COOPERATIVE DEVELOPMENT OF AN

ASTM Accelerated Dirt Pickup Resistance Method with Correlation to Natural Outdoor Weathering

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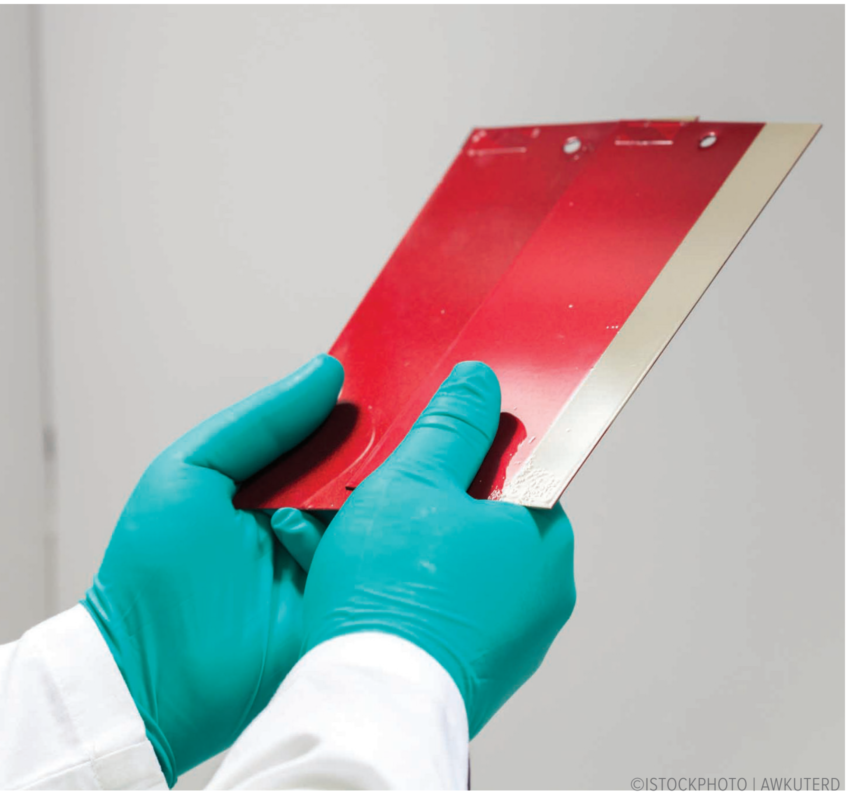


The Architectural Subcommittee of ASTM's Paint and Related Coatings, Materials and Applications Committee (D01.42), in collaboration with industrial partners, investigated an accelerated laboratory method to assess the exterior dirt pickup resistance of architectural coatings.

A concurrent exterior exposure study was conducted with seven commercial paints across seven locations with varied climates throughout the United States for 36 months. The accelerated laboratory method explored multiple variables including dry time of the coating and QUV exposure time.

For the exterior study, to understand the impact of multiple variables and their interactions on final response, a robust statistical methodology was applied utilizing both a mixed model with fixed and random effects to account for repeated measure analysis and a standard least square linear regression. Finally, the results from the two methods, laboratory and exterior, were compared to understand the similarity of the overall trends.

INTRODUCTION

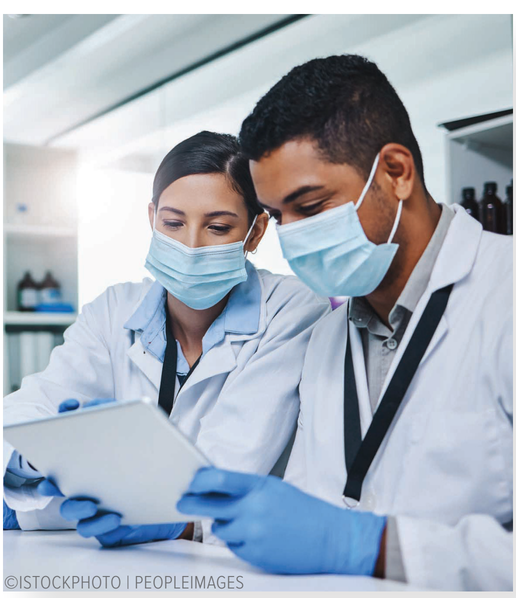
ASTM Subcommittee D01.42 is responsible for paint and coatings test methods, and dirt pickup resistance (DPUR) is an important performance attribute for exterior coatings. As the global emphasis on sustainability increases in the coatings industry, the reduction of volatile organic compounds (VOCs) for low-VOC latex paints is an industry imperative.

The coatings industry often employs nonvolatile coalescing agents and low-T_g polymers with a reduced need for coalescents, resulting in coatings that are especially prone to unsightly dirt collection. DPUR assessment by natural weathering is costly and time consuming.[1,2] Since DPUR is also a function of climate and air quality, careful coordination of exposure data collection across multiple geographical locations is key to the data quality and subsequent analysis and interpretation.

The ASTM Subcommittee D01.42 is committed to developing a standard method for accelerated DPUR with strong correlation to natural exposure. Early in the development of the accelerated method it was deemed critical to generate natural weathering dirt pickup data for comparative purposes.

Exterior exposures began in June 2015 and were completed by April 2019. Each study accumulated three years' worth of data. The exposures and data collection were carefully implemented across multiple geographical locations to ensure high data quality for analysis and interpretation. A preliminary laboratory study confirms the correlation of the proposed accelerated DPUR method with natural exterior exposure.

The completion of this work relied on the broad involvement from several paint companies and raw material suppliers in the program, summarized in the acknowledgments at the end of this article. Along with individual contributions, a wealth of corporate resources has been committed to enable this collaborative study. Participating companies included Arkema, ASTM Interlaboratory Study Program, Atlas, Azelis Americas, Boeing, The Dow Chemical Company, Eastman Chemical Company, Marschall Laboratories, PPG Industries, Sherwin Williams Company, and Wacker.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EXPERIMENTAL

Materials

Seven anonymous commercial white base paints were selected and purchased to be used for both natural exposure and accelerated laboratory testing. Samples of each paint were shipped to participating labs with the expectation that the same paints will be used in a future interlaboratory study to validate the accelerated methods. Paints were selected to represent three sheen categories (semigloss, satin, and flat) as well as two quality categories (standard and premium). The paint descriptions are summarized in *Table 1*.

TABLE 1—Description of Paints Used in the Study

PAINT ID	SHEEN CATEGORY	QUALITY CATEGORY
Paint 1	Flat	Premium
Paint 2	Flat	Standard
Paint 3	Flat	Standard
Paint 4	Satin	Premium
Paint 5	Satin	Standard
Paint 6	Semigloss	Premium
Paint 7	Semigloss	Standard

TEST PROCEDURES

Accelerated Lab DPUR Method

The accelerated lab method for testing DPUR was carried out using a drawdown of the test coating on an aluminium panel followed by a lab condition cure time of the coating, a standard QUV exposure period and an application of an aqueous dirt slurry. Soiled panels were then washed with water, and reflectance measurements were used to determine the tendency of the coating to retain the dirt.

The substrate used was an aluminium panel 10 x 15 cm. A drawdown applicator tool was used to create an approximate wet-film thickness of 5 mils. Cure time and QUV exposure time were varied. Cure time was either one or seven days at room temperature while QUV conditioning was for a duration of either one or five days.

The dirt slurry used in the experiment was an aqueous dispersion of brown iron oxide pigment. Panels were cleaned with gentle agitation under tap water. Two replicate panels were prepared for each paint under each testing condition. Y-reflectance (45/0) and L*a* b* (D65/10 CIE SPIN) were taken on panels before being soiled and after cleaning of dirt slurry application. ΔY (initial Y- final Y) and ΔL* (initial L*-final L*) values were calculated from the readings. Larger ΔY or ΔL* values indicate higher collection of dirt and poorer dirt pickup resistance.

Natural Weathering of Paints for Assessment of DPUR

Natural weathering was implemented for the ASTM study with paints exposed at seven different locations managed by different organizations and personnel. As a result, it is difficult to describe the exterior testing with a single method, and there are likely uncontrolled variables that are not known.

Exposure sites for the study included facilities across the United States with various climates representing northeastern, mideastern, southeastern, central, southwestern, and northwestern regions of the country. To manage differences in substrate and paint application technique, all paints were applied to panels from a single source at a single location. Paints were applied to 102 x 305 cm aluminum panels.

A drawdown applicator tool was used to create an approximate wet-film thickness of 5 mils. Dried panels were shipped to the locations with the intent to begin natural exposure within one week of paint application.

Panels were exposed in four orientations: horizontal up-5° facing south (S5), 45° angle facing south (S45), vertical facing south (SV), and vertical facing north (NV). Panels were exposed at four different start dates intending to represent paint application occurring during each of four seasons: June 2015, September 2015, December 2015, and April 2016.

Readings were taken at nine time intervals including: 0, 1, 3, 6, 12, 18, 24, 30, and 36 months. Multiple measurement types were taken at each time interval, including Y-reflectance (45/0), L*a* b* (D65/10 CIE SPIN), Gloss (20, 60, 80°), and subjective dirt and mildew by ASTM method D3274. Results from each exposure location were collected by the ASTM ILS committee and coded for exposure site anonymity.

RESULTS AND DISCUSSION

Statistical analysis of the results from accelerated DPUR and exterior weathering was conducted using JMP® Pro 15.1 software.

Results of Accelerated DPUR Single Lab Study

Variance components analysis with accelerated DPUR results was conducted with Paint ID, Dry Time, and QUV Time considered as input groups and ΔY as the response. The variability chart is shown in *Figure 1* and the breakdown of the variance components is illustrated in *Figure 2*. From *Figure 2*, Paint ID is the dominant factor determining the ΔY response, with Paint ID accounting for 96% of the variability.

Initial paint dry time and QUV exposure time do not appear to have a significant impact on the response. The variance components table in *Figure 2* indicate that variability from unexplained factors is low. In *Figure 1* the bottom row of the chart x-axis label identifies Paint ID and shows that paints across the series are well differentiated.

Linear regression analysis using Paint ID, Dry Time, and QUV Time as input variables results in a reasonably good linear model for ΔY as the output response. The summary of the whole model fit and ANOVA of the accelerated lab DPUR method is shown in *Figure 3*. R^2 and the F Ratio from the analysis indicate a robust statistical model.

FIGURE 1—Variability Chart for ΔY in Accelerated Lab DPUR Method

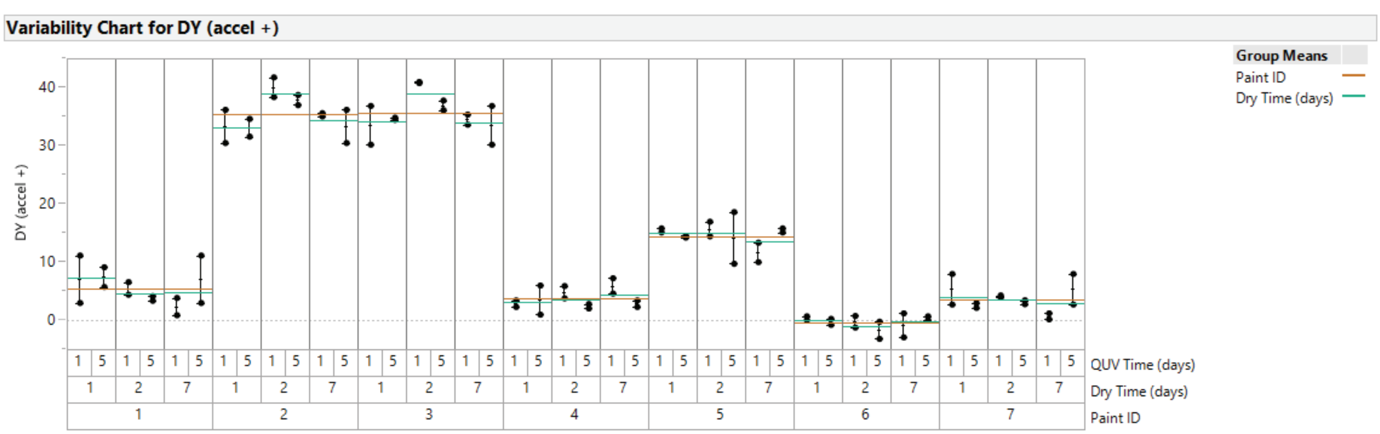


FIGURE 2—Variance Components for ΔY in Accelerated Lab DPUR Method

Variance Components

Component	Var Component	% of Total	20 40 60 80	Sqrt(Var Comp)
Paint ID	178.78979	96.1		13.371
Dry Time (days)	0.21587	0.116		0.465
QUV Time (days)	0.11615	0.0624		0.341
Within	6.92043	3.7		2.631
Total	186.04224	100.0		13.640

The least squares means predicted by the model for each paint were used to compare the performance of the paints against the accelerated lab DPUR method. Tukey-Kramer analysis was used to derive a connecting letters report contained in *Table 2*. Least-squares means numerical values are also shown in *Table 2*.

Although the paints used throughout the study are anonymous, the paints were categorized by sheen and by quality. It is notable that within each of the sheen categories the connecting letters report indicates significant differentiation between premium and standard quality paints. Paints identified with different letter classifications have significantly different responses for dirt pickup resistance by the accelerated method.

Results and Analysis of Exterior Weathering DPUR Study

The planning, design, and data collection for exterior exposure was significantly more complex compared to the lab study. This underscores the complexity of developing a lab method to simulate exterior exposure conditions when real world exposure conditions can vary widely and are inconsistent.

The team sought to capture as much variability in exposure conditions as possible by placing exposure panels at seven locations across the United States, facing panels in four directions at each location and starting new sets of panels at each location and direction during each of four seasons.

Measurements were taken at nine intervals from 0 to 36 months in order to capture DPUR over time. It should be noted that dirt collection and natural washing can be cyclical as weather and seasons change and as paints begin to degrade and erode. Several measurement types were taken at each reading to capture a full color and gloss assessment, and the subjective ASTM method D3274, which includes separate evaluation of dirt and mildew growth, was also used. Mildew growth could be considered a confounding factor if severe mildew failure is observed. Mildew ratings are not presented in our DPUR analysis in this case because significant or consistent mildew growth was not observed.

The final data set contains more than 11,000 rows of data out of a total of 14,000 possible rows from the planned experiment. Missing data exists from each of the locations and occurs across time points and directions. ΔL^* data appeared to be the most meaningful measurement collected from the exterior data set. A significant concern was found with Y-reflectance as a disparity in how Y-values were collected across the participating labs which resulted in unusable Y-reflectance data from the exterior exposure.

ΔE values were calculated from $L^*a^*b^*$ and modeled as well but showed less differentiation across the paints. Although differences were observed in dirt collection from the directions of exposure, the relative comparisons between paints were similar. The paints that collected the most dirt in one direction also tended to collect more dirt in the other directions.

The angle of orientation appeared to have most significant impact on the dirt collection potential and follows the anticipated trend. S5 (5° from horizontal-pointed south), which is nearly face-up, led to the most dirt collection. S45 (south facing at a 45° angle) accumulated nearly the same amount of dirt

TABLE 2—Results of Accelerated Lab DPUR Method by Paint ID, Sheen Category, and Quality Category

PAINT ID	SHEEN CATEGORY	QUALITY CATEGORY	TUKEY HSD CONNECTING LETTER REPORT	LEAST SQUARE MEAN
Paint 1	Flat	Premium	C	5.50
Paint 2	Flat	Standard	A	35.49
Paint 3	Flat	Standard	A	35.67
Paint 4	Satin	Premium	C	3.71
Paint 5	Satin	Standard	B	14.45
Paint 6	Semigloss	Premium	D	-0.42
Paint 7	Semigloss	Standard	C	3.50

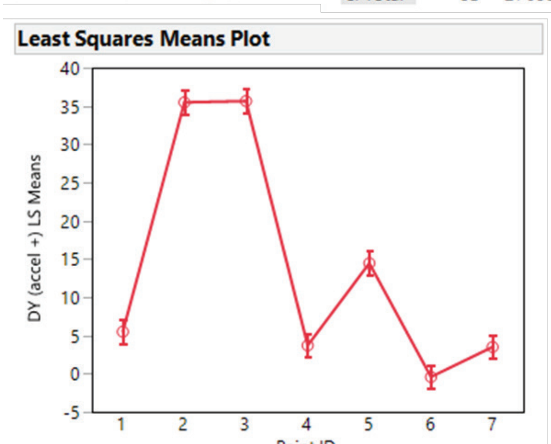
FIGURE 3—Accelerated Lab DPUR Least-Squares Whole Model Fit Output

Summary of Fit

RSquare	0.969614
RSquare Adj	0.965919
Root Mean Square Error	2.695322
Mean of Response	13.98373
Observations (or Sum Wgts)	84

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	9	17154.584	1906.06	262.3713
Error	74	537.592	7.26	Prob > F
C. Total	83	17692.176		<.0001*



as S5. Both vertical positions, south vertical and north vertical, collected significantly less dirt and were similar to each other.

The similarity between south vertical and north vertical is important because it suggests minimal contribution from mildew, as mildew is more prevalent on northern-facing exposures because dry times are extended due to lesser direct sunlight. Not only does the S45 data have one the highest degrees of dirt collected, it is also the most consistently populated data set.

Figure 4 plots the exposure data (ΔL*) over time by Paint ID and exposure direction. The results show that S5 and S45 were more aggressive to accumulate dirt and Paints 2, 3, and 5 appeared to have higher delta values than the other paints. Although delta values were generally increasing with time, the trend was inconsistent across Paint ID. The data contained in the plot also includes parameters that are not shown such as exposure sites, start date, and replicate panels, which could have a significant impact on the variation of results.

DATA ANALYSIS/MODELING

The database generated from natural weathering over 36 months was large with many inputs. Statistical data analysis was conducted to identify patterns and factors that influence weathering. Two approaches are described here: (a) Analysis of variance (ANOVA) via standard least squares linear regression modeling and (b) Mixed Model approach for Repeated Measure Analysis (RMA). Input and output parameters were treated slightly differently based on the modeling approach.

For standard least square linear regression method the response ΔL* values were separated into two bins based on the exposure time. Exposure times 18 months or greater were considered late whereas times less than 18 months were considered early.

Input variables and their levels for analyzing ΔL* values from late times are listed in the left side of *Figure* 5. Overall model fit and model fits by directions under the condition late times are shown in the right side of *Figure* 5. Considering the complexity, the R^2 values were reasonable, and the models could explain up to 76% of the data based on direction of exposure.

Plots of least squares means for comparing impact of input variables from main effects and interactions are shown in *Figure* 6. Two directions, S5 and S45, were more aggressive compared to NV and SV. Similarly, exposure starting in April and December was more aggressive compared to starting in either June or September. Paint IDs and their interactions with locations could be very significant. For example, Paint 5 had the highest value of ΔL* associated with Lab C whereas Paint 3 had significantly higher ΔL* values at Lab D and Lab G.

CORRELATION OF PROPOSED ACCELERATED METHOD WITH EXTERIOR EXPOSURE

Comparisons of accelerated lab test and natural exterior weathering from S45 direction based on least square mean values from linear regression are shown in *Figure 7*.

FIGURE 4—ΔL* Value vs Exposure Time and Direction

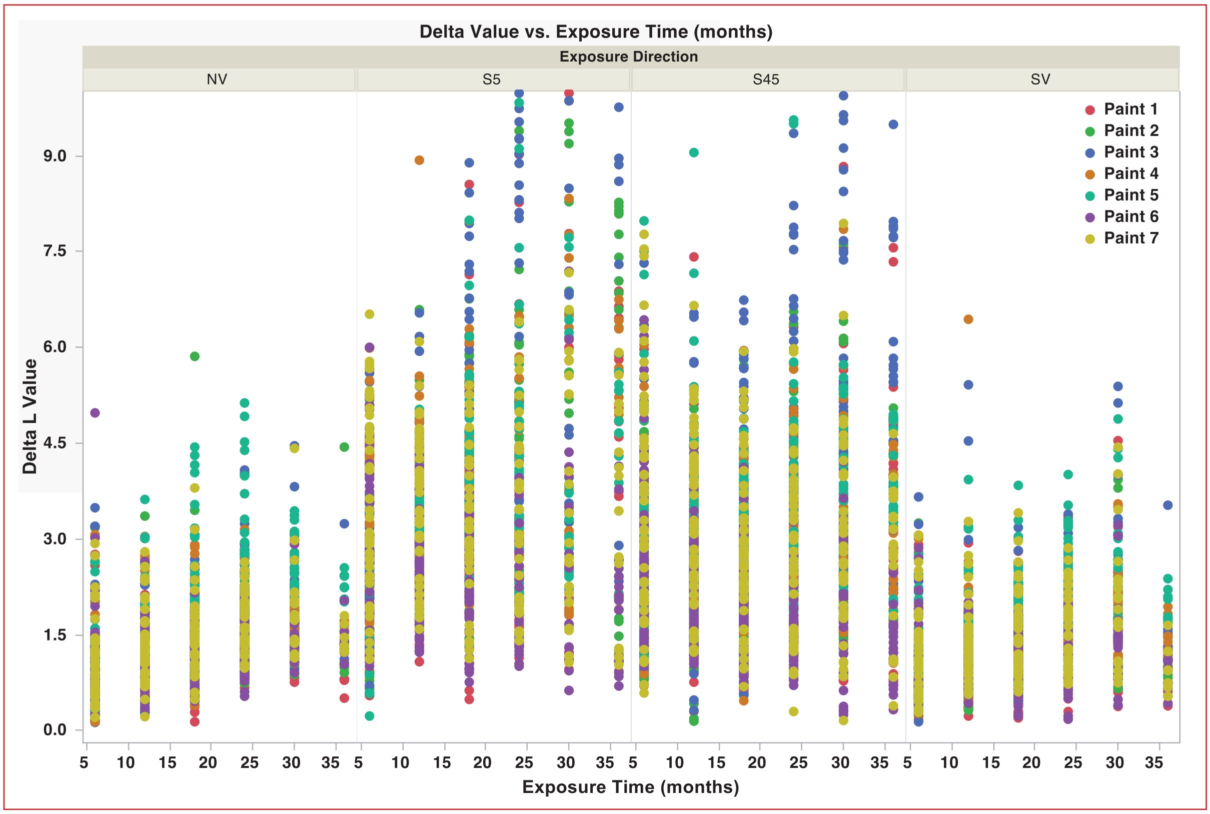


FIGURE 5—Standard Least-square Linear Regression Input Variables and Model Summary

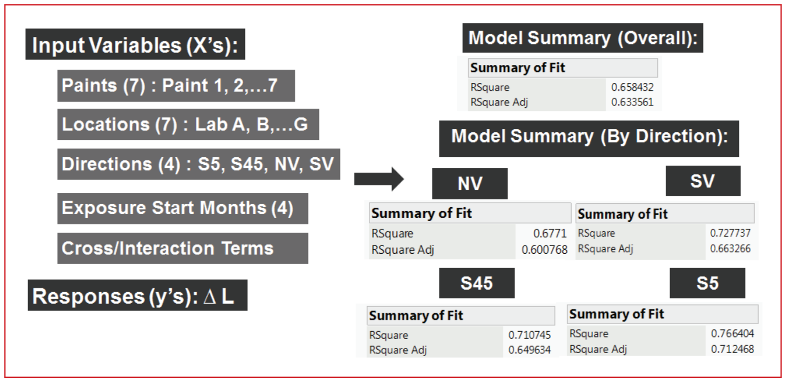


FIGURE 6—ΔL* Late Data Model: Least Squares Means Plot for Comparing Impact from Input Variables

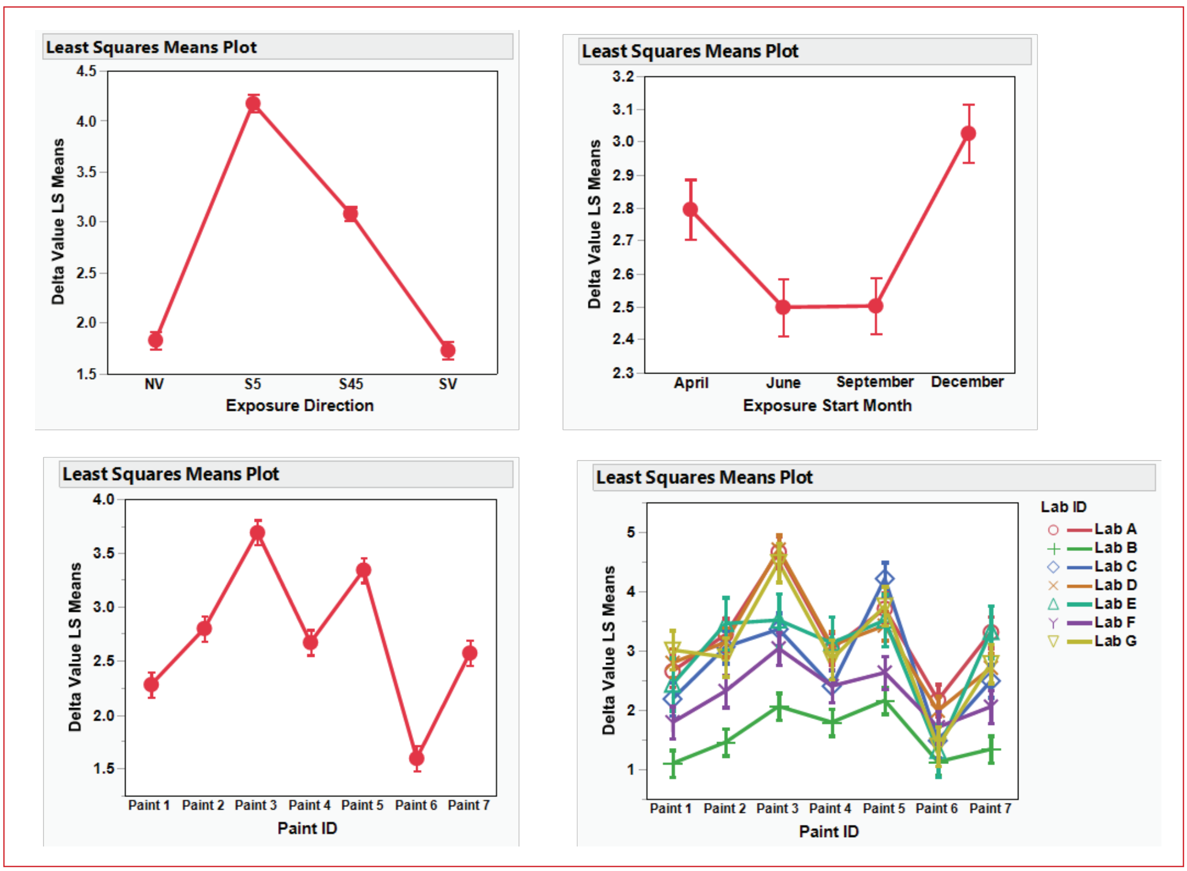


FIGURE 7—Least Squares Means Plots by Paint ID in Accelerated vs. Natural Exposure

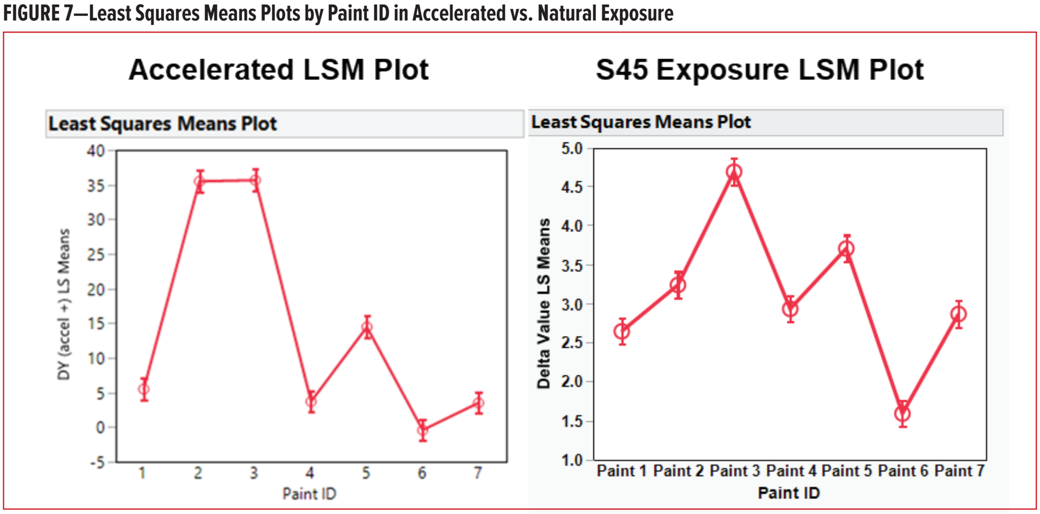


Table 3—Comparative Results of Accelerated Lab and Natural Exposure Dirt Pickup Resistance Methods

PAINT ID	SHEEN	QUALITY	ACCELERATED DPUR		EXTERIOR L* (FROM S45)	
			Tukey HSD Connecting Letter	Least Squares Means	Tukey HSD Connecting Letter	Least Squares Means
Paint 1	Flat	Premium	C	5.50	D	2.65
Paint 2	Flat	Standard	A	35.49	C	3.24
Paint 3	Flat	Standard	A	35.67	A	4.69
Paint 4	Satin	Premium	C	3.71	C D	2.93
Paint 5	Satin	Standard	B	14.45	B	3.71
Paint 6	Semigloss	Premium	D	-0.42	E	1.59
Paint 7	Semigloss	Standard	C	3.50	D	2.87

Table 3 lists the least square mean values and connecting letters report from Tukey-Kramer for mean comparison.

While results for individual paints do not correlate well between the two methods, premium and standard paints are differentiated within each sheen category by each of the DPUR methods. In *Figure 7*, Paint 2 is the most apparent example where a different response relative to other paints is observed between the two methods.

Mixed Model Approach for RMA

RMA modeling provided a unique way to analyze exterior data by utilizing the entire time sequence to enable the assessment of time-ordered aging effects. While contrasting two time points (i.e., data at late exposure times versus the initial data at 0 months) could provide an understanding of a single difference between these two time points as discussed in the previous section, considering all the data over the entire time period provided additional understanding of the rate of change expected over time.

Here the L* value as a response was measured repeatedly over time on each panel. Hence, this type of RMA associated with the entire time sequence could be analyzed as a mixed model, known for its flexibility in handling different covariance structure and missing data.

Other fields of study where a measurement is recorded more than once over time from the same subjects have successfully applied RMA by a mixed model approach such as crop growth, neurotoxicology, and pharmacology.[3,4] For this dataset, ΔL* values from 6 months to 36 months were considered and transformation as shown in *Figure 8* was applied to avoid skewness so the model fit could be applied over a normal distribution.

The RMA model fit in this data was considered a mixed model because both fixed and random effects were contained within it. For examples, variables like Paint ID, location-direction combinations, exposure starting month, time, and two factor interaction were considered fixed effects, while variables like panel were considered random effects.

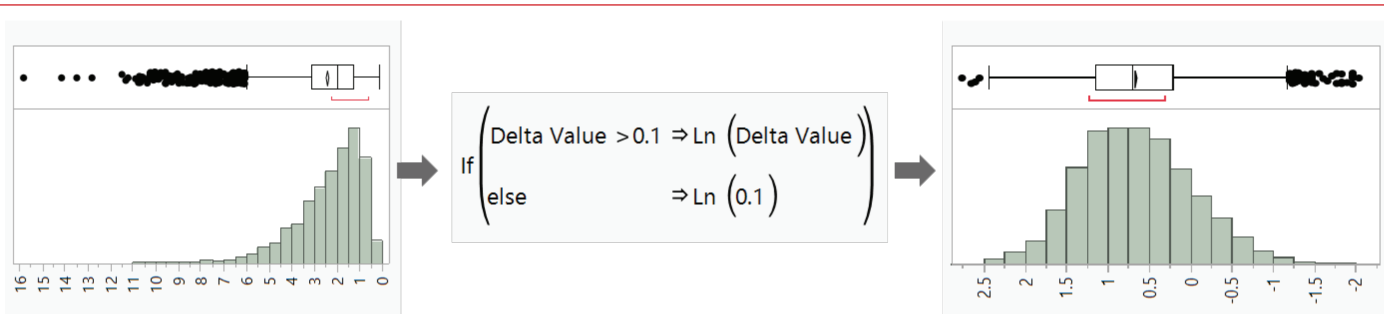
Figures 9 and *10* show the models associated with the transformed ΔL* data. *Figure 9* presents two versions of the actual by predicted plot that can be used as a general visual assessment of the model fit. The plot on the left is the typical actual by predicted plot that can be thought of as representing the model not accounting for differences across the paint panels.

The actual by predicted plot on the right labeled "Conditional Predicted" is a representation of the model accounting for the observed differences between the paint panels. Both plots show that there was still considerable unexplained variation even after fitting this RMA model.

Figure 10 provides some clarity regarding the paint panels' "aging" behavior over time while considering the significance of the fixed effects. The Random Effects Covariance Parameter Estimates table suggests significant variation between the paint panels at initial times (time = 6 months) because the Var(Intercept) term is statistically significant (p-value < 0.0001)).

Likewise, the Wald p-value next to the Var(Exposure Time) term (p- value < 0.0001) suggests that the slopes of the paint panels were significantly different from zero. The p-value for the "Covariance" term (p value < 0.0001) suggests there is evidence of a relationship between the ΔL* at the start and its subsequent rate of aging.

FIGURE 8—Data Transformation to Achieve Normal Distribution



In addition to testing the random effects, statistical testing can be performed on the fixed effects within the Fixed Effects Tests table. Again, small p-values (typically less than 0.05) suggest significant impact from the fixed sources.

The results in *Figure 10* demonstrate that the location (lab-direction combination), Paint ID, and interaction term (location and Paint ID) had a significant impact on response. In addition, there was a significant difference found between starting month of exposure initiation and duration of exposure. Although the fit of this RMA model is not stellar, it still provides several interesting results consistent with past experience and previous weathering knowledge.

FIGURE 9—RMA Mixed Model Actual by Predicted Plots

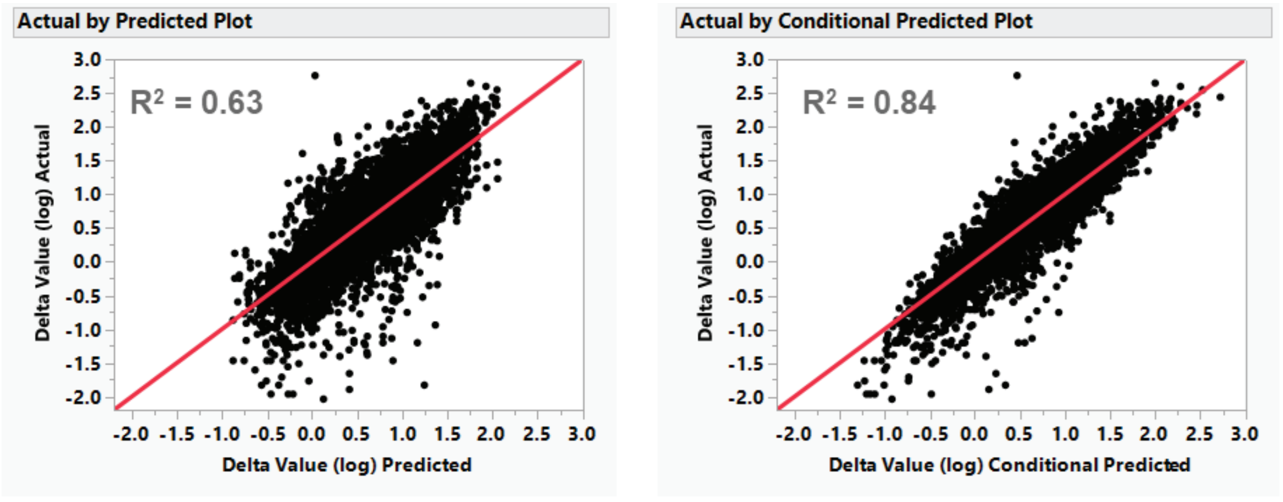


FIGURE 10—RMA Mixed Model Random Effects Covariance Parameter Estimates and Fixed Effects Tests

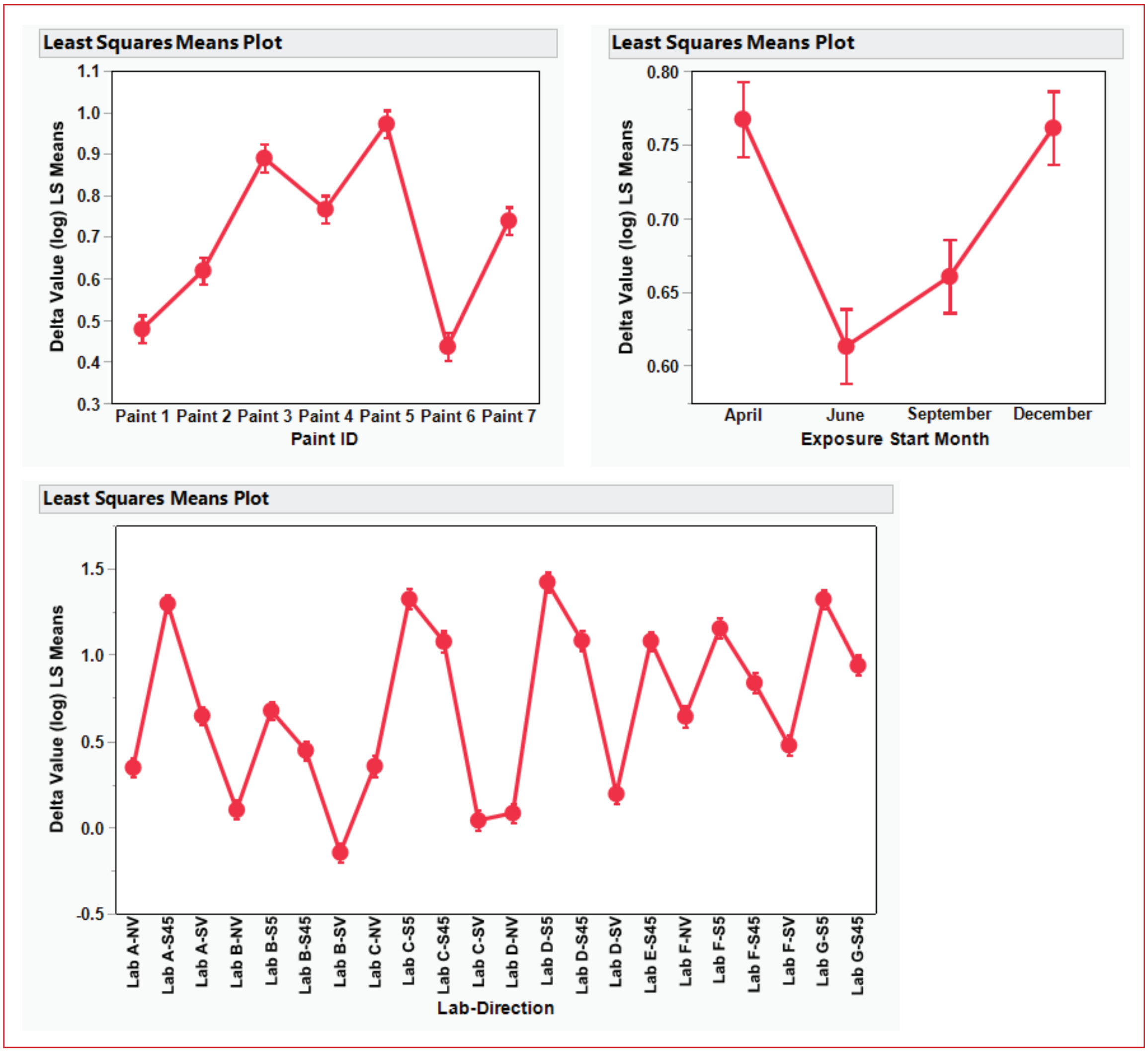
Random Effects Covariance Parameter Estimates

Covariance Parameter	Subject	Estimate	Std Error	95% Lower	95% Upper	Wald p-Value
Var(Intercept)	Paint Panel	0.2535945	0.0177461	0.2188128	0.2883762	<.0001*
Cov(Intercept,Exposure Time (months))	Paint Panel	-0.011702	0.0007715	-0.013214	-0.01019	<.0001*
Var(Exposure Time (months))	Paint Panel	0.0005845	3.8829e-5	0.0005084	0.0006606	<.0001*
Residual		0.1037438	0.0025822	0.0988632	0.1089967	

Fixed Effects Tests

Source	Nparm	DFNum	DFDen	F Ratio	Prob > F
Paint ID	6	6	1011.2	150.82418	<.0001*
Exposure Start Month	3	3	1098.2	47.470164	<.0001*
Exposure Time (months)	1	1	1125.9	541.6291	<.0001*
Lab-Direction	21	21	984.5	288.28484	<.0001*
Paint ID*Lab-Direction	126	126	968.4	4.1007695	<.0001*

FIGURE 11—Fixed Effect Least Square Means Plots



Fixed effects were compared by plotting least squares means as shown in *Figure 11*. For Paint ID, Paint 1 and Paint 6 were expected to show significant better performance (low ΔL*) compared to Paint 3 and Paint 5. Similarly, exposure started in either April or December would show more aggressive impact on ΔL* compared to June or September as a starting time. Irrespective of labs, two directions (S5 and S45) would be more aggressive, and ΔL* values were higher compared to the other directions.

Interaction plots between lab-direction and paints show "more" or "less" differentiation between paints based on locations as shown in *Figure 12*. For example, the Lab A-NV location-direction combination showed less differentiation between paints whereas in Lab A- S45 and Lab D-S5 location-direction combination the paints were well separated. The plot also shows that Paint 3 had the highest and Paint 6 had the lowest values in most location-direction combinations.

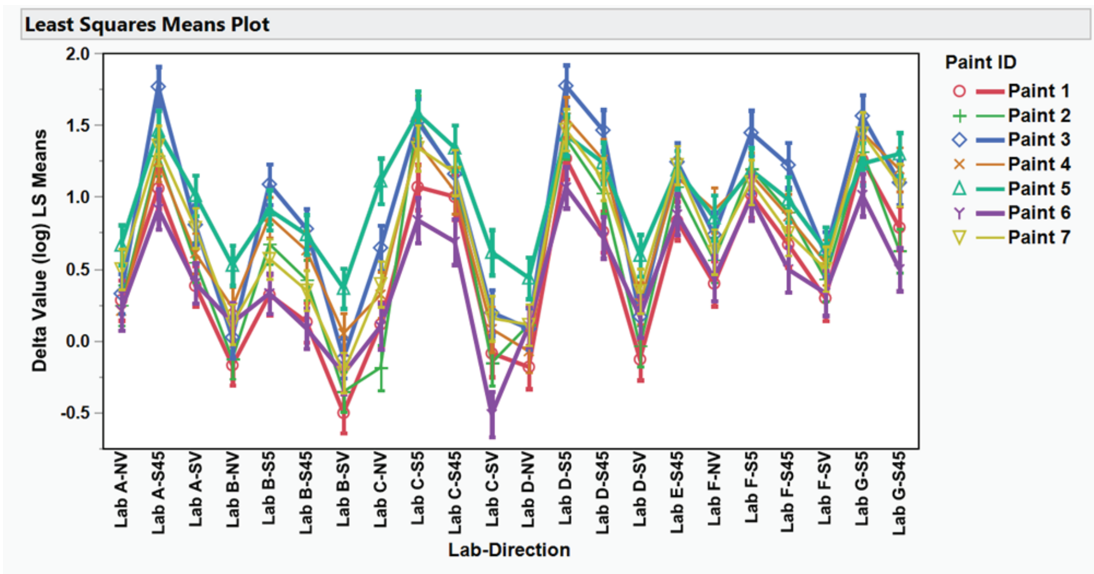
CONCLUSION

The results of a single lab study of the proposed accelerated DPUR lab method finds good differentiation between the seven paints chosen for the analysis. Paints selected as premium quality demonstrate significantly whiter results within each gloss/sheen category which indicates premium paints have better dirt pickup resistance. Results of exterior weathering also find paints selected as premium quality demonstrate significantly whiter results within each gloss/sheen category after extended exterior exposure in varied U.S. climates.

The exterior weathering data set is complex and has required extensive analysis and modeling of the data, but the data consistently finds significant effects across the paints tested and other identifiable variables in the study. While horizontal exposure (S5) accumulates the most dirt on exterior exposure, the south-facing 45° angle (S45) exposure data set is the most preferred single direction set because all locations are included for the exterior testing. The S45 direction also shows the most differentiation for the paints evaluated.

A single comprehensive and robust statistical model was derived considering repeated measure analysis and applying a mixed model with fixed and random effects. The single model includes data points from all paints, labs, directions, start months, and exposure

FIGURE 12—Interaction Plot for Lab-direction vs. Paint ID



times from 6 to 36 months. The combined analysis of these studies provides sufficient confirmation by correlation of the accelerated lab and natural weather results to justify progression of the proposed accelerated DPUR lab method to a full ASTM interlaboratory study.

THE PATH FORWARD

The Architectural Subcommittee of ASTM's Paint and Related Coatings, Materials and Applications Committee (D01.42) will continue with the development of a standard test method for accelerated dirt pickup resistance. The team is currently revising the standard test method document. Once the test method document is agreed upon, the team will cooperate in a formal ASTM-led interlaboratory study (ILS) of the lab method. The ILS intends to include a similar number of labs (7) where each lab will complete the proposed test method in duplicate. Provided the ILS finds good reproducibility and repeatability, the new ASTM standard method for accelerated dirt pickup resistance will be published.

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