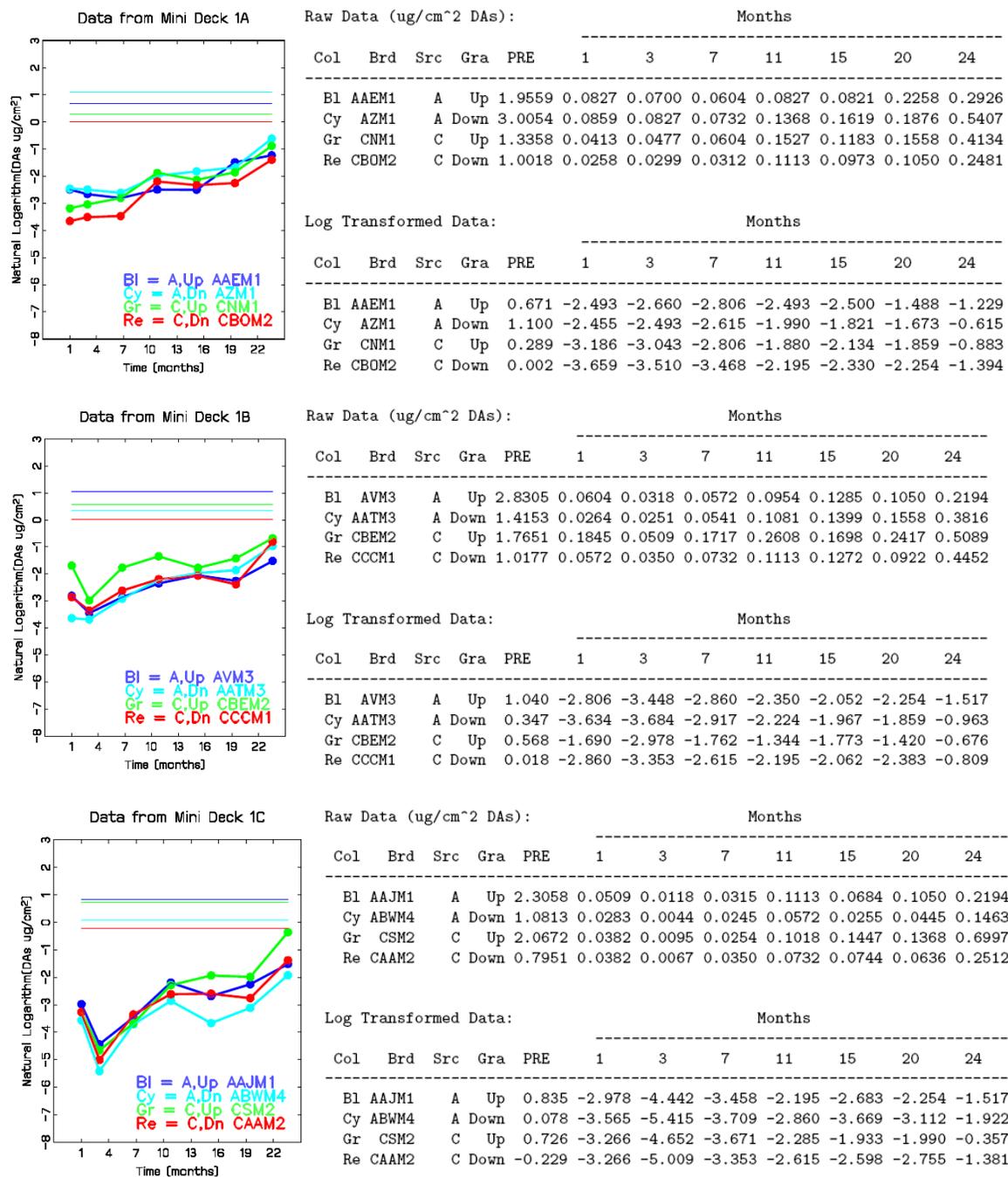


Figure 3-14. Data from Minideck 1A, Data from Minideck 1B, Data from Minideck 1C

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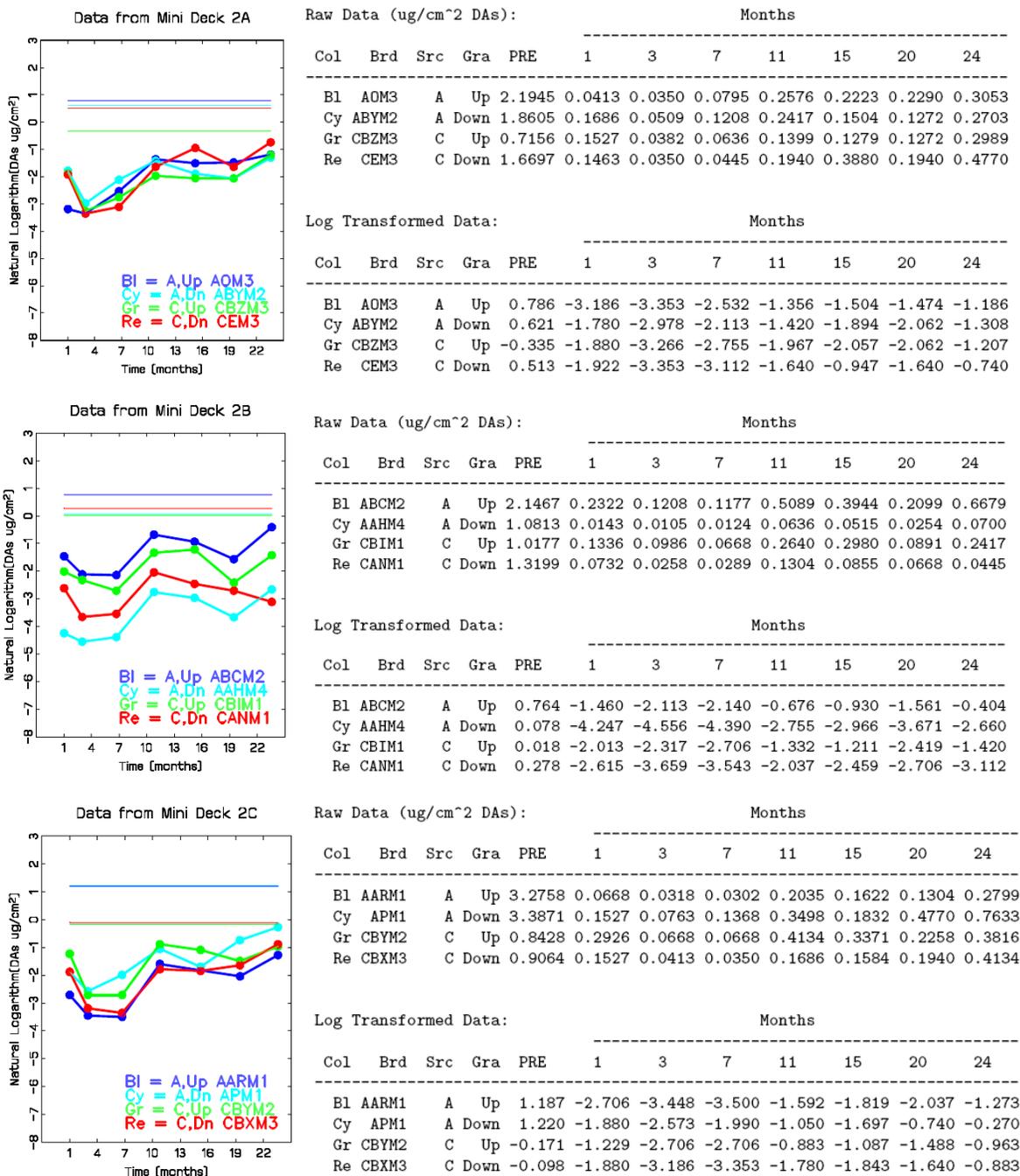


Figure 3-15. Data from Minideck 2A, Data from Minideck 2B, Data from Minideck 2C

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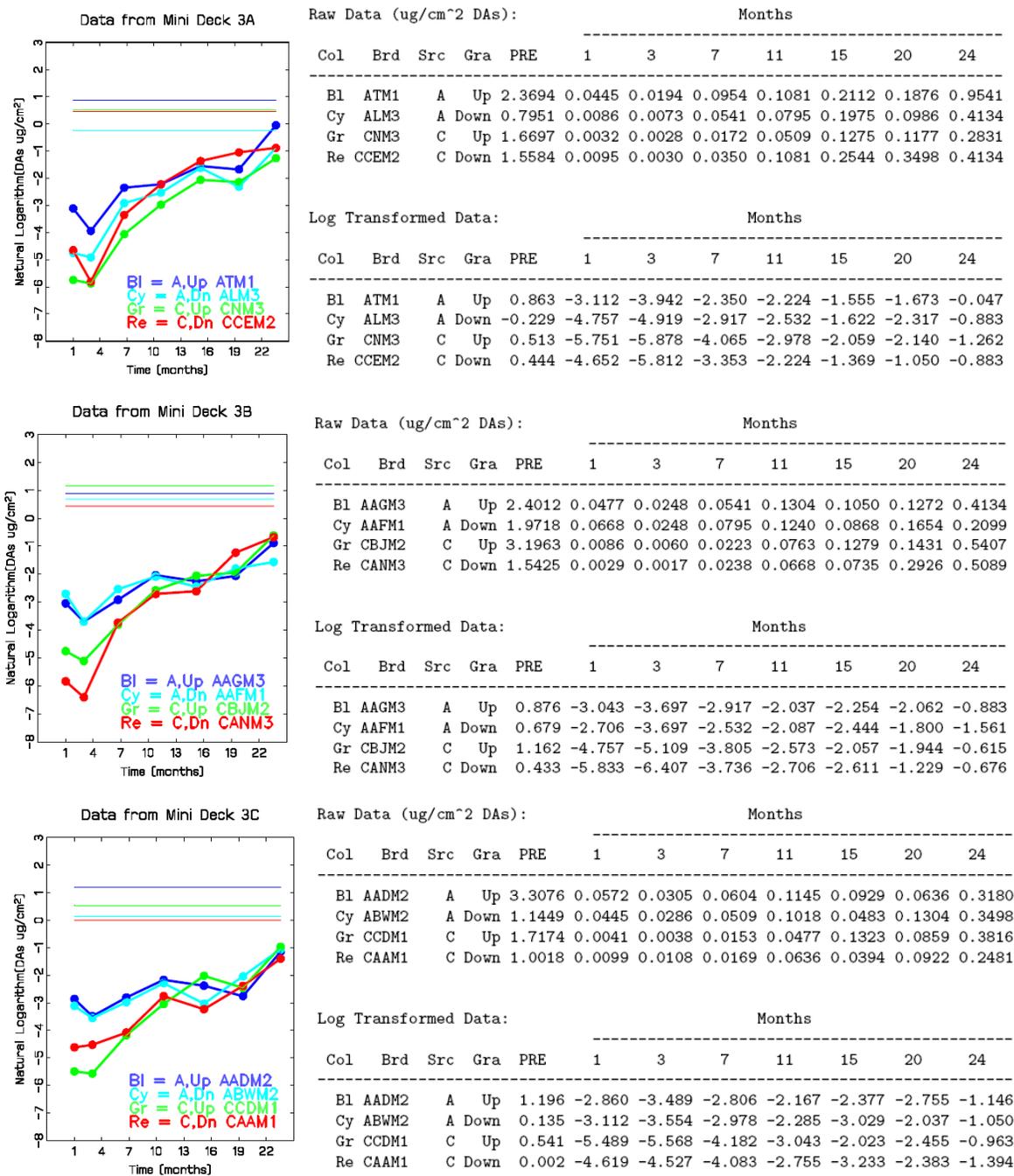
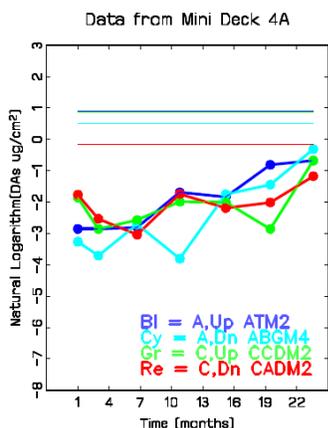


Figure 3-16. Data from Minideck 3A, Data from Minideck 3B, Data from Minideck 3C

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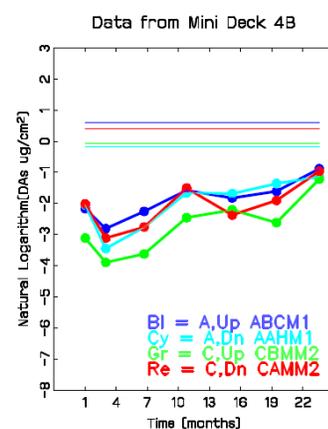


Raw Data (ug/cm<sup>2</sup> DAs):

Col	Brd	Src	Gra	PRE	Months							
					1	3	7	11	15	20	24	
Bl	ATM2	A	Up		2.4648	0.0572	0.0572	0.0604	0.1845	0.1600	0.4452	0.5089
Cy	ABGM4	A	Down		1.6379	0.0382	0.0245	0.0668	0.0223	0.1730	0.2354	0.7315
Gr	CCDM2	C	Up		2.3694	0.1558	0.0572	0.0763	0.1368	0.1349	0.0572	0.5089
Re	CADM2	C	Down		0.8428	0.1717	0.0795	0.0477	0.1749	0.1119	0.1336	0.3085

Log Transformed Data:

Col	Brd	Src	Gra	PRE	Months							
					1	3	7	11	15	20	24	
Bl	ATM2	A	Up		0.902	-2.860	-2.860	-2.806	-1.690	-1.833	-0.809	-0.676
Cy	ABGM4	A	Down		0.493	-3.266	-3.709	-2.706	-3.805	-1.754	-1.447	-0.313
Gr	CCDM2	C	Up		0.863	-1.859	-2.860	-2.573	-1.990	-2.004	-2.860	-0.676
Re	CADM2	C	Down		-0.171	-1.762	-2.532	-3.043	-1.743	-2.190	-2.013	-1.176

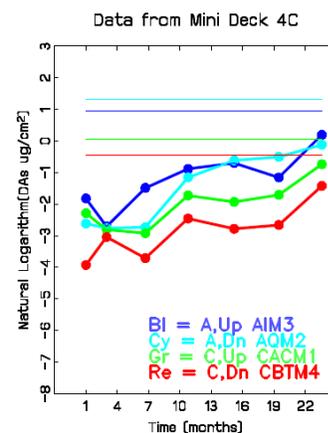


Raw Data (ug/cm<sup>2</sup> DAs):

Col	Brd	Src	Gra	PRE	Months							
					1	3	7	11	15	20	24	
Bl	ABCM1	A	Up		1.8446	0.1145	0.0604	0.1050	0.2067	0.1622	0.2004	0.4134
Cy	AAHM1	A	Down		0.8269	0.1304	0.0318	0.0636	0.1908	0.1857	0.2576	0.3180
Gr	CBMM2	C	Up		0.9382	0.0445	0.0204	0.0267	0.0859	0.1107	0.0732	0.2958
Re	CAMM2	C	Down		1.4948	0.1336	0.0445	0.0636	0.2226	0.0935	0.1495	0.3816

Log Transformed Data:

Col	Brd	Src	Gra	PRE	Months							
					1	3	7	11	15	20	24	
Bl	ABCM1	A	Up		0.612	-2.167	-2.806	-2.254	-1.576	-1.819	-1.608	-0.883
Cy	AAHM1	A	Down		-0.190	-2.037	-3.448	-2.755	-1.656	-1.683	-1.356	-1.146
Gr	CBMM2	C	Up		-0.064	-3.112	-3.895	-3.622	-2.455	-2.201	-2.615	-1.218
Re	CAMM2	C	Down		0.402	-2.013	-3.112	-2.755	-1.502	-2.370	-1.901	-0.963



Raw Data (ug/cm<sup>2</sup> DAs):

Col	Brd	Src	Gra	PRE	Months							
					1	3	7	11	15	20	24	
Bl	AIM3	A	Up		2.5920	0.1622	0.0668	0.2258	0.4134	0.4961	0.3149	1.2085
Cy	AQM2	A	Down		3.6733	0.0732	0.0636	0.0652	0.3149	0.5407	0.6043	0.8905
Gr	CACM1	C	Up		1.0495	0.1018	0.0604	0.0541	0.1781	0.1453	0.1813	0.4770
Re	CBTM4	C	Down		0.6361	0.0197	0.0477	0.0245	0.0859	0.0620	0.0700	0.2417

Log Transformed Data:

Col	Brd	Src	Gra	PRE	Months							
					1	3	7	11	15	20	24	
Bl	AIM3	A	Up		0.952	-1.819	-2.706	-1.488	-0.883	-0.701	-1.156	0.189
Cy	AQM2	A	Down		1.301	-2.615	-2.755	-2.730	-1.156	-0.615	-0.504	-0.116
Gr	CACM1	C	Up		0.048	-2.285	-2.806	-2.917	-1.725	-1.929	-1.708	-0.740
Re	CBTM4	C	Down		-0.452	-3.926	-3.043	-3.709	-2.455	-2.780	-2.660	-1.420

Figure 3-17. Data from Minideck 4A, Data from Minideck 4B, Data from Minideck 4C

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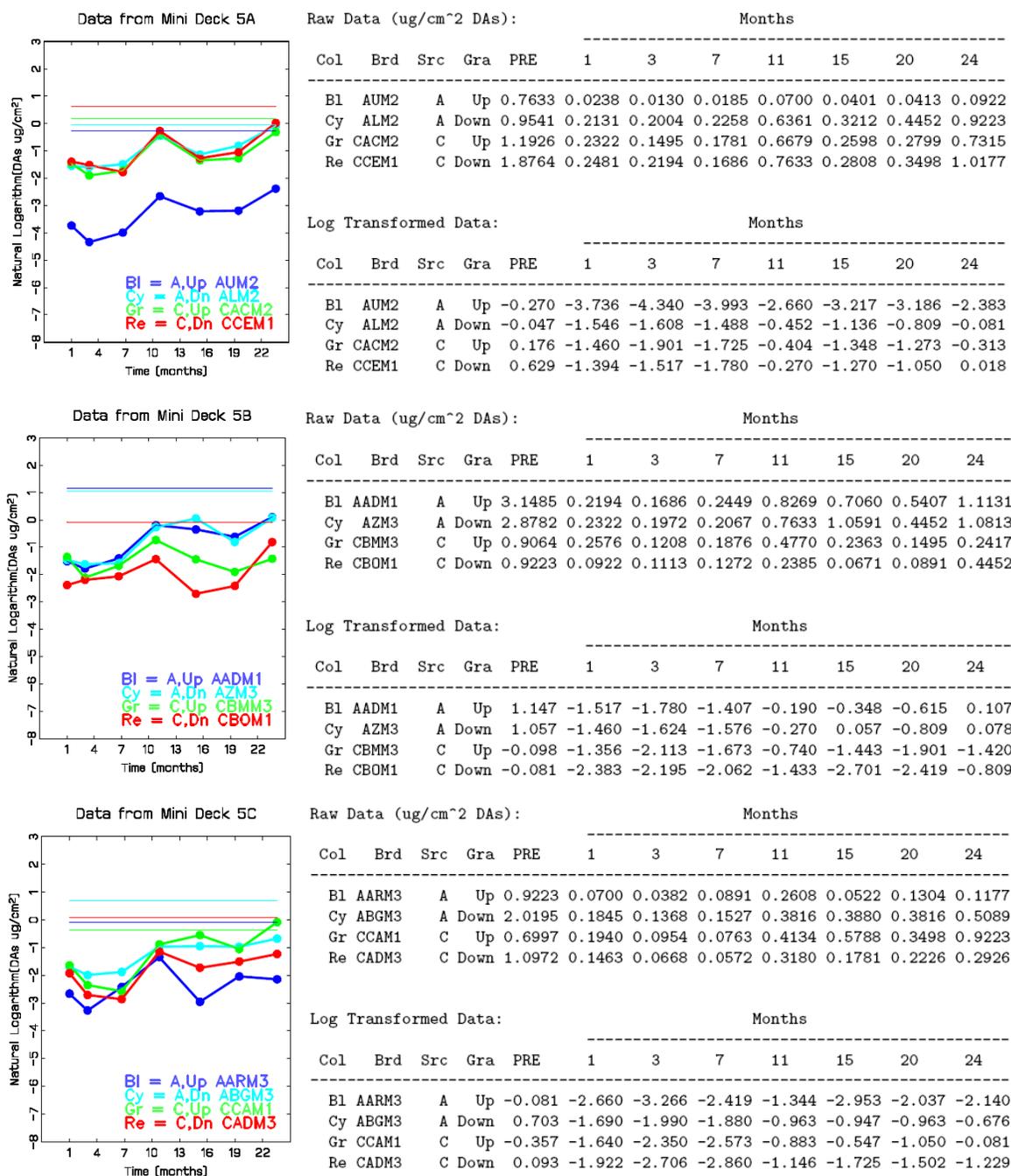


Figure 3-18. Data from Minideck 5A, Data from Minideck 5B, Data from Minideck 5C

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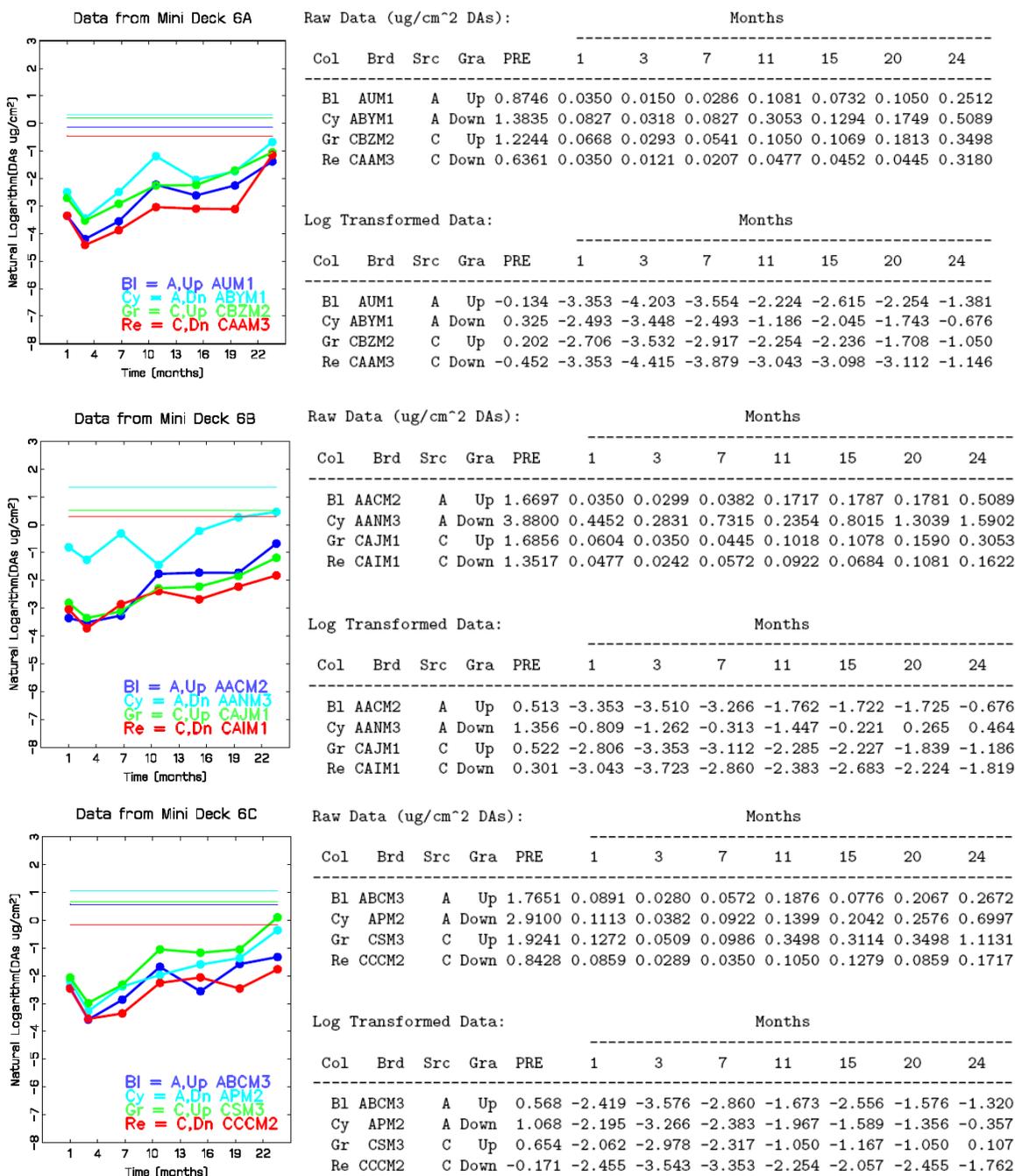


Figure 3-19. Data from Minideck 6A, Data from Minideck 6B, Data from Minideck 6C

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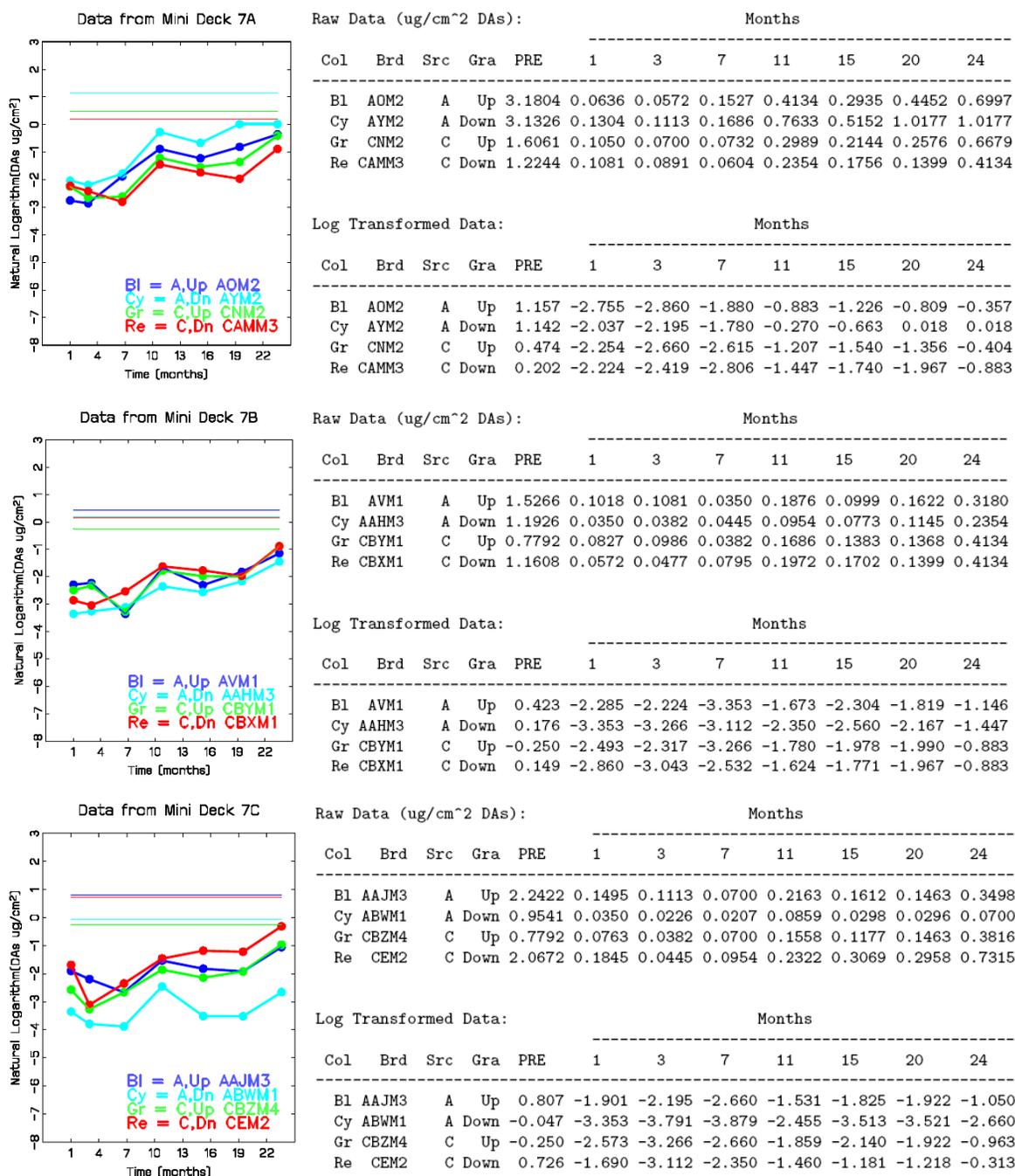


Figure 3-20. Data from Minideck 7A, Data from Minideck 7B, Data from Minideck 7C

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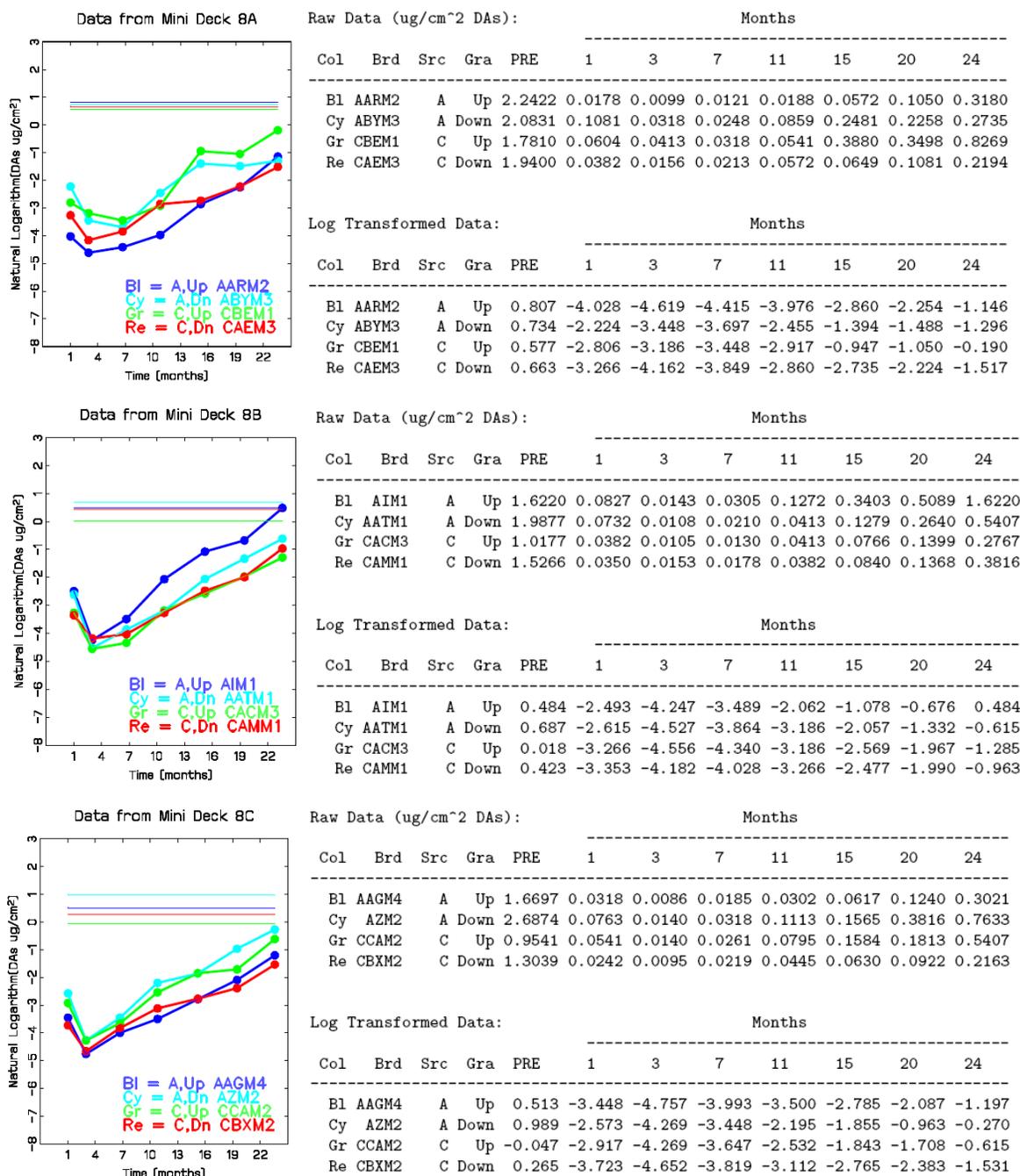


Figure 3-21. Data from Minideck 8A, Data from Minideck 8B, Data from Minideck 8C

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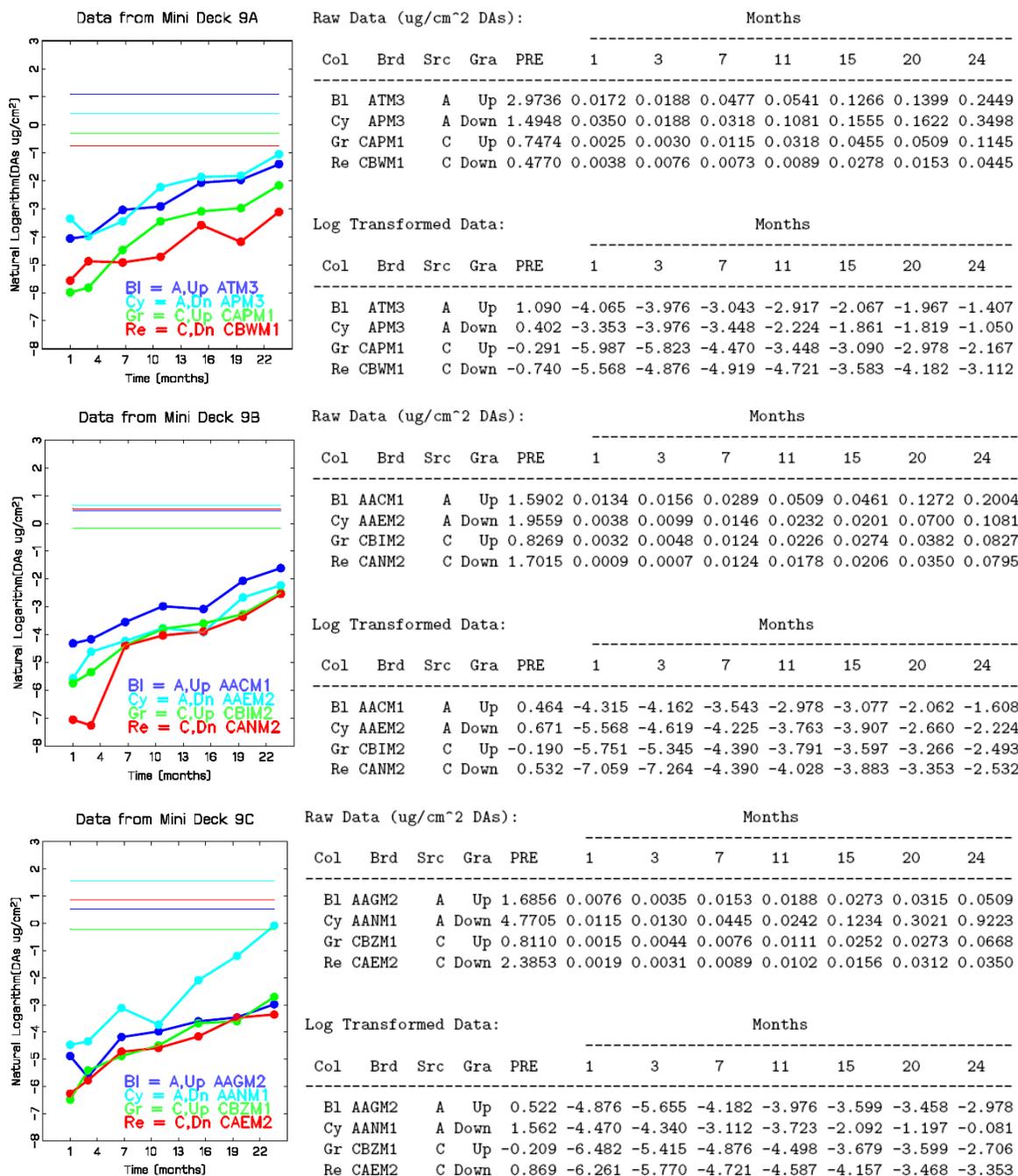


Figure 3-22. Data from Minideck 9A, Data from Minideck 9B, Data from Minideck 9C

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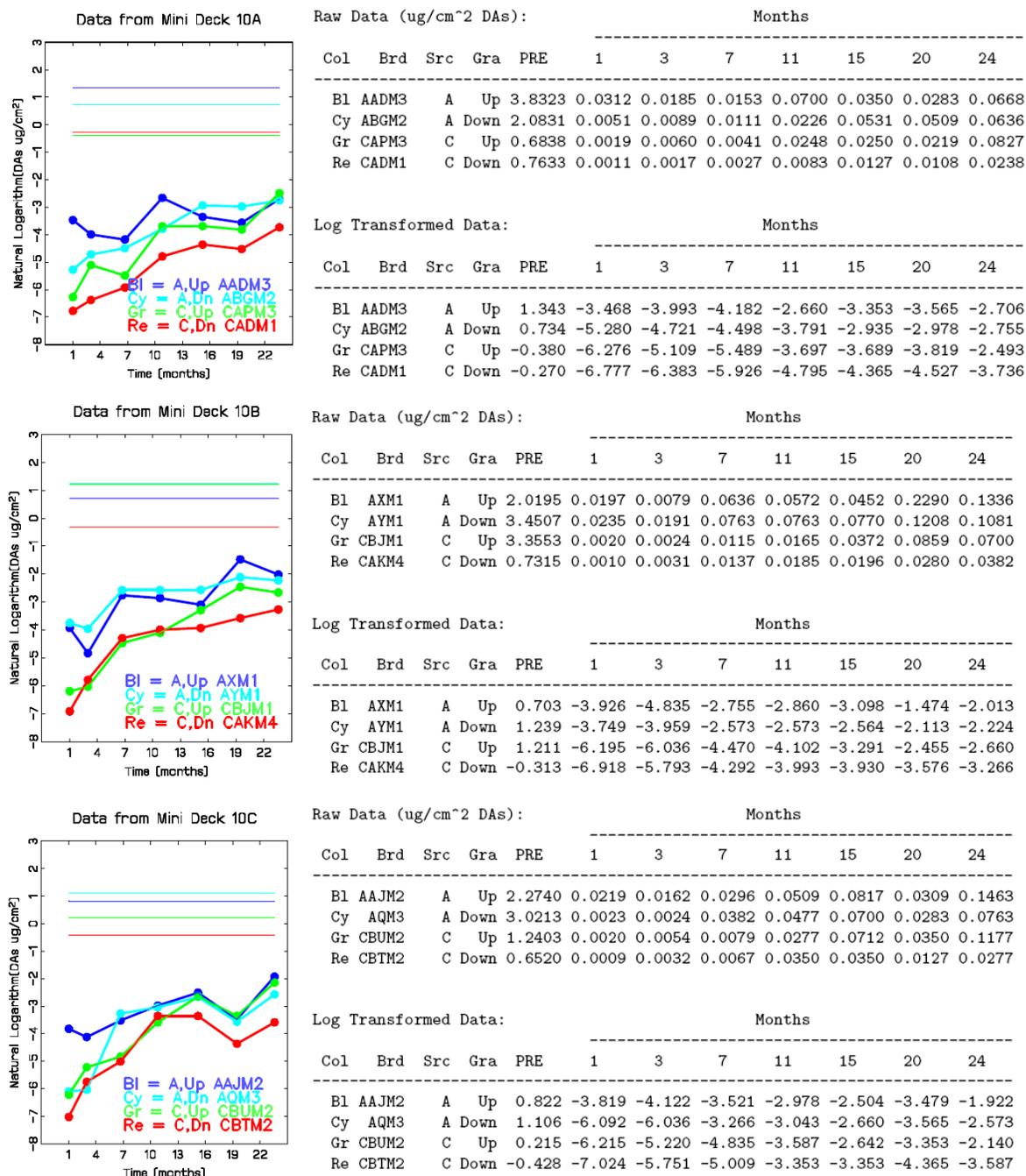


Figure 3-23. Data from Minideck 10A, Data from Minideck 10B, Data from Minideck 10C

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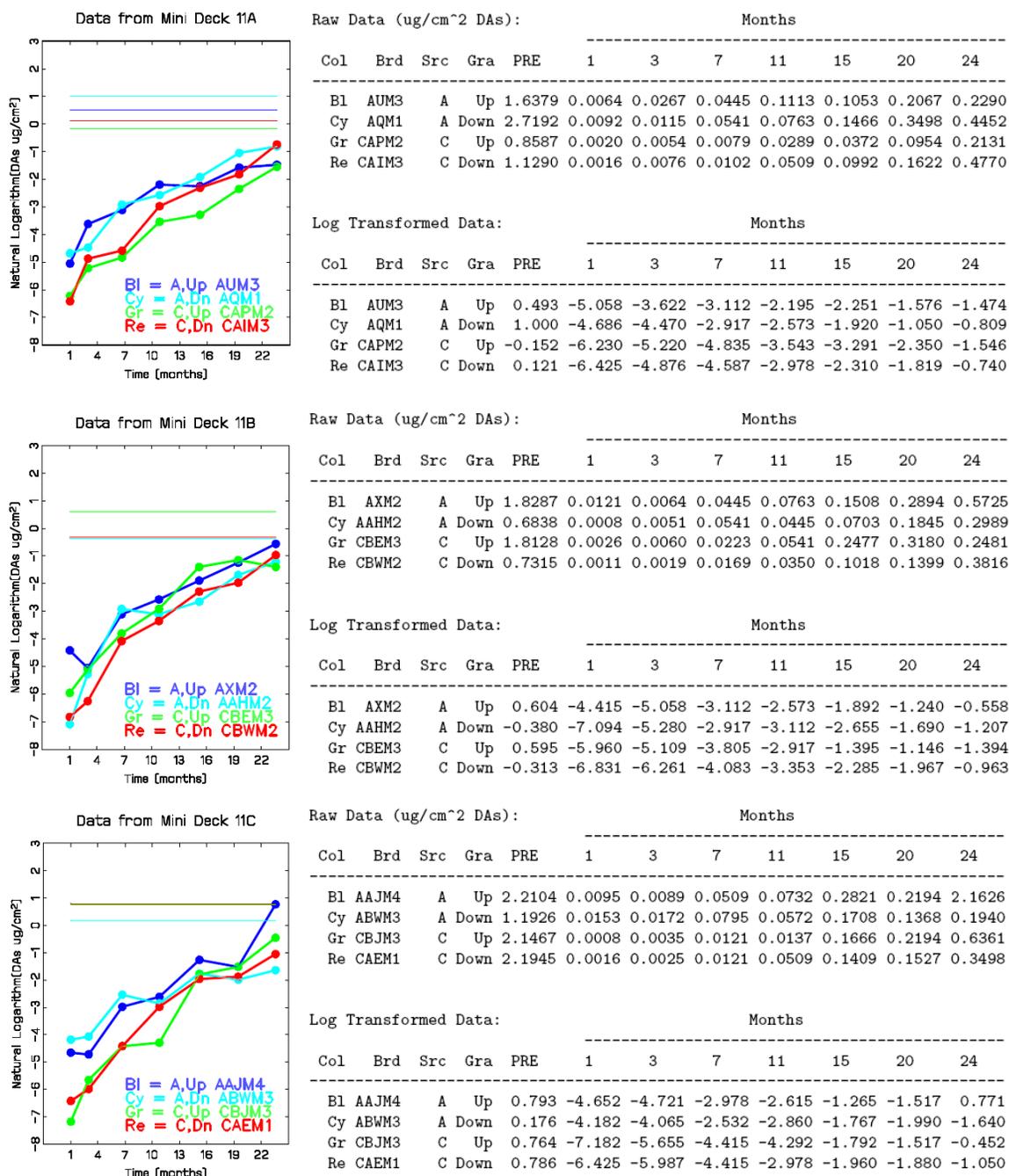


Figure 3-24. Data from Minideck 11A, Data from Minideck 11B, Data from Minideck 11C

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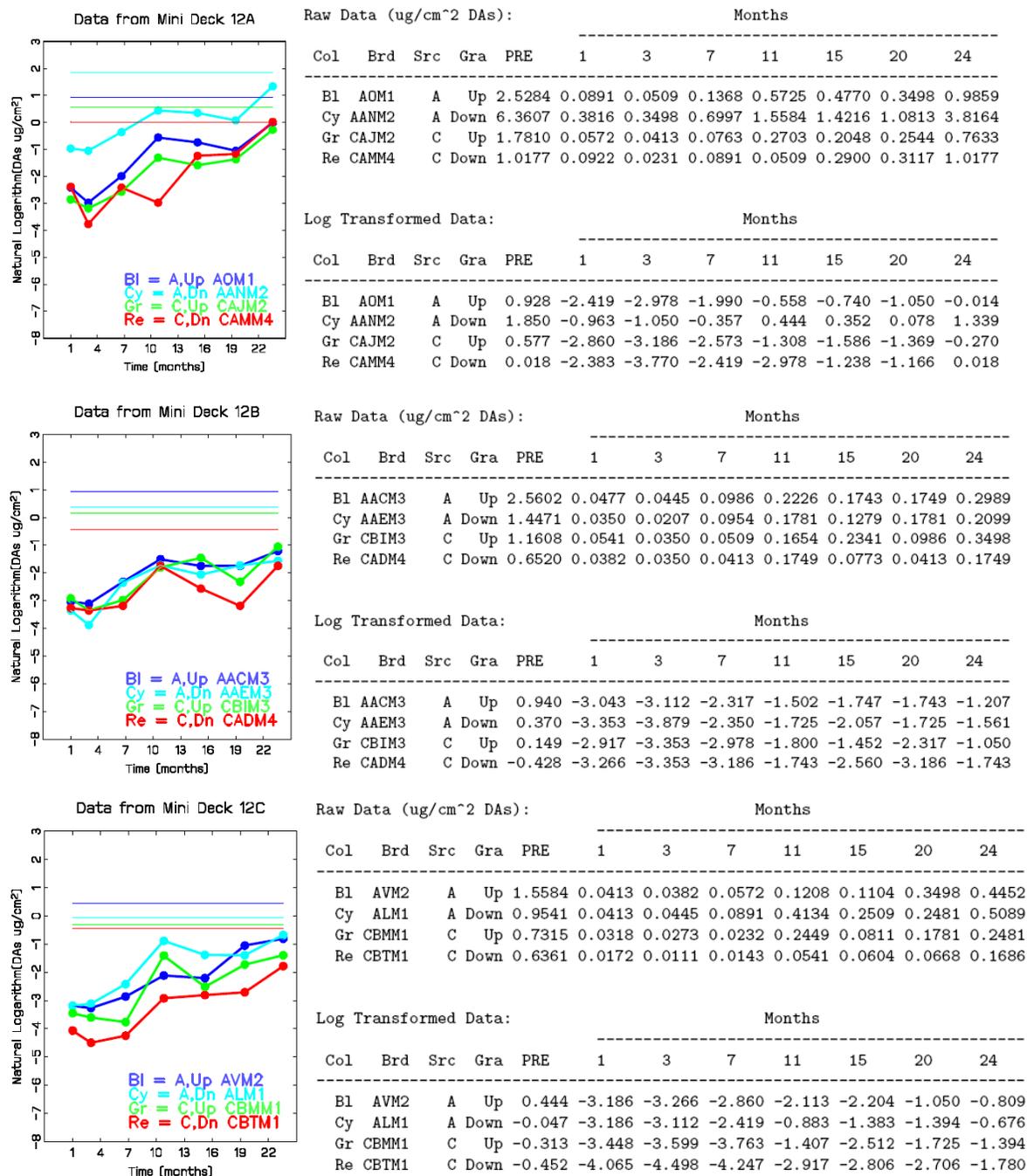


Figure 3-25. Data from Minideck 12A, Data from Minideck 12B, Data from Minideck 12C

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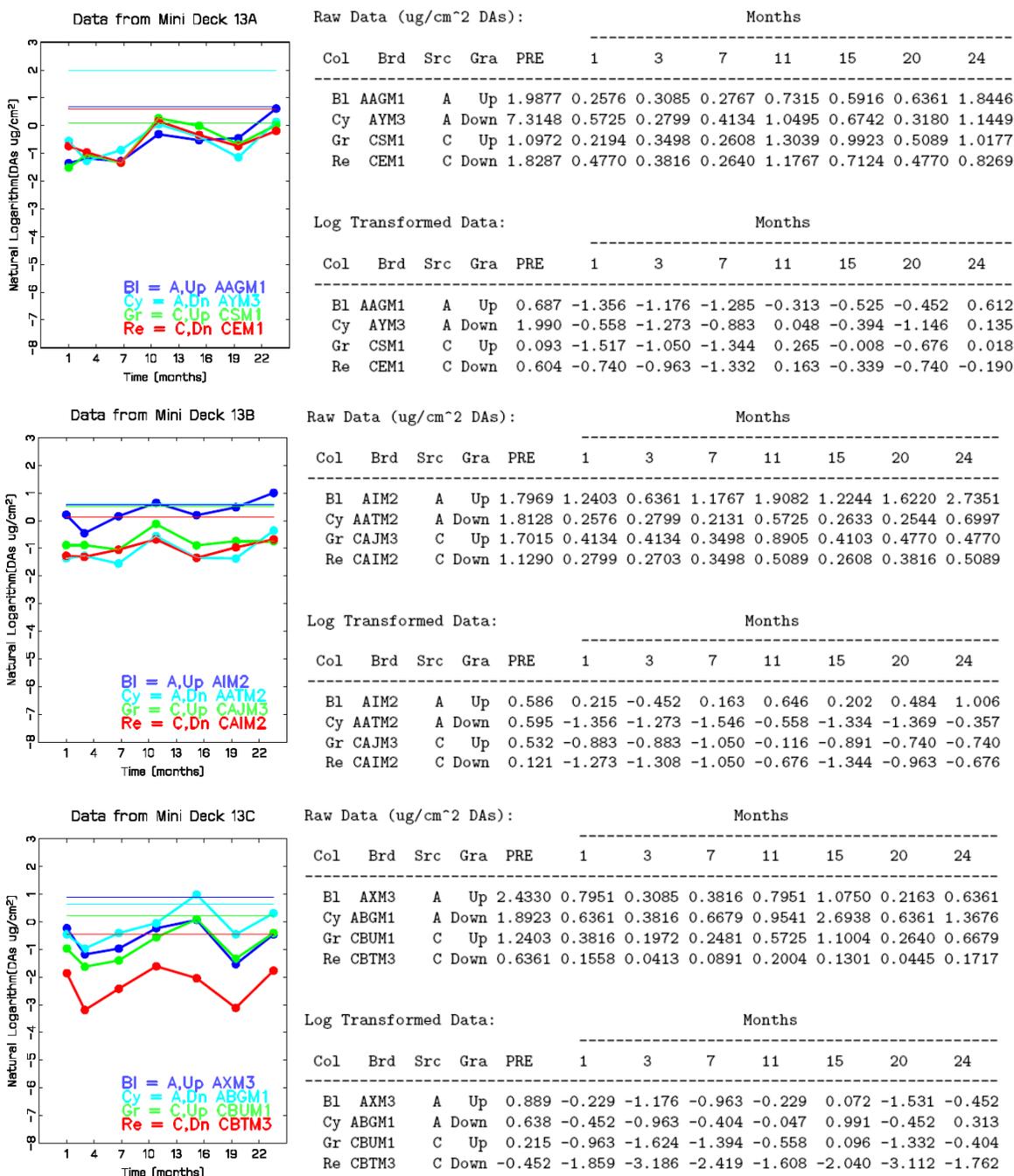


Figure 3-26. Data from Minideck 13A, Data from Minideck 13B, Data from Minideck 13C (all uncoated)

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There is one minor difference between the minideck plots in Figure 3-27 and the plot described in the previous section for Minideck 9-A, Figure 3-13. In Figure 3-13, the baseline values were plotted as straight horizontal lines. With the decreased resolution inherent in Figure 3-27 many of the baseline lines would overlay and obscure one another. So in the plots in Figure 3-27, the baseline measurements horizontal lines have been replaced by lines that have been intentionally “wiggled” or “jittered” to avoid complete over plotting. This is an established graphical technique for increasing the resolution of crowded graphs.

Each separate minideck plot can be examined for the effects of source deck, grain orientation and time as we did for Minideck 9-A. However, this is not the intended purpose of the composite plot (rather, the larger data plots in Figures 3-14 through 3-26 should be used to examine the data for specific minidecks). Here the primary interest is in comparing patterns in DAs measurements among the thirteen coatings, and, secondarily, in visually assessing repeatability of the experimental method by comparing among sets of three replicate minidecks treated with the same coating.

Repeatability is indicated by the similarity in patterns and trends within coatings. For example, it is evident that Coating 13 (uncoated) exhibited the least trend with time (although Coating 5 is a close second), and that this relative lesser trend is manifested in all three minidecks. This is even more noticeable upon consideration of other coatings that exhibit more pronounced trends with time; for example, Coatings 9–11 and to a lesser extent, Coatings 3, 8, and 12. It is evident that certain coatings yielded DAs measurements with different patterns than did Coating 13 (the uncoated control), and furthermore that these patterns were consistent among minidecks treated with the same coating.

As stated earlier, the DCCA observed at any point in time is a function of the time elapsed since the previous wipe event and weathering factors. The two paints and the vinyl elastic product had the lowest initial postcoat (1 month) values and, generally, DCCA showed an increasing trend with time for these products. Most of the other plots in Figure 3-28 exhibit an upward slanting down/up/down/up pattern, suggesting that, for some periods between samples, the wipe sampling (and perhaps other factors that remove DCCA from the surface or affect DCCA retention during wipe sampling) had a greater effect than the time/weathering effect on DCCA.

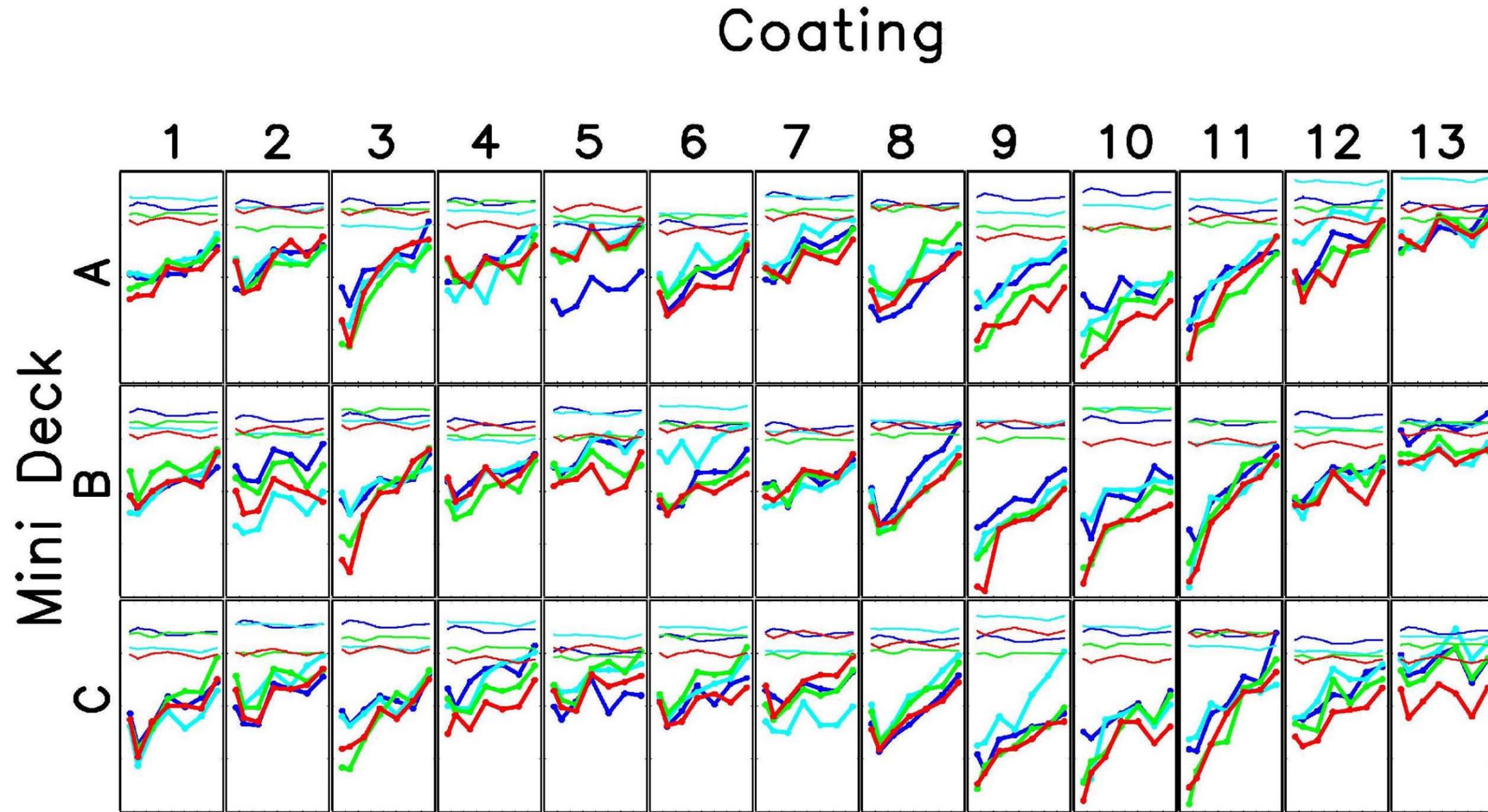


Figure 3-27. Composite Data Plot of All 39 Minidecks (note that scale and axis labels are the same as for Figure 3-13)

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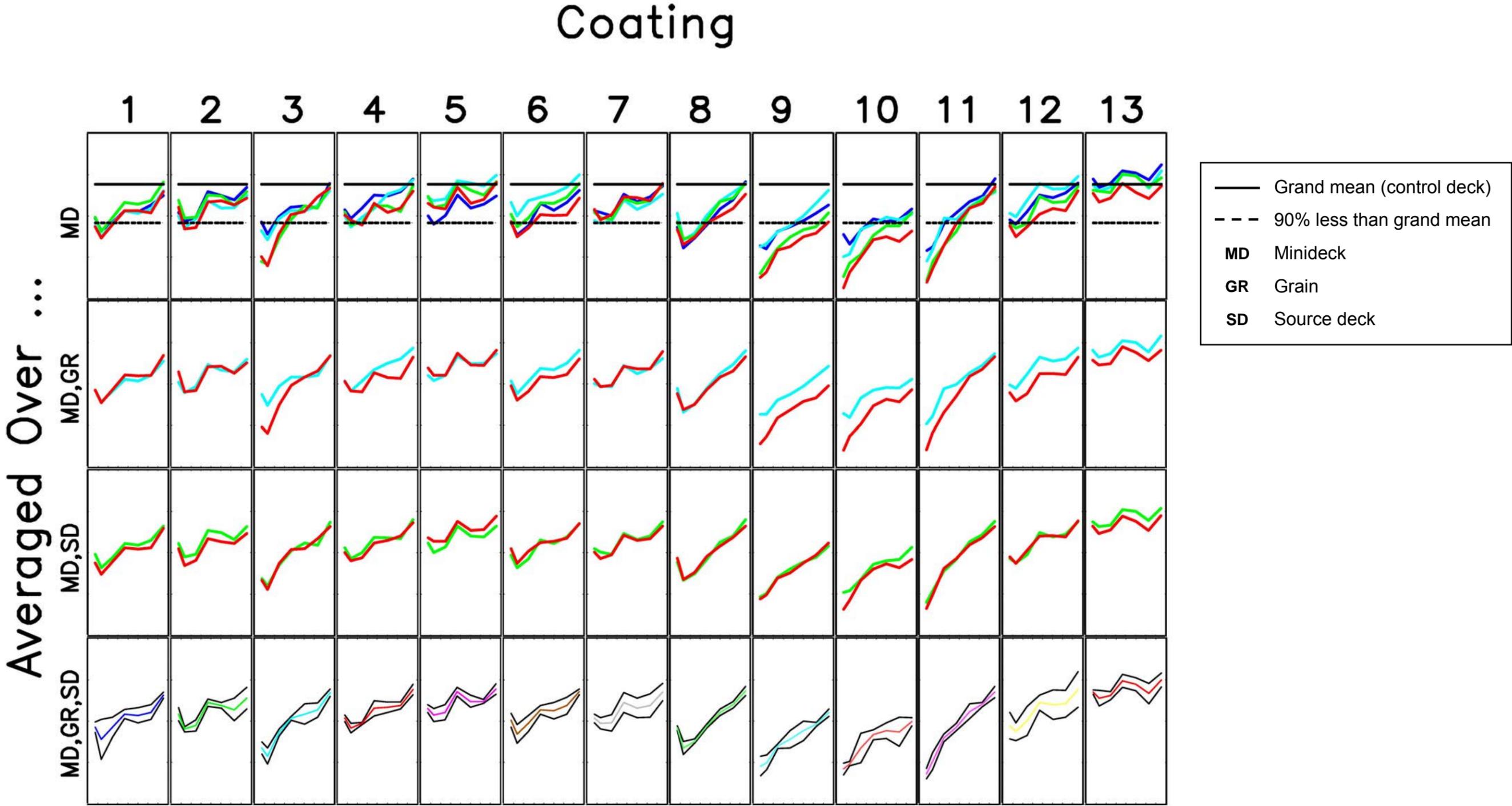


Figure 3-28. Averages from All Thirty-nine Minidecks (See Section 3.2.2.4 for further explanation)

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Before proceeding to the next graphical display one more observation should be made that bears on the validity of the experimental method, and the role/utility of the baseline measurements in particular. A critical scan of all thirty-nine minideck plots reveals two minidecks where the DAs measurements from a single board are noticeably lower, for all time periods, than the DAs measurements from the other boards on the same minideck: minidecks 5-A and 13-C (uncoated). For minideck 5-A, the board with noticeably low post baseline DAs values also had the lowest baseline DAs measurement of all boards, even though it was from Source Deck A, which generally produced with higher-than-average DAs measurements. In other words, this is a board for which the baseline measurement was noticeably low and also resulted in noticeably-low post baseline values. For Minideck 13-C (uncoated), the board with noticeably-low DAs measurements came from Source Deck C and had the lowest baseline DAs measurements of all boards on that minideck.

#### 3.2.2.4 Averages from All Thirty-nine Minidecks

A graphic that displays a series of plots of averages constructed to isolate and illustrate the effect of different factors in the experiment is presented in Figure 3-28. The variables of interest are: source deck, grain orientation and time. In order to reveal the effects of these factors, we present in Figure 3-28 a series of averages over minideck (MD), source deck (SD) and grain (GR).

##### 3.2.2.4.1 Averages over Minidecks

The first row of plots in Figure 3-28 displays coating-specific averages over the replicate minidecks. The color coding is the same as the plots of the data in the previous data displays, as indicated in Table 3-4. Statistically, these plots are the least variable manifestations of the patterns and trends in DAs measurements as they relate to source deck, grain orientation and time period (least variable because they are averaged over all replicate minidecks).

Two horizontal lines have been added to facilitate comparison with Coating 13 (uncoated), the uncoated control. The upper, solid black line is plotted at the grand mean of all post baseline Coating 13 (uncoated) DAs measurements. Recall that Coating 13 (uncoated) exhibited little trend over time and thus the grand mean is an appropriate indicator of overall average Coating 13 (uncoated) DAs value. The upper, black line makes it easy to see how much less the other coating measurements are than the Coating 13 (uncoated) grand average. The lower, dashed black line is plotted at a value equal to  $\ln(10)$  less than the upper black line. In order to understand its

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relevance let  $DA_{s_{13}}$  denote the Coating 13 (uncoated) grand mean. Then,  $DA_{s_{13}/10}$  is the value equal to 90 percent less than the Coating 13 (uncoated) grand mean, and on the log scale this is

$$\ln(DA_{s_{13}/10}) = \ln(DA_{s_{13}}) - \ln(10).$$

Thus the lower black line appears at a level corresponding to a 90 percent reduction from the Coating 13 (uncoated) grand mean.

#### 3.2.2.4.2 Averages over Minidecks and Grain

The second row of plots in Figure 3-28 displays coating-specific DAs values averaged over minidecks (MD) and grain orientation (GR). The purpose is to illustrate more clearly the effects of source deck. The color coding for this row of plots necessarily differs from the previous plots. In this case cyan (Cy) denotes averages from Source Deck A, and red (Re) denotes averages from Source Deck C. In effect, the cyan curves are the averages of the cyan and dark blue curves from the plots in row one; and the red curves are the averages of the red and green curves from the plots in row one. The feature to note is that when the averages differ noticeably, it is generally the case that the red curve lies below the cyan curve, indicating that DAs measurements from Source Deck A boards were generally greater than those from Source Deck C boards.

#### 3.2.2.4.3 Averages over Minidecks and Source Deck

The third row of plots in Figure 3-28 displays coating-specific DAs values averaged over minidecks (MD) and source deck (SD). Its purpose is to illustrate more clearly the effects of grain orientation. The color code for this is row of plots necessarily differs from the previous plots. In this case green (Gr) denotes averages for Grain Up boards, whereas red (Re) denotes averages from Grain Down boards. In effect, the green curves are the averages of the Green and Dark Blue curves from the plots in row one; and the red curves are the averages of the Red and Cyan curves from the plots in row one. The primary feature to note is that the separation between the red and green curves in the third row is generally less than that between the different colored curves in the second row, meaning that the observed effects of grain orientation on measured DAs is noticeably less than that of source deck.

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#### 3.2.2.4.4 Averages over Minidecks, Grain, and Source Deck

The fourth and bottom row of Figure 3-28 displays averages of measured DAs averaged over minideck (MD), grain orientation (GR) and source deck (SD). The purpose is to illustrate the effect of time for the thirteen different coatings *averaged over the other factors*. Each coating is now plotted with its own unique color. The black lines surrounding the time plots are pointwise error bands and are included to give a visual assessment of the variability in the averages. Each colored curve is the average of the four curves in the top row of Figure 3-28. The error-bands were calculated as twice the standard deviation of the four measurements that comprise the plotted averages (i.e., each standard error is associated with three degrees of freedom). This manner of calculating standard errors is conservative (i.e., biased high), because it includes variability due to the effects of source deck and grain orientation. In spite of the conservative nature of the standard errors it is apparent that the averaged DAs data clearly indicate practically significant, as well as statistically significant, trends with time.

#### 3.2.2.5 The Comprehensive Plot

One final graphical display for the DAs measurements that is a composite of all the previous plots is presented in Figure 3-29. Its primary advantage is that all of the graphical information presented thus far is compactly displayed on a single page. This necessarily entails some loss of resolution, but has the advantage that the larger, and thus practically more significant, relevant patterns and trends are still apparent, and can be seen entirely in one graphic. The subplots are displayed in a seven-by-thirteen display with columns corresponding to coatings. The first three rows are the same as in Figure 3-27; and the bottom four rows are the same as in Figure 3-28.

#### 3.2.3 Analysis of Variance of Coatings by Time

The top row of plots in Figure 3-30 is similar to the seventh row in Figure 3-29, but there are important differences. As in the former figure, average  $\ln(\text{DAs})$  values (averaged over minidecks and board types) are plotted versus time. However, in these new plots the thickness of the plotted line corresponds to estimation variability. The widths of the lines are approximately two standard errors of the difference between two means. The standard error used to determine line thickness was determined from the analysis of variance modeling using the baseline covariate. It is approximate because the analysis of variance standard errors differ from one mean to the next due to imbalance (two missing values) and the covariate adjustment. However, the approximation is sufficient for visual comparisons.

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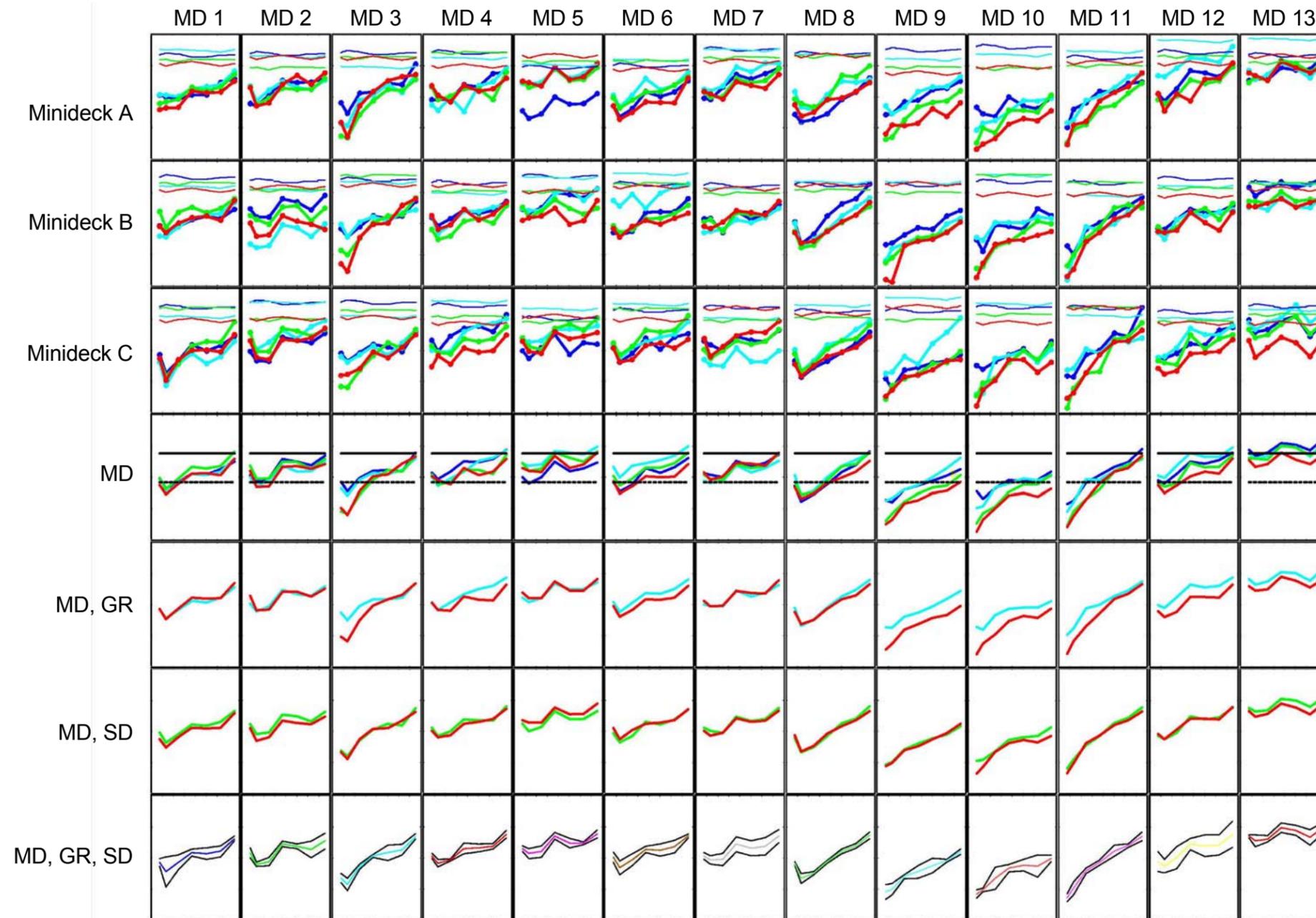


Figure 3-29. Comprehensive Data Plot (note that scale and axis labels are the same as for Figure 3-13)

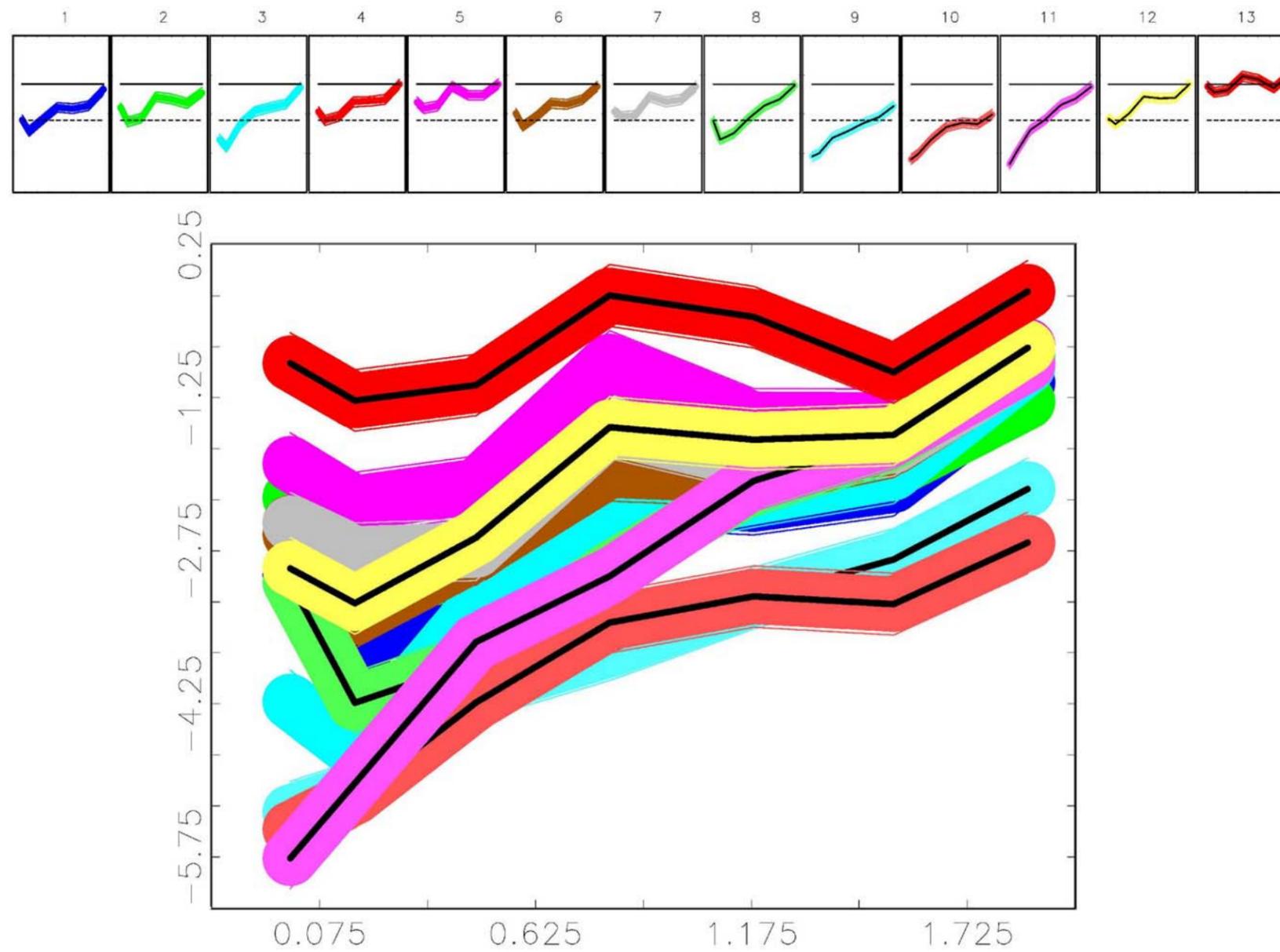


Figure 3-30. Analysis of Variance Plot of Coatings by Time [each subplot has ln(DAs) as its y-axis and time, in years, as its x-axis]

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Because the lines in Figure 3-30 are plotted using the approximate standard error from the difference between two means, it follows that when two of these curves are overlaid, points where they touch are not significantly different; whereas points where they do not touch are significantly different (as judged by a two-sided, pairwise t-test at the 0.05 level of significance with no adjustment for multiple tests). The black horizontal lines are the same as those in the fourth row of plots in Figure 3-29. While these plots are designed to make visual comparisons easy, for verifying statistical significance, the tables in Appendix N should be consulted for the p-values<sup>2</sup> adjusted for the multiplicity of hypothesis tests. Additionally, statistical significance with respect to the uncoated control minidecks is discussed in greater detail in Section 3.1.4.

The top row of plots serves as a legend for the large graph displaying the coatings-by-time plots overlaid. Except for the overplotting, the larger plot makes it is easy to compare means at various time points and to determine whether they are significantly different (not touching) or not (touching).

#### 3.2.4 Analysis of Variance of Coating Pair Comparisons by Time

Figure 3-31 overcomes the overplotting deficiencies of the large plot in Figure 3-30 by plotting all possible pairs of coating-by-time plots. There are 13 coatings and thus there are "13 choose 2" or 78 possible pairwise comparisons among them. Figure 3-31 displays all 78 possible comparisons of the coatings-by-time plots. Running down the diagonal are the 13 individual time plots (1 to 13, in order). In row *j*, the time plot of coating *j* is overlaid with the time plot of coating *k* (for  $k = j+1, \dots, 13$ ). Color coding is established by the diagonal plots and is maintained in the off diagonal plots. Figure 3-31 makes visual comparisons easy and relevant. However, for claiming statistical significance, the tables in Appendix N should be consulted for p-values adjusted for the multiplicity of hypothesis tests.

The results presented above pertain only to DAs on the true time scale measured in years from start date.

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<sup>2</sup>A p-value is a measure of the amount of statistical evidence supporting an alternative hypothesis relative to a particular null hypothesis, with smaller p-values corresponding to greater evidence. The null hypothesis is usually the hypothesis of "no difference" or "no effect"—no difference in mean DAs between Coating 1 and the Coating 13 at time period 1. Whereas the alternative hypothesis asserts that a difference (or effect) exists—Coating 1 and Coating 13 have different mean DAs at time period 1. P-values always lie between 0 and 1 and thus are probabilities. In fact, the p-value is the probability of finding a difference (or an effect) as large as the one observed in the data, when the null hypothesis is true. A p-value less than 0.05 is generally considered to be "statistically significant" although the choice of 0.05 is somewhat arbitrary and sometimes other cutoff values—0.1 or 0.01, are used to classify finding as statistically significant or not.

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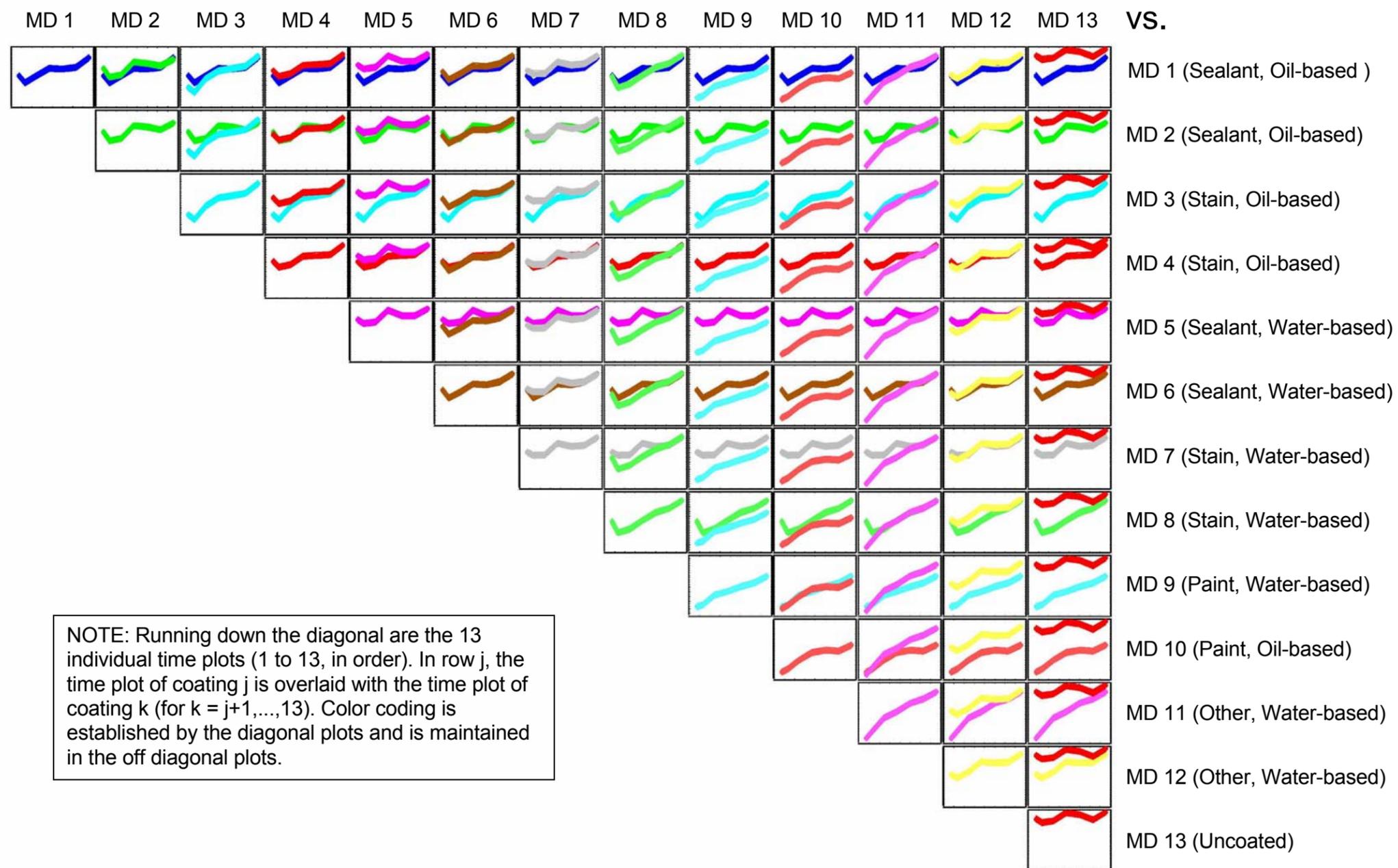


Figure 3-31. Analysis of Variance Plot of Coating Pair Comparisons by Time [each subplot has ln(DAs) as its y-axis and time, in years, as its x-axis]

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### 3.2.5 Analysis of Variance Results

The split-split-plot analysis of variance results for ln(DAs) is now presented. The statistical models (one with baseline covariate adjustment, one without) used are described in Section 1.3. Here only the analysis of variance results for the fixed effect factors coating, source deck, grain and time is presented. Table 3-5 displays the test statistics and p-values for the standard split-split-plot analysis of variance with and without the baseline covariate.

Table 3-5. ln(DAs) ANOVA Table

Effect	Without Baseline Covariate		With Baseline Covariate	
	F-Value	P-value	F-Value	P-value
Coat	23.24	<.0001	42.44	<.0001
Bbf	2.21	0.1416	0.67	0.4164
Sdeck	30.1	<.0001	1.11	0.2958
bbf*sdeck	5.47	0.0219	5.5	0.0216
coat*bbf	0.72	0.7235	1	0.4554
coat*sdeck	2.49	0.0081	2.91	0.0023
coat*bbf*sdeck	1.44	0.1657	1.22	0.2884
Time	708.49	<.0001	708.48	<.0001
coat*time	15.3	<.0001	15.3	<.0001
bbf*time	0.89	0.5037	0.9	0.4962
Sdeck*time	3.92	0.0007	3.93	0.0007
bbf*sdeck*time	1.21	0.2987	1.21	0.2991
coat*bbf*time	0.56	0.9986	0.56	0.9986
coat*sdeck*time	2.51	<.0001	2.51	<.0001
coat*bbf*sdeck*time	0.63	0.9914	0.63	0.9914
Lnbas	NA	NA	46.42	<.0001

Note the differences between the analyses with and without covariate adjustment. Qualitatively, the only notable difference between the analyses with and without the baseline covariate is the attenuation of significance of the main effect for source deck (sdeck). The baseline covariate adjustment is designed to account for board-to-board differences in DAs. Thus, if the DAs levels differ (on average) from one source deck to

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the other, these differences should be reflected in the baseline measurements. Consequently, the inclusion of the baseline covariate in the statistical modeling would tend to lessen the effect of source deck because, in effect, the baseline measurements explain some of the differences between source decks.

Thus, the difference in significance level of source deck (sdeck) with and without covariate adjustment is not unexpected. In fact it is consistent with the conclusion that the baseline measurements are useful covariates; that is, they are a useful component of the experimental design. The latter conclusion is supported more directly by the larger F-statistic for coating (coat) in the model with baseline, and also the statistical significance of the covariate itself (p-value < .0001).

The pattern of significance in the ANOVA tables is consistent with the patterns seen in the graphics in Figure 3-13 and Figures 3-27 through 3-31 and confirms the visual assessment of the data to a great extent.

Coating main effects are clearly evident in the figures and are statistically significant (p-value < .0001). Refer to Appendix O for estimated main effects of coating and also for multiplicity-adjusted p-values for pairwise comparisons of coatings main effects. The significance (p-value < .0001) of time main effects is due largely to the increasing time trends apparent in Figure 3-27 for most coatings. The significance (p-value < .0001) of the time-by-coating interaction effect is also apparent: the shape, or pattern, of the time trends differ among coatings. The significance of the source deck and coating, and source deck, coating and time interactions are manifest somewhat in the second row of Figure 3-28 where there is some difference between the cyan and red lines for different time periods and also among coatings.

In summary, the analysis of variance suggests some, but not great, effect of grain orientation (in Figure 3-28, the third row lines are similar); some evidence of an effect of source deck and its interaction with time and coating (in Figure 3-28, the separation in some of the lines in the second row differ by time and coating); and strong effects due to time (obvious in all plots) and coating (also obvious in all plots). Appendix O contains the results of pairwise tests of main effects of coating and coating-by-time interactions adjusted for multiple testing effects using the Kramer-Tukey method for pairwise comparisons. These can be consulted for detailed specific comparisons of mean ln(DAs).

In addition to the pairwise tests reported in Appendix O, differences among certain identifiable subgroups of coatings were also tested. The results of these exploratory analyses are reported in Table 3-6 (the p-values are not adjusted for multiplicity).

**Table 3-6. Pairwise Comparisons of Coating Characteristics**

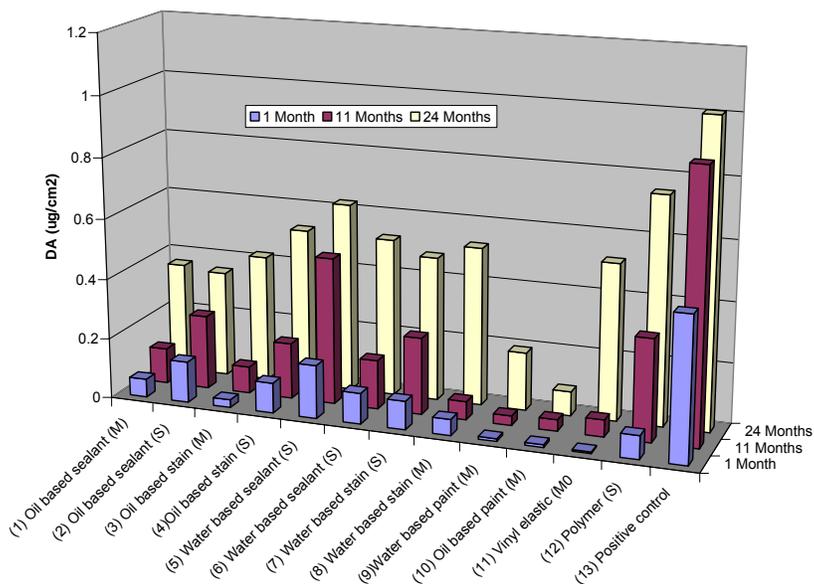
Comparison	t-statistic	p-value
Sealant vs. Stain	1.4663	0.0007
Oil-Based vs. Water-based	-3.21	0.0035
Penetrating vs. Film-Former	15.68	<.0001
Clear Cover vs. Semi-Transparent	-0.5055	0.5579
No Acrylic vs. Acrylic	7.2761	0.0018
No Alkyd vs. Alkyd	-29.31	0.0292
Multi-coat vs. Single Coat	-7.3019	<.0001

The "sealant vs. stain" comparison tested the average difference between sealants and stains. Sealants had higher ln(DAs) than stains (p-value = .0007). The non-film formers performed worse (p-value < 0.0001) than the film formers. Also the non-acrylics performed worse than the acrylics (p-value = 0.0018); and the non-alkyds performed better than the alkyds (p-value = 0.0002). The difference in performance between oil- and water-based products was significant when the analysis includes the paints (p-value 0.0035), however, the difference is not significant at the 0.05 level when paints are excluded from the analysis (p-value = 0.0885); a potentially useful observation in light of the phase-out of oil-based products in response to phased implementation of the Clean Air Act amendments restricting the atmospheric release of volatile organic compounds. The products for which application of more than one coat was recommended performed significantly better than the products for which single coat was recommended (p-value <0.0001). However, the number of coats is confounded with both the recommended application procedure and with the product. As such, one cannot infer from this observation that multiple coats of a product provide improved reduction of DCCA because the study design did not examine this question directly by testing multiple application levels for any product. A bar chart comparing performance of single versus multi-coat products at 1, 11 and 24 months is provided as Figure 3-32.

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**Figure 3-32. Single (S) versus Multi-Coat (M) Analysis Summary**

3.2.6 Summary of Coating Comparison Results

To summarize, inspection of the 13th column in Figure 3-31 reveals that each coating mitigated DAs somewhat when compared to the positive control. However, recall that the visual comparisons made possible in Figure 3-31 are not adjusted for the effects of multiple hypothesis testing.

Table 3-7 summarizes the differences between the performance of each coating versus the positive control [minideck 13 (uncoated)] as well as the statistical significance of the differences at each time interval. The color-coding of cells indicates level of significance: blue indicates a p-value of less than 0.01; green indicates a p-value between 0.01 and 0.1; and beige indicates a p-value of greater than 0.1.

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**Table 3-7. Summary of Estimated ln(DAs) Difference and Associated Statistical Significance between Each Coating and Control [#13 (uncoated)] Minidecks at Each Time Interval)**

Coating		Time After Coating						
		1 Month	3 Month	7 Month	11 Month	15 Month	20 Month	24 Month
1	p-value	<.0001	<.0001	<.0001	<.0001	<.0001	0.0614	0.5753
	est. diff. (ln)	-2.0151	-2.3877	-1.8192	-1.912	-1.78	-1.0473	-0.8385
2	p-value	0.0031	<.0001	<.0001	0.0051	0.0124	0.4819	0.1412
	est. diff. (ln)	-1.2272	-1.7567	-1.686	-1.2007	-1.1501	-0.8643	-0.986
3	p-value	<.0001	<.0001	<.0001	<.0001	<.0001	0.1299	0.8733
	est. diff. (ln)	-3.3166	-3.4455	-2.1913	-2.2258	-1.7665	-0.9912	-0.7455
4	p-value	<.0001	<.0001	<.0001	<.0001	0.0016	0.9971	1
	est. diff. (ln)	-1.4618	-1.6663	-1.4842	-1.5374	-1.2631	-0.6165	-0.4527
5	p-value	0.7966	0.7258	0.7615	1	0.7245	1	1
	est. diff. (ln)	-0.7799	-0.8022	-0.7913	-0.4451	-0.8026	-0.2627	-0.3332
6	p-value	<.0001	<.0001	<.0001	<.0001	<.0001	0.9946	0.9989
	est. diff. (ln)	-1.5753	-2.0258	-1.5524	-1.6148	-1.4609	-0.6315	-0.5945
7	p-value	<.0001	<.0001	<.0001	0.0055	0.0006	0.9969	0.9981
	est. diff. (ln)	-1.4673	-1.4017	-1.5155	-1.1967	-1.3108	-0.6177	-0.6064
8	p-value	<.0001	<.0001	<.0001	<.0001	<.0001	0.9879	0.997
	est. diff. (ln)	-2.1235	-2.9407	-2.6892	-2.6673	-1.6328	-0.6529	-0.6156
9	p-value	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
	est. diff. (ln)	-4.2973	-3.7228	-2.8825	-3.3713	-2.655	-1.73	-1.833
10	p-value	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
	est. diff. (ln)	-4.5354	-3.8573	-3.08	-3.1754	-2.7103	-2.241	-2.436
11	p-value	<.0001	<.0001	<.0001	<.0001	<.0001	1	0.9985
	est. diff. (ln)	-4.7347	-3.6372	-2.4045	-2.6383	-1.4936	-0.5302	-0.6015
12	p-value	<.0001	<.0001	0.0002	0.0118	0.0503	1	1
	est. diff. (ln)	-1.872	-1.774	-1.3581	-1.1549	-1.0639	-0.4727	-0.4167

From inspection of the pairwise tests summarized in Table 3-7, the adjusted p-value for comparing the main effects of coatings 5 and 13 (uncoated) is not significant at the .05 level of significance. Thus, there is very little evidence of a difference in performance between coatings 5 and 13 (uncoated). All of the other coatings exhibited statistically

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significant differences in performance versus the control through the time = 4 sampling event, or after approximately one year of weathering. The paints were the only products that continued to show statistically significant performance differences versus the control throughout the two-year study period. All of these results are borne out visually in the graphical presentation in Section 3.2.2 through 3.2.4.

Table 3-7 reports estimated differences of the means of the natural logarithms of DAs. This is the best scale for the statistical analysis, but not the best scale for interpretation. A preferable scale for interpretation is percent reduction relative to the control deck. The estimated differences of logs in Table 3-8 are converted to estimated percent reductions relative to the control deck by exponentiating the differences, subtracting from 1, and multiplying by 100. These estimated percent reductions are reported in Table 3-8 along with lower and upper 95 percent confidence limits (UCL/LCL) for all coatings and time periods. The confidence limits were calculated by first constructing 95 percent confidence intervals for estimated difference of logs and then transforming those lower and upper bounds using the same formula for converting the estimated difference of logs to percent reductions.

Table 3-8 further illustrates what has been demonstrated graphically in Figure 3-31, that is, product performance in reduction of dislodgeable arsenic residues decreases with time. For the products tested, the paints ranked highest, followed by multi-coat stains, sealants, and the vinyl elastic encapsulant, followed by the single-coat products. However, confidence intervals for estimated percent reduction are generally large and this challenges attempts to rank and compare product performance based upon estimates of percent reduction. However, the information in the table provides an easily interpreted picture of product performance range, and if used in conjunction with the information in Figure 3-31, it is possible to easily differentiate between individual products by consulting Figure 3-31 to determine which products are statistically different at any point in time.

The study design stipulated use of deck preparation steps recommended by the coating manufacturer. Thus, mini-decks for ten of the products were cleaned using a specific recommended cleaning agent followed by a water rinse, whereas mini-decks coated with Product 3, an oil-based stain, received only a water rinse, and the mini-decks coated with Product 7, a water-based stain, were neither cleaned nor rinsed. The positive control decks were rinsed with water but not treated with a cleaning agent. As seen in Table 3-7, DAs for mini-decks coated with Product 7 were statistically different than the positive controls through the first 15 months of weathering. Thus, anecdotal evidence suggests that coating is more effective than rinsing with water, at

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least through a one-year period. The decks coated with Product 3 received the same treatment as the positive controls and DAs was significantly less than the positive controls through 15 months. Thus, anecdotal observations indicate that coating (with most products) is more effective than washing alone in reducing DAs.

**Table 3-8. Estimated Percent Reduction for Each Coating and Estimated 95 Percent Confidence Intervals**

Coating		Time After Coating						
		1 Month	3 Month	7 Month	11 Month	15 Month	20 Month	24 Month
1	%Reduction	87	91	84	85	83	65	57
	LCL/UCL (%)	61/95	73/97	53/94	57/95	51/94	-1/88	-25/85
2	%Reduction	71	83	81	70	68	58	63
	LCL/UCL (%)	15/90	50/94	46/94	13/90	8/89	-22/85	-8/87
3	%Reduction	96	97	89	89	83	63	53
	LCL/UCL (%)	90/99	91/99	68/96	69/96	51/94	-7/87	-37/84
4	%Reduction	77	81	77	79	72	46	36
	LCL/UCL (%)	33/92	45/93	33/92	38/93	18/90	-56/81	-84/78
5	%Reduction	54	55	55	36	55	23	28
	LCL/UCL (%)	-33/84	-31/85	-32/84	-87/78	-30/85	-124/74	-109/75
6	%Reduction	79	87	79	80	77	47	45
	LCL/UCL (%)	40/93	62/95	39/93	42/93	33/92	-54/82	-60/81
7	%Reduction	77	75	78	70	73	46	45
	LCL/UCL (%)	33/92	29/91	36/92	13/90	22/91	-56/81	-58/81
8	%Reduction	88	95	93	93	80	48	46
	LCL/UCL (%)	65/96	85/98	80/98	80/98	44/93	-50/82	-56/81
9	%Reduction	99	98	94	97	93	82	84
	LCL/UCL (%)	96/100	93/99	84/98	90/99	80/98	49/94	54/94
10	%Reduction	99	98	95	96	93	89	91
	LCL/UCL (%)	97/100	94/99	87/98	88/99	81/98	69/96	75/97
11	%Reduction	99	97	91	93	78	41	45
	LCL/UCL (%)	97/100	92/99	74/97	79/98	35/92	-70/80	-59/81
12	%Reduction	85	83	74	68	65	38	34
	LCL/UCL (%)	55/95	50/94	25/91	9/89	0/88	-81/78	-91/77

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### 3.3 Coating Appearance

The coating appearance after weathering was qualitatively assessed in person and by viewing photographs of each minideck at each sampling event (precoat and after 1 month, 3 months, 7 months, 11 months, 15 months, 20 months and 24 months of weathering). The photos can be viewed in Appendix P. Notable observations are summarized in Table 3-9.

### 3.4 Weather Data

A number of meteorological measurements were made during this study, as described in Section 2.8. The most relevant parameters in terms of coating performance and sampling are: Solar Radiation, Rainfall and Temperature. Tables 3-10 and 3-11 summarize key weather parameters by sampling interval, where the two numbers in the first column refer to the sequentially numbered sampling events defining the period over which the weather parameters are summarized. Figures 3-33 through 3-35 plot key parameters versus time. In all plots, the dates of the sampling events are superimposed for reference. Complete weather data collected are provided in Appendix Q.

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Table 3-9. Summary of Visual Observations of Minidecks

#	Product Type	Base	Cover	Pigmentation	Main Ingredients	Summary of Visual Observations
Coating 1	Sealant	Oil	Semi	"Cedar"	Aliphatic, Napthalene	Deep red coloration. Some wear-through where wiped.
Coating 2	Sealant	Oil	Clear	"Clear"	Acrylic, alkyd, urethane	No visible signs of coating. Relatively light and bright wood appearance.
Coating 3	Stain	Oil	Clear	"Deep Tone Base" (no pigment added)	Acrylic	Extensive black mold or mildew on untreated boards. Varying amounts, from slight to extensive, on treated boards. Growth appeared between 7 and 11 months after coating. Otherwise, no visible signs of coating, though with fresh coating the wood appearance was significantly darkened.
Coating 4	Stain	Oil	Clear	"Clear Stain"	Alkyd	No visible signs of coating. Mold or mildew on untreated boards.
Coating 5	Sealant	Water	Clear	"Clear"	Unknown	No visible signs of coating. Relatively dark (gray) wood appearance.
Coating 6	Sealant	Water	Clear	"Clear"	Acrylic, alkyd	Slightly yellow tint to treated boards. Some wear-through where wiped.
Coating 7	Stain	Water	Semi	"Cedar"	Alkyd	Lighter red coloration. Wear-through where wiped.
Coating 8	Stain	Water	Clear	"Tint base, solid" (no pigment added)	Acrylic	Very slight tint on treated boards, but generally no visible signs of coating. Relatively light and bright wood appearance.
Coating 9	Paint	Water	Opaque	Gray	Acrylic	Retained gray paint coloration, but moderate-to-extensive chipping, especially at cracks, starting around 7 months.
Coating 10	Paint	Oil	Opaque	Gray	Alkyd, polyurethane	Retained gray paint coloration, slight-to-moderate chipping at cracks. Some black mold or mildew on untreated boards.
Coating 11	Other		Clear	Clear	Elastic vinyl	Extensive black mold or mildew on untreated boards. Varying amounts, from none to moderate, on treated boards. Growth appeared between 7 and 11 months after coating. Seems to visually perform better on A source than C source. Some limited chipping and peeling at large cracks. General appearance is slick and waxy, with an amber coloration that has held well on treated boards.
Coating 12	Other		Clear	Clear	Polymer	No visible signs of coating.
Coating 13	No coating					No visible signs of coating. Some mold and mildew on untreated boards.

MSDS does not include any ingredients >1 percent by weight

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Table 3-10. Summary of Key Weather Parameters

Sampling Interval	Total Solar Radiation (W/m <sup>2</sup> × day)	Avg. Solar Radiation (W/m <sup>2</sup> )	Total Rainfall (inches)	Precip Events >0.25" (#)	Precip Events >0.10" (#)
0-1	6075	196.0	4.71	5	7
1-2	8567	142.8	4.63	4	10
2-3	11953	105.8	8.54	13	17
3-4	26801	216.1	11.14	13	18
4-5	23980	179.0	24.53	22	24
5-6	15145	116.5	9.02	11	16
6-7	27860	222.9	13.36	13	18

Table 3-11. Summary of Temperature Measurements

Sampling Interval	Avg. Temp.(degrees F)	Avg. High Temp (degrees F)	Avg. Low Temp (degrees F)	Max. High Temp (degrees F)	Min. Low Temp (degrees F)
0-1	74.5	86.0	68.1	95.4	52.9
1-2	61.1	73.5	51.7	86.3	31.8
2-3	41.3	54.0	32.3	82.2	9.5
3-4	65.3	77.3	55.7	95.1	21.3
4-5	69.8	78.8	59.4	95.9	28.0
5-6	44.3	56.3	34.5	78.0	11.0
6-7	70.8	82.3	59.3	103.3	35.0

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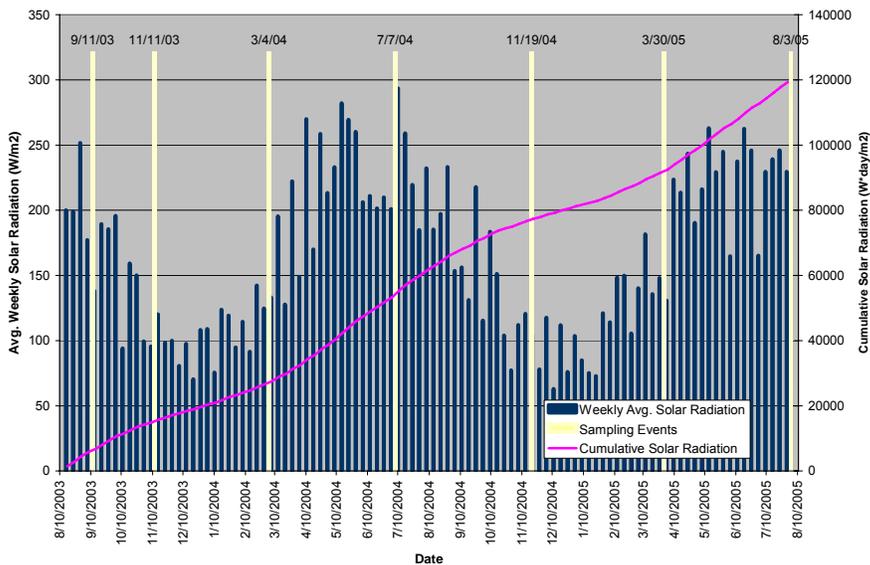


Figure 3-33. Solar Radiation Data Summary

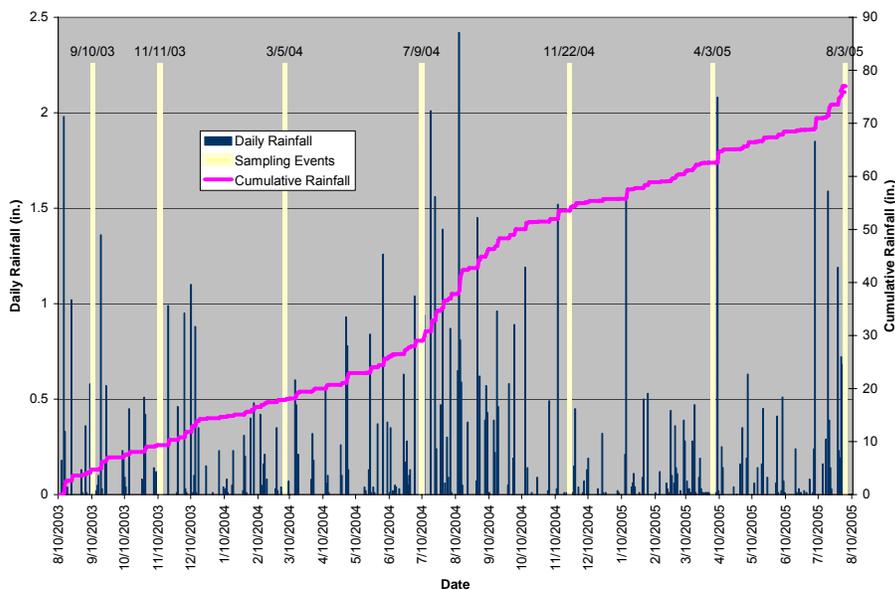


Figure 3-34. Rainfall Data Summary

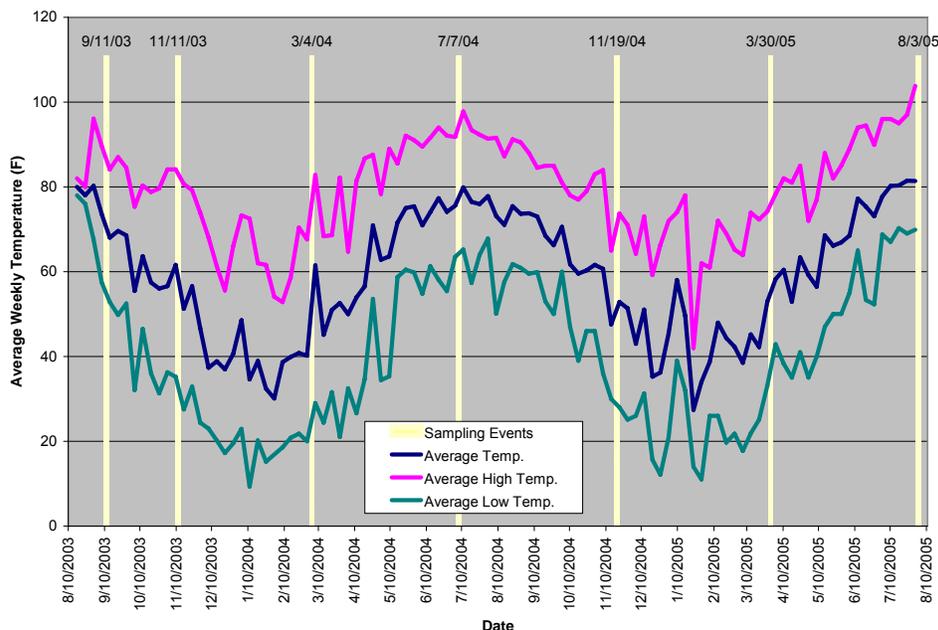


Figure 3-35. Temperature Data Summary

### 3.4.1 Exploratory Analysis of Weather Effects

It is possible and perhaps even likely that the effects of weather on coating performance will differ from one coating to another—film formers might be affected by weathering differently than non film formers. Thus, in searching for possible weather effects, it is necessary to consider coatings separately. We accomplished this by examining the effects of weather and time jointly using regression methods for each of the 13 coatings. The response variables in these analyses are the ln(DAs) values averaged over grain and source deck—the ln(DAs) averages plotted in the bottom row of Figure 3-28. We looked for weather effects using multiple linear regressions models with time as one predictor and a second predictor corresponding to one of the climatological variables taken in succession. For these analyses we assumed that the averaged ln(DAs) values vary linearly with time and the included weather variable. That is, with time and one weather variable, we assumed the following statistical model:

$$\ln(\text{DAs}) = b_0 + b_1 \cdot \text{time} + b_2 \cdot \text{weather} + (\text{random error})$$

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The problem is that there are only 7 data points to fit such a model. Nevertheless, it can be done and the question becomes whether  $b_2$ , the coefficient of the weather variable, is significantly different from 0.

A total of 15 weather variables, as listed in Table 3-12, were selected for analysis. Since there were 13 coatings by 15 weather variables,  $13 \times 15 = 195$  such regressions were conducted. The results are summarized in 13-by-15 arrays and one figure.

**Table 3-12. List of Weather Variables Considered in Statistical Analysis**

#	Weather Variable	Description
1	TSRad	Total solar radiation
2	Cum_TSR	Cumulative solar radiation
3	Rad_Int	Average solar radiation
4	TRain	Total rainfall
5	#HRain	Number of rain events greater than 0.25"
6	#LRain	Number of rain events greater than 0.10"
7	AT	Average temperature
8	AHT	Average high temperature
9	MHT	Maximum high temperature
10	ALT	Average low temperature
11	MLT	Minimum low temperature
12	0-#Frz	Number of freezing events
13	ATDiff	Average daily temperature difference
14	#LTDiff	Number of days with a temperature difference greater than 26 F
15	#STDiff	Number of days with a temperature difference greater than 32 F

The first array (not shown) provides the partial correlations between  $\ln(\text{DAs})$  and weather after adjusting for time effects. Table 3-13 includes the correlations for all cases where the p-value  $< .10$ . These are the only correlations worthy of further consideration.

Table 3-13. Partial Correlations for Pairs with p-values < 0.10

Coating	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	---	---	0.79	---	---	---	---	---	0.73	---	---	---	---	---	---
2	---	---	0.87	---	---	---	0.75	0.75	0.74	0.77	---	---	---	-0.8	---
3	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
4	---	---	0.93	---	---	---	0.82	0.84	0.86	0.84	0.79	---	---	-0.77	---
5	0.76	---	0.87	---	---	---	---	---	---	---	---	---	---	---	---
6	---	---	0.78	---	---	---	---	---	---	---	---	---	---	---	---
7	---	0.74	0.93	---	---	---	0.8	0.82	0.74	0.81	---	0.77	---	-0.73	---
8	---	---	---	---	---	---	---	---	---	---	0.83	---	-0.77	-0.8	-0.75
9	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
10	0.82	---	---	---	0.77	0.86	---	---	---	---	---	---	---	---	---
11	---	---	---	---	0.78	0.91	---	---	---	---	-0.74	---	---	---	---
12	0.83	---	0.78	---	---	---	---	---	---	---	---	---	---	---	---
13 (uncoated)	0.88	---	0.9	---	---	---	0.74	0.74	0.85	0.73	---	---	---	---	---

Figure 3-36 shows the 195 partial regression plots resulting from the 13 x 15 matrix of coating x weather factor. Columns correspond to the 15 weather variables shown in Table 3-13, while rows correspond to coatings 1, 2,..., 13. Partial regression plots are constructed to show how ln(DAs) relates to weather after the linear effects are removed. Each plot contains 7 points and also the partial regression line. A black solid line means that the correlation is statistically significant at .10 significance level (.10 is used because of the exploratory nature of the evaluation—we do not want to miss relationships that might exist but are weak).

The black lines in columns 7, 8, 9, 10 and 11 of Figure 3-36 are all temperature variables and are essentially the same for the purposes of this analysis. So if one of them is significant then it is highly likely that the others will also be significant (or close to significant).

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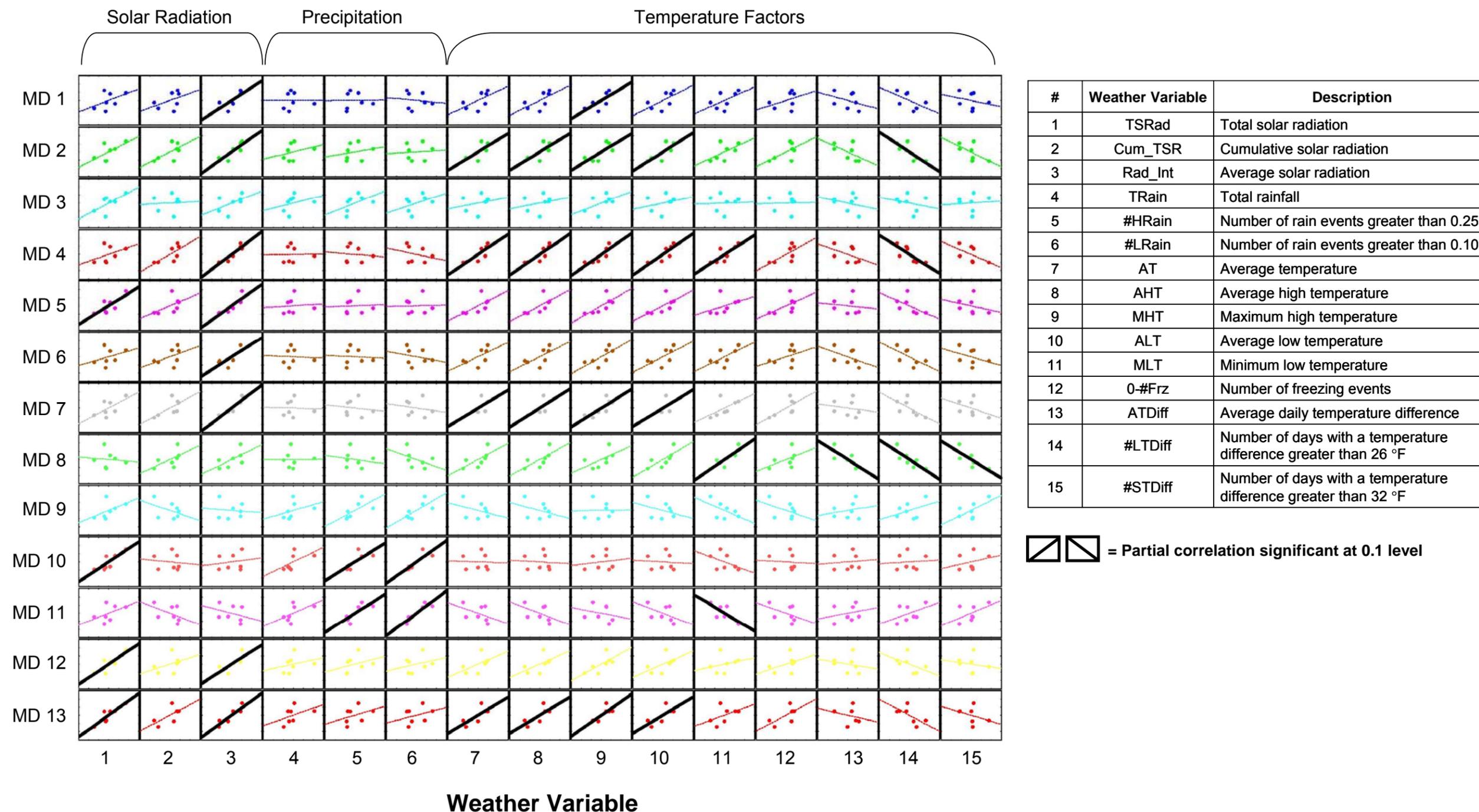


Figure 3-36. Partial Regression Plots of Coating x Weather Factor ([each subplot has ln(DAs) as its y-axis and the weather variable as its x-axis])

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The largest correlations (0.93) are associated with Rad\_Int; nine of the thirteen treatments correlated significantly with at least one of the radiation measures. Two of the products, both film-formers had a significant correlation with number of precipitation events greater than 0.1 and 0.25 inches, consistent with the expectation that shrink and swell due to moisture impact performance of film-forming products. Significant temperature correlations were observed for about one-half the treatments, and in nearly all cases, those treatments also responded positively to a solar radiation measure. No significant correlations were observed for products #3 and #9, products that performed well in terms of reduction of DCCA.

### 3.5 Coating and Paint Chip Sample Results

The results of the raw coating analyses are presented in Table 3-14 and expressed in µg of total arsenic, chromium and copper per g of raw (wet) coating and per g of dry coating. While the paints, coatings #9 and 10, had higher concentrations than did the other coatings, all of the concentrations were relatively low. A “U” entry in Table 3-14 is a qualifier indicating that the result was below reporting limits, meaning that the raw value of concentration for the digestion fluid was less than 2 µg/L.

Table 3-14. Raw Coating CCA Analyte Concentrations (raw/wet weight basis)

Coating	Dry Solids	raw concentration (µg/g)			dry concentration (µg/g)		
		As	Cr	Cu	As	Cr	Cu
1	25.7 percent	0.62	2.09	2.09	2.42	8.15	8.15
2	28.3 percent	U	0.32	0.59	U	1.13	2.08
3	44.2 percent	0.41	1.14	1.21	0.93	2.58	2.74
4	35.6 percent	U	0.46	0.78	U	1.29	2.19
5	8.8 percent	U	0.38	0.71	U	4.32	8.07
6	13.6 percent	U	0.74	1.57	U	5.46	11.58
7	10.3 percent	U	0.47	1.3	U	4.54	12.57
8	28.8 percent	U	1.23	3.21	U	4.28	11.16
9	55.4 percent	1.64	1.89	18.87	2.96	3.41	34.07
10	67.2 percent	3.99	20.48	6.86	5.94	30.49	10.21
11	48.1 percent	U	3.07	1.25	U	6.38	2.60
12	17.9 percent	U	1.08	0.42	U	6.03	2.34
Primer	34.1 percent	0.90	1.53	0.77	2.65	4.50	2.25

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By contrast, Table 3-15 summarizes the results of the paint chip sampling and analysis. There are several key points:

1. The concentrations of total arsenic, chromium and copper in the paint chips taken from the treated wood are significantly higher than in the paint chips from the untreated wood.
2. The concentrations of total arsenic, chromium and copper in the paint chips taken from the treated wood are significantly higher than in the raw paint, even after accounting for weight reductions due to volatile losses upon drying of the paints.
3. No matrix effects appear to be confounding the analyses. This is evidenced by the close agreement between the split samples of paint chips with different masses.
4. Additionally, archived paint chips samples from Paint 9 Treated and Paint 10 Untreated were manually examined using a LEO/Zeiss Model 440 SEM equipped with a PGT IMIX energy-dispersive X-ray spectrometer (EDX) for providing elemental composition information on individual particles. This examination confirmed arsenic associated with top and bottom surfaces of the paint chips recovered from the CCA-treated boards of deck 9. The full analysis report is included in Appendix R.

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**Table 3-15. Paint Chip CCA Analyte Concentrations**

Sample	Mass of paint chips (g)	Concentration (ug/g)		
		As	Cr	Cu
Paint 9 Treated	1.00	225	100	110
Paint 9 Treated	0.50	220	118	108
Paint 9 Untreated	0.70	13	8	29
Paint 9 Untreated	0.34	15	22	33
Paint 10 Treated	0.25	249	247	165
Paint 10 Treated	0.10	310	330	190
Paint 10 Untreated	1.00	12	32	16
Paint 10 Untreated	0.50	12	38	16

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## 4. Quality Assurance and Quality Control

### 4.1 Assessing DQI Goals

This section summarizes the assessment of data quality goals. The full QA/QC Summary report that includes all QC results is included in Appendix S. The critical measurements for the natural weathering tests are total arsenic, total chromium, and total copper concentrations. Data quality indicator (DQI) goals for concentration in terms of accuracy, precision, and completeness, as established in the QAPP for this project, are shown in Table 4-1.

Table 4-1. Data Quality Indicator Goals for Critical Measurements

Analyte	Method	Accuracy (Percent Recovery)	Precision (Percent RSD/RPD)	Completeness (Percent)
Arsenic (total)	SW-846 Method 6020 (modified)	90-110	10	90
Chromium (total)	SW-846 Method 6020 (modified)	90-110	10	90
Copper (total)	SW-846 Method 6020 (modified)	90-110	10	90

After reviewing sample results, the DQI goals for precision and accuracy have been revised for concentrations <10 µg/L. Acceptance criteria of ±25 percent RPD for precision between duplicates and 75-125 percent recovery for accuracy will be used for concentrations <10 µg/L.

#### 4.1.1 Precision

A large number of blind field duplicates (wipe samples split following extraction) were performed and delivered to the laboratory for analysis. These duplicates were performed at a rate of 7 percent of the total number of samples collected and provide an indication of the repeatability of the analytical method. The DQI goal for precision was set at ±10 percent RPD. For the majority of samples, agreement between field duplicates was very good (i.e., the RPD was small). The DQI goal of ±10 percent was increased to ±25 percent for samples with concentrations <10 µg/L because smaller differences in lower concentrations have a greater effect on RPD. With the modification, there were still some cases where the DQI goal was slightly exceeded. In only one set of duplicate samples was RPD >50 percent for each analyte; this was the only sample set where the data was qualified as estimated "J" due to the RPD. Achieved completeness was >80 percent for all three analytes, which did not meet the

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DQI goal of 90 percent established in the QAPP. There are no acceptance criteria given in the analytical method for agreement between duplicate samples. A DQI goal of  $\pm 15$  percent RPD may be more realistic for these types of samples. If DQI goals of  $\pm 15$  percent RPD, with  $\pm 25$  percent RPD for duplicate measurements where the mean was  $< 10 \mu\text{g/L}$ , are used, completeness would be greater than 90 percent for all three analytes. All data were used in the analysis.

#### 4.1.2 Accuracy and Bias

Spiked samples were performed by the laboratory at a minimum of three of four concentration levels with the samples from each sampling event. Those concentration levels were  $10,000 \mu\text{g/L}$ ,  $1,000 \mu\text{g/L}$ ,  $50 \mu\text{g/L}$  and  $1 \mu\text{g/L}$ . In addition to the laboratory spikes, ARCADIS/EPA provided  $1000 \mu\text{g/L}$  spikes (in triplicate) and submitted these blind to the laboratory. The DQI recovery goal for the  $1 \mu\text{g/L}$  spike sample was increased to 75-125 percent for the same reason the precision DQI was increased, as discussed in Section 4.1.1. Completeness was calculated separately for each spiking level and each analyte. Bias results generally improve as the concentration of the spike level is increased, although there were less spikes performed at the  $10,000 \mu\text{g/L}$  level. In an effort to improve completeness results, sample groups for the 15-, 20- and 24-month sampling events utilized a higher rate of spike samples than did the preceding events.

#### 4.1.3 Completeness

The ratio of the number of valid data points taken to the total number of data points planned is defined as data completeness. Completeness goals of  $> 90$  percent were not achieved for a number of measurements. Data results suggest that the DQI goal of  $\pm 10$  percent for precision between duplicates may be too ambitious. There is no specific acceptance criteria given in the analytical method for precision between duplicate samples. If the DQI goal were slightly increased to 15 percent RPD, completeness goals would have been met for all metals. Also, the analytical method cites acceptance criteria for recovery of spiked blanks as 85-115 percent which is slightly higher than the DQI goal of 90-110 percent. Using the analytical method criteria, completeness of accuracy results would improve. DQI goals should be reviewed and revised as appropriate for future studies.

#### 4.2 Data Validation Summary

The subcontract laboratory was required to submit calibration and QC data along with each data package. All data packages received by ARCADIS/EPA were internally

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validated by a qualified staff scientist according to the QA/QC criteria set forth in the U.S. EPA *Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, July 2002 (NFG). When parameters called out in the NFG were different from those established in the QAPP or the analytical method (EPA Method 200.8), the more stringent criteria were used. Validation reports were prepared for each sample delivery group and the reported data were qualified as appropriate. These reports are also included in Appendix S.

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## 5. Conclusions

This study evaluated the effect of coatings on DCCA on the surfaces of CCA treated wood.

Each coating, as well as the positive (uncoated) controls, exhibited a significant decrease in DCCA between baseline (prerinse and precoat) and samples taken 1 month after coating, demonstrating that rinsing provides some reduction in DCCA, however, the reduction is relatively small compared to the reduction attributable to the coatings. The coated minidecks all had lower DCCA than the positive controls, which indicates that coating (using any of the coatings tested) reduces DCCA to some degree (although not always at a statistically significant level). Over the course of the two-year study, DCCA increased with time after coating, although decreases in DCCA were sometimes observed between sampling intervals, presumably where the sum of removal processes (previous the wipe sampling event, precipitation, etc.) have greater impact on DCCA than the weathering and other factors that increase DCCA.

The coatings that were tested were compared against one another and uncoated, CCA treated positive controls based on their performance in reducing DAs. Several coatings exhibited DAs reductions estimated to be between (lower and upper 95 percent CI) 50 and 99 percent over the positive control minidecks for a period of time, typically through roughly the first year of weathering. All of these products fell in the “multi-coat” category and included two penetrating products; product #3, an oil-based stain with acrylic, product #8, a water-based stain with acrylic, and three film-forming products; the two paints, and product #11, the vinyl elastic coating.

The efficacy of each product decreased over the period of the study, and by 24 months, only two of the products had DAs levels that were significantly different than the positive controls. Additionally, the reductions of DAs for one product, a water-based sealant, were not statistically different from the positive controls over the entire course of the study.

Exploratory statistical tests that investigated differences among identifiable subgroups of coatings for the products tested (excluding the paints) indicate that, in terms of DAs reduction:

- stains performed better than sealants;
- products with acrylic performed better than products without acrylic;

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- non-alkyd products performed better than products with alkyd;
- film-forming products performed better than penetrating products;
- multi-coat products performed better than single-coat products; and
- the difference between products with clear vs. semi-transparent cover was not significant, and the difference between oil and water-based products was not significant at the 0.05 level when the paints are not considered.

The statistical evaluation suggests that in terms of reduction of DCCA, the most effective non film-forming product characteristics would include: multi-coat, oil or water-based non-alkyd stain with acrylic. This is consistent with the observation that products #3 and #8 were more effective than the other non film-forming products through the first year of the tests. It must be noted, however, that the number of representatives from each coating subgroup was limited and therefore, it must be emphasized that these results may not apply to other untested coatings within these subgroups. Furthermore, there is some overlap between subgroups so the statistical tests lack independence and there may be redundancy in the conclusions. Therefore, these observations may be best suited for forming hypotheses for experiments that isolate and evaluate specific variables or for testing of a broader selection of products with more representatives within the different product classifications. Either approach may reveal more convincing differences with respect to product characteristics, such as ingredients.

Although the products for which application of more than one coat was recommended (and applied) performed significantly better than the products for which a single coat was recommended, one cannot infer that multiple coats necessarily provide improved reduction of DCCA because the study design did not examine this question directly by testing multiple application levels for the products. Also, the lack of statistical significance between non-paint oil-based and water-based products does not imply that products perform equivalently; rather, only that differences in performance, if they exist, were not great enough to be detected in this experiment. However, one of the two most effective non-film-forming products tested included a water-based stain. This finding is of interest because oil-based products may be less available after 2006 in response to implementation of the Clean Air Act amendments restricting the atmospheric release of volatile organic compounds from architectural coatings.

While film-forming products performed significantly better than the non-film-formers in these tests, elevated arsenic concentrations were measured in paint chips recovered

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from the painted CCA-treated minidecks (note that CCA treated wood fibers may be associated with the paint chips). This finding is considered exploratory and warrants further investigation. The occupational exposures due to sanding and milling CCA-treated wood, as reported by Decker et. al., 2002, and Nygren et. al., 1992, underscore the need for research that evaluates potential exposures to CCA-chemicals due to preparation and maintenance activities, for instance, particle generation due to scraping and sanding of CCA-treated wood to remove cracked, chipped, or weathered coatings.

Taken as a whole, the results of this study suggest that typical deck coating products (sealants and stains) need to be re-applied periodically in order to maintain significant levels of DAs mitigation, perhaps on an annual basis. However, this study did not examine the effect on DAs of recoating after a period of weathering, and the results of this study may not apply to reductions in DAs following periodic re-treatment. However, the methodology should enable evaluation of this variable.

The striking differences in efficacy of products in reducing DAs suggests that products could be tailored to reduce DAs. Broad-based testing of products may be needed to empower consumers to make informed choices. The paint chip results also indicate a need to better understand and characterize the potential impacts of recoat preparation steps, particularly for film forming products.

The protocol utilized in this investigation yielded results that provide a coherent picture of the changes in DCCA as coated CCA wood weathers and, with appropriate development or modification, may serve as a useful tool for the coatings industry or product testing laboratories in efforts to develop new coatings and to verify coating performance for reducing dislodgeable CCA. Specific findings that may inform future use of the protocol include:

- There were significant differences in DCCA (pre and postcoat) between the two different source decks. That is, source deck is an important variable. Grain orientation, however, was not a significant variable in this study. This may be due to the minideck design, climate, or products that were tested.

Additionally:

- The effects of abrasion resulting from the wipe sampling method used for this study appear to be negligible, thus avoiding potential complications, or false positive interferences, as a result of the sampling methodology. However, rewipe

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effect – that is, the reduction in DCCA by “cleaning” the surface by sampling – may be significant.

- Cross-contamination controls (untreated boards between each treated CCA board on each minideck) showed that carry-over of DAs to the untreated boards generally increased substantially over time, but still represented only fractions of the measured DAs on treated (coated and uncoated) boards. As such, it may not be necessary to sample these cross-contamination controls, but they should be provided as buffers between boards.
- The data suggests that baseline (precoat) DCCA can be determined either for each specific wipe area or averaged over each board. Precoat measurements taken both before and after preparing the wood surface (e.g., washing, rinsing, etc.) for coating would illuminate the impacts of preparation steps and provide an unequivocal basis for initial precoat DCCA.
- As with any pilot experiment, there are many important variables that this study did not address, including: impact of stresses associated with full-scale decks, impact of climatic regions, abrasion, multiple coats of the same product, surface preparation procedures, and periodic recoat. These are all potentially important variables for which additional testing is recommended. Abrasion (e.g., resulting from foot traffic on in-service deck surfaces), in particular, is considered a likely important coating performance factor. Similar testing, including a realistic simulation of abrasion from foot-traffic, is needed.
- The study design did not control the time period for each wipe stroke. Differences in wipe period were due primarily to roughness characteristics of the sources and therefore, could not be standardized as the polyester wipe material occasionally hung-up on rough areas of the board. Thus, it is possible that for example, differences in DAs observed between source decks A and C may be due to factors associated with relative roughness of the two sources and the wipe technique.
- The sampling method does not differentiate between particles and soluble DCCA components unless collected particles are the size of a grain of rice, which are removed from the wipe prior to extraction. Therefore, ratios of DCCA components may be different than those observed in leaching studies because the wipes contain dislodged particles as well as soluble components. One cannot deduce the impact of particle collection on coating performance because the study design does not address this issue.

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- The study design does not directly address the impact of preparation steps. However, results from coatings 3 and 7 indicate that coating is much more effective than cleaning in reducing DAs through at least 15 months.

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