OUTDOOR WEATHERING MUST VERIFY ACCELERATED TESTING

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ABSTRACT

Outdoor exposure racks and accelerated chambers are the two main methods for testing materials for weathering durability. In the fast paced current world, time constraints have determined that outdoor weathering exposures take too long to provide the necessary results. The emphasis has therefore been placed on accelerated laboratory testing to provide data on the long-term outdoor durability of many products. In many cases now, outdoor exposure is not even performed or required.

Not all of the factors of weathering can be recreated in an accelerated chamber however, and there are a number of other variables that may cause an incorrect result and therefore the wrong conclusion. Cycle times and the effect on the exposed specimens are different between the outdoors and the chamber. Over-exaggerated test conditions may also cause the wrong failure type. Too much reliance only on accelerated testing can be potentially misleading.

In this paper we will discuss the inherent problems that accelerated testing can have, and show techniques that can be used to ensure that the results from the accelerated testing are correct. Outdoor weathering must be used to verify the accelerated testing, and it is possible with the correct testing procedure to get meaningful data from an outdoor test in a short time frame. Those techniques include correct experimental design, proper evaluations, control materials, and the use of reliable statistical analysis. These processes will allow the user to determine acceleration and correlation factors and will ensure that the accelerated testing is giving the right results.

Introduction to Weathering Testing

All testing currently is done using either an outdoor exposure location or an indoor accelerated laboratory test. These two major test types are not the same, as accelerated tests are made with controlled artificial light sources in the laboratory, whereas natural weathering is performed in large outdoor fields with real sunlight but where all the exposure parameters are not controlled. See Table 1 for a comparison of the outdoor tests and accelerated tests. The choices that are laid before us are whether to use the fast, controlled, exposure that uses simulated conditions, or the slow, non-controlled, test that is known for its realism. For many, the choice has been to pick speed over substance.

Tuble 1. Comparison of Recelerated and Outdoor Tests		
Accelerated Tests	Outdoor Tests	
Testing chamber	Racks in large fields	
Controlled conditions	Non controlled conditions	
Artificial light source	Natural sunlight	
Simulated weather conditions	Real weather conditions	

 Table 1: Comparison of Accelerated and Outdoor Tests

When making the decision over which type of testing program to follow, there may be a number of "psychological" factors that unintentionally affect the outcome. In order to make a more informed and reliable choice about testing, we need to understand those biases, or what we might call Weathering Myths.

Note. A myth is an unproven legend, a story that has been around for so long that it takes on a truth of its own.

Weathering Myths

1. The Myth: Accelerated tests are 100% repeatable. The truth is that accelerated tests are as variable as any other test. In fact accelerated weathering, even in the most controlled test, can produce a great deal of variability in the results. Repeat tests in the same device and reproducible tests in different laboratories may have high differences. A major round-robin (ring study) was performed by ASTM Committee $G03^1$ in the 1990s and found that there could be as much as 30% difference in results from several devices. This difference is reduced with correct operation.

2. The Myth: Any Degradation is good. The truth is that the wrong degradation mode is a bad result. If the failure mode in the real world or outdoor test is not reproduced by the accelerated test, then the accelerated test should not be used. For example, if a coating normally exhibits moderate chalking outdoors but displays severe cracking in the accelerated test, this shows that a different failure mechanism is occurring and the results are therefore meaningless. The accelerated test chamber must create the same failure type as the outdoors.

3. The Myth: Outdoor weathering requires 5 yrs to provide useful data. The truth is that outdoor testing can yield useful data in 12 months. While it may be necessary to wait 5 years to find out 5 years of degradation, there is much useful information that can be obtained in the first 12 months of exposure, such as the distinction between good and bad material. The truth is that with the correct experimental design and plentiful evaluations, meaningful data can be obtained which will greatly increase the confidence of the accelerated data.

4. The Myth: Weathering test data is absolute. The truth is that no single test will yield perfect correlation. There are a number of material specifications that require a single test specimen to be exposed and where a pass/fail threshold is set. For example, if a color difference of 3.0 delta E units is the criteria, then a value of 2.9 passes, but 3.1 fails. Yet we know the variability of the color measuring equipment is such that any comparison where the color difference is less than 1 delta E unit is meaningless. The limit of human perception is also no better than 1.0 delta E.

5. The Myth: Ranked data is weak data. The truth is that ranked data can be powerful if correctly applied. There is a thought that non-parametric statistics, such as ranking, cannot be of any use because they are based on less powerful techniques. When properly applied, ranked data can be used to prove many of the hypotheses we test in weathering. Ranked data works well with numerical data produced in subjective evaluations.²

Unforeseen Problems

The problem we are facing as a testing industry is that many companies perform only the accelerated weathering tests on their materials. The outdoor testing is either given second class status, or is just simply ignored. The time related pressure of moving a new material to market has forced a reduction in the development cycle and this affects the durability testing. Most product development now is done with only the results from accelerated testing, and many material specifications will accept a material with only accelerated test results.

Using ONLY accelerated testing can have unforeseen problems. The main consequence is that the material is formulated for resistance to an artificial climate. As the cycle of testing, reformulation, and retesting continues, the end result is a material that can withstand exposures to a simulated and controlled environment. This can lead to field failures when the material that has passed testing in the laboratory then fails in the end use environment.

Accelerated tests are not 100% reliable in predicting what will happen outdoors. The search for a universal test method with perfect correlation is our industry's equivalent of the quest for the Holy Grail. Correlation factors are not transferable from one material to any other or for the agreement to a different outdoor climate. If we could be sure of a complete agreement in an accelerated test, the only need to conduct weathering outdoors again would be if we were comparing to a new climate. But even repeat tests where nothing has supposedly changed will give different results. Using only accelerated testing means taking a big risk when trying to determine the true reliability of the material.

Correlation

For the purposes of weathering testing, correlation is defined in ASTM G113 as follows:³ "The agreement of results between outdoor and accelerated tests." Therefore, the results from the accelerated test are supposed to match the results from the outdoor test. Most research effort into this elusive target has focused on the accelerated tests, but it is apparent that the key to correlation is the outdoor test. Without the outdoor weathering as a baseline, there is nothing to provide a fixed comparison for the accelerated test.

Costs

It is surprising that one of the major excuses for not doing the outdoor test is the cost factor. This should be the least of any worries and while it may not be a technical issue, we do recognize that many important technical concerns are not being addressed because of the misconception that weathering testing is expensive. What should be considered is the cost of not testing.

Note: These figures are representative and do not reflect any particular charges. Testing costs may differ amongst different commercial providers of these services, as well as between commercial services and conducting the testing yourself.

Table 2 below compares a typical outdoor test cost to the cost of not doing a test. For the upfront saving of up to \$1,000 per year you could be risking your company's reputation as well as the potential for millions in warranty replacements, and potential litigation. The small investment in outdoor weathering tests should be compared to the potential costs of not testing.

Table 2: Cost Comparison of Testing		
Factor	Testing	Not Testing
Cost of Test	\$500 to \$1,000 per test	\$0 per test
Downside	None	Potentially \$millions
Customer Service	Happy Customers	Unhappy Customers
Image	Great Reputation	Loss of Reputation

Cyclic Conditions

One of the reasons why we see many reports indicating poor correlation is because the factors of weathering are different in the weathering chamber than in the outdoor environment. The conditions within the chamber can cycle between any of the conditions in the following list but there are many factors missing.

Table 3 shows that the three basic factors of weathering are included in most accelerated weathering test devices. The right-side column shows that there are many more factors that can

influence weathering outside. Each new factor gives the possibility for many more related interactions.

Table 3: Factors of Weathering Comparison	
Accelerated Test	Outdoor Test
Artificial Light	Sunlight
Temperature	Temperature
Water Spray	Rain
	Condensation
	Humidity
	Biological
	Acid Deposits
	Dirt and Contaminants

Cycle Times Compared

Even when we look at only those factors that occur in both the outdoor and the accelerated test, there is a difference in the cycle timing. For outdoor testing we are dealing with one complex cycle per day, whereas in the accelerated device, we are dealing with multiple, complex, repeating cycles per day. A summary of the differences in the cyclic conditions can be found in Table 4 below.

Table 4: Comparison of Cyclic Effects		
	Accelerated Tests	Outdoor Tests
Average Cycle time	2 - 4 hours	24 hours
Cycles per day	6 -10 cycles	1 cycle
Dark Period?	Possible	Always
Cycle Consistency	Same every time	Different every time

Cycle Rate Comparison

Compared to the outdoor environment where there is only one cycle per day, there may be as many as 12 complete cycles in the accelerated test. We have known for some time that cyclic exposures tend to be more severe than steady-state exposures. It is the changeover from one steady state condition to another that puts the material under stress and causes degradation. Therefore, faster turnaround in the cycles should increase the degradation of the material because it is being subjected to many more of the conditions that may cause degradation. In reality, this may or may not be true.



Figure 1: Environment and Material Equilibration

As conditions change, the exposed material will be attempting to equilibrate to its surroundings. For example, if the material is wet and the atmosphere is dry, the material will become drier. Another case is if a cold material is placed in a warm environment it will begin to warm up. The environment contains the possibility of changing faster than the material. If the material does not

equilibrate to the conditions before the conditions change, the materials will never reach a steady state (see Fig 1). All materials will have a different rate at which they can adapt to the changing conditions around them. So it is quite possible that in the same exposure, some materials will equilibrate and some will not. Some materials will not completely attain the steady state.

If the material under test does not attain steady state equilibrium with the surrounding atmosphere, the effects will not penetrate into the depths of material. Short cycles will only have an effect on the surface layers. To reach the interior of a specimen, it is necessary to lengthen the cycle times in order for the whole of the material to be under the influences of the exposure conditions (see Fig 2).



Figure 2: Compare Cycle Effects on Material

This difference can be summed up by the term "Chamber Factor". This implies immediately that the changing conditions inside the weathering device cannot match the outdoors unless the cycle times and the ramping rates are identical to the outdoors. If the changing conditions do not match the outdoor the effect on the material will be different.

We can draw the following inferences from this. First, it is clear that weathering chambers cannot match the complexity of the outdoors. There are more variables in the outdoors, and the cycle timings are significantly different. Second, as a consequence, enclosed chambers and open spaces may not yield the same results

Accelerated Test Design

Once we comprehend the shortcomings of accelerated testing, we can plan our approach to creating an accelerated weathering test program. This is a bigger commitment than simply slipping some specimens into the first accelerated weathering device we find with some extra space. The following is the basic outline of a plan that can be followed to get the best possible test cycle.

- 1. Accelerated test conditions and cycles should be developed using a plan of simulate first, then accelerate.
- 2. Determine the extremes in the weathering factors. For example, highest and lowest temperatures, maximum irradiance, relative humidity ranges.
- 3. Determine the response rates of the material. What is the rate of change in moisture content when moving from a 50% to a 100% RH atmosphere (and in the reverse direction)?
- 4. Determine the minimum cycle time based on the material reaching the steady state condition

- 5. Consider an unbalanced cycle (such as the SAE J2527) in lieu of the same-old repetition (such as the 102/18 cycle).
- 6. Once a test method is demonstrating some promise, the test can be repeated to prove repeatability
- 7. Ruggedness testing, by increasing one parameter at a time, can be used to determine the limits of acceleration.

How Much Acceleration?

When you have zero acceleration in the simulated test, you can expect to get close to 100% agreement. As the degree of acceleration increases, the obvious reaction is that there will be a corresponding decrease in agreement. In an ideal test there will be a direct relationship between agreement and acceleration, but the typical situation is that there are thresholds. This is where the agreement remains pretty good for a certain amount of acceleration, but then drops off dramatically (see Fig 3). This is due to one of the influencing factors exceeding a critical point.



Figure 3: Simulation, Acceleration, and Correlation

Outdoor Weathering

How do you make sure that you get good data in a meaningful time frame? The design of the outdoor test must be done correctly before the test is started. Your outdoor weathering program also requires some advance planning. The objectives of the test must be determined before you begin your exposures. The following table gives a list of good practices to follow in order to maximize the success chances from the correlation.

- Begin testing as soon as possible.
- Use many replicates.
- Evaluate regularly and often.
- At least 5 evaluation intervals per test.
- Use control or reference specimens.
- Establish a baseline of material degradation.
- Typically 12 to 24 months sufficient.

Specimen Matrix

The typical experimental material variable array is unbalanced. This will cause problems as large amounts of raw data need to be reduced into groups in order to see patterns develop. For many years we have heard that the right way to do an exterior exposure test is to expose as many specimens as possible, as quickly as possible. If not planned correctly though, the excessive amounts of data collected by the end of the test can make all the results appear random. Our desired evaluation of a new test method might look like the following example of the test of an automotive paint.

ck, White, Gray (7)
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Number of specimens is the multiple of all the variables and in this case is 7 x 3 x 2 x 3 x 2 = 252. The total of 252 is considered to be an average test for an outdoor exposure test, but for many studies this is too big of a sample set to expose in the accelerated test. We could then consider a partial sample set which reduces the quantity of specimens. A smaller matrix extracted from the above plan might look like this. Colors 3, Substrates 2, Binder 1, Color Coat 1, Top Coat 2 for a total number of variables of 3 x 2 x 1 x 1 x 2 = 12. This set can be tested and if the results are promising, the rest of the matrix can then be evaluated.

Balanced Design

The specimen matrix must be balanced so that meaningful comparisons can be made. In order to get meaningful conclusions, individual results need to be grouped, and in order to do this properly, all specimen types must be represented in all the tests.

- Every variable must be in every test.
- Use an equal number of specimens in each test.
- Use the same number of exposure periods in each test.
- Use the same evaluation techniques throughout.

The schedule of evaluations does not have to be evenly distributed. It is acceptable to schedule more evaluations in the early periods of the test. This is likely to be the time when the greater amount of change is achieved. The use of replicates is most important. Too many tests are conducted with only a single specimen representing each variable. The end of the test, when it is time to analyze the data, is not the time to find out that not enough specimens were exposed.

- More specimens lead to better data analysis.
- At least three of each kind allows mean, median, and standard deviation calculation.
- More specimens permit a conclusion with smaller difference between results.

Reference Materials

Reference materials, which are also known as control materials, are a valuable tool in comparing accelerated and outdoor tests. A reference, or control, material is one with known performance and this can be used to verify that the accelerated test is yielding the correct failure types. Use the reference material to compare different tests or different exposures. The accelerated test is only useful if it produces the same type of degradation as the outdoor exposure. By basing the end point of any test on the amount of degradation on the control material, we can make judgments as to whether the other materials are better or worse.

The results from the reference materials can be used to "normalize" the results. If we know that we get a certain amount of degradation on the control material in a set time frame outdoors, we can use that to determine the amount of time in the accelerated device is equal to the time outdoors. When the amount of time to reach this point changes from one test to another or from one exposure to another, we can always use the response of the reference material to reset the time scale.

Another "best practice" method is to always expose one good and one bad "control" material. When only one control material is included in the test, it should have a degradation end point somewhere in the middle of the pack. That is to say, the control material should not be the best performer nor should it be the worst performer. This is sometimes very difficult to predict, so a more reliable approach is to include two control materials. If you incorporate one relatively good and one relatively bad control material, it is easier to make sure their results fall somewhere in the group, and this also has the advantage of always knowing the "difference" between the two controls.

When we talk about control or reference materials for accelerated or outdoor weathering, this material is one of your own products. This material is not one of those standard reference materials such as blue wool, or polystyrene chip. The reference or control material we mean here is a specimen of your own product. This material has been tested often, or there is a large amount of data on its known performance in the end use environment.

Repeat the Test

It is most important but often overlooked, but you must repeat the tests. This will prove the test method is correct by ensuring repeatability. We need the data from repeat tests to show us the inherent variability in the testing. The differences in the performance of two materials must differ by more than the inherent variability of the test before it is possible to say with any certainty that there is a real difference. The second step in developing a standard test is to prove it can be done again. Lastly it would be good practice to see if the test can be reproduced by another lab or by another technician.

Unknowns

Determine and measure the unknown factors which will appear when testing. It is a fact of weathering testing that we should expect the unexpected. Therefore when unexpected events happen, make notes and store the data. There may be a time when the odd data reveals a pattern. If something happens differently in one test versus another or between the outdoor and accelerated tests, this might mean there is a different failure mechanism.

Gather the Data

To correctly examine the testing, our recommendation is to ensure there are at least 5 evaluation intervals during the extent of the test. If the initial condition is one of these intervals, that means we need 4 other sets of results. In the opinion of this author, this is the minimum quantity of data sets needed to properly track the degradation. The following three rules should be followed.

- 1. You must know your degradation mode. This will ensure that when the results are forthcoming, only the correct type of degradation will be acceptable.
- 2. The degradation result must be the same in all tests. The test is invalid if you get different failure modes in the different tests. If cracking is found outdoors, and blistering occurs in the accelerated test, the wrong failure mode is being recreated.
- 3. You must evaluate for all known degradation modes. It is not good practice to only judge a test by one failure mode. In most cases there are typically multiple degradation symptoms. Testing for only one failure mode may prevent us from seeing if we have a problem per rule

2 above. Keep all your original data, especially if you are testing for instrumental color difference.

Choose a type of evaluation that most closely meets your needs. See Table 5 for a comparison of advantages of the different evaluation types. The measurement techniques described in Table 6 are a sample of the type of analyses that can be performed on weathered specimens. Non-destructive testing can be used to rate surface properties and have the advantage of requiring fewer specimens. Destructive testing will determine inherent properties of materials but as the specimens are destroyed during the evaluation process, more specimens are needed initially.

Table 5: Comparison of Evaluation Groups		
Destructive	Non-Destructive	
Increases specimen quantity	Reduces specimen quantity	
Looks at internal properties	Only gives surface data	
Objective	Subjective	
Variable	Consistent	
Table 6: Weatherin	g Evaluation Methods	
Destructive Non-Destructive		
(Inherent Properties)	(Surface Properties)	
Tensile	Gloss	
Impact	Color	
Bend	Visual Appearance	
Hardness		
Abrasion		

Acceleration Factor

Acceleration factors are the most widely requested information. We are often asked "How many hours in a weathering chamber equals 1 year in Florida?", or "How many years in Europe equals one year in Florida?" We cannot answer these but we can provide instructions on how those numbers can be determined for a particular specimen in a specific test. The following list is a guide for how to determine your own acceleration factors. An example of how to calculate acceleration factors can be found later in the section on time degradation curves.

Summary of steps to create Acceleration Factor

- 1. Use Time versus Degradation curves.
- 2. Compare the accelerated and outdoor.
- 3. Check for time to reach same amount of degradation.
- 4. Verify by ranking or compare means.
- 5. If results are same, calculate acceleration factor (AF).
- 6. AF = Time Outdoor/Time in Accelerated.

Data Analysis

In the general field of weathering, there are two good practices for examining data.

1. Time Degradation Curves

As described above, this technique requires nothing more than to track the degradation results over time. By plotting the points on a graph we can see the shape of the curve. Not all degradation is linear, in fact most degradation curves show a stable period, followed at some point by a marked increase in degradation. By comparing the degradation curves of the same group of materials in both the accelerated and outdoor test we can determine such things as:

a. Is the shape of the curve similar? *This tells us whether the failure mechanism is the same.*

- b. Does the relative performance of the specimens change during the test? *This tells us that we must be careful about when we perform the determination about the comparison of the tests.*
- c. Is the overall duration of the test correct?

The most favorable point in the test for determining differences in performance is where there is the greatest separation. The greater the difference between the specimens means we can more easily and confidently conclude relative performance rankings.



Figure 4: Time Degradation Curve Accelerated Test



Figure 5: Time Degradation Curve Outdoor Test



Figure 6: Acceleration Factor Creation

The time degradation graphs in Figures 4 and 5 can be used to generate the acceleration factor calculation in Figure 6. The example shown in Figures 4, 5, and 6 is only an example of how this type of calculation can be made. One other indication that the test comparisons are good is that all the individual specimens have approximately the same acceleration factor. If the factors are different, this will show the accelerated test is not a good simulation of the outdoor exposure.

2. Statistical Analysis

All testing results should be reviewed by some form of statistical analysis. Using the right statistics will tell you all the important things you need to know from a test.

- a. Is the test method realistic?
- b. Are the test results reliable?
- c. What is the acceleration factor?

Although there are many statistical methods available, two simple yet powerful statistics are recommended. These recommendations are based on many years of application to weathering data⁴. Comparison of Means (Student t) see Eq. 1, and Rank Correlation (Spearman's rho) see Eq. 2, are recommended by ASTM Committee G03 and can be found in ASTM G169⁵.

A. Comparison of Means

- Directly compare two sets of data Is one test different from another? Did two specimens perform differently?
- Compare experimental to production Is the new product better than the old?

Is there a significant improvement?

Student t Test

- Determine the two means X₁, X₂
- Determine the two standard deviations S₁, S₂
- Count the specimens N₁, N₂

$$t = \frac{\bar{X}_{1} - \bar{X}_{2}}{\sqrt{\left(\frac{(N_{1} - 1)S_{1}^{2} + (N_{2} - 1)S_{2}^{2}}{N_{1} + N_{2} - 2}\right)\left(\frac{1}{N_{1}} + \frac{1}{N_{2}}\right)}$$

(1)

One major advantage of this statistic is that it can be applied independent of specimen quantity, so that there does not need to be an equal number of specimens in each test⁶. This statistic is best when used to compare 1 material, or 1 color, or 1 type in each test.

B. Rank Data

- Rank the specimens in order of performance.
- Use measurable targets.

Time to 50% gloss (example) Ranking at greatest differentiation Ranking at end of test

• Can be used to determine if an accelerated test is a good predictor of real time.

Spearman Rank Correlation

- Determine the Difference
- Sum the square of differences ΣD^2
- Count the specimens N₁, N₂

$$r_{s} = 1 - \frac{6(\Sigma D^{2})}{N(N^{2} - 1)}$$
(2)

In this statistic, equal rankings are allowed but must be accounted for by using half values. This statistic may only be applied if the exact same number of specimens were exposed in both tests. This statistic is best when used to evaluate various colors, or multiple materials, in two tests.

Best Practice Testing

When it comes time to start your weathering test, you have many options to choose from. The best practice is to run several outdoor and several accelerated tests and use the statistics above to determine which test method gives you the most realistic results. Table 7 is a list of the current technical possibilities for both outdoor and accelerated testing.

Table 7: Testing Opportunities		
Outdoor Location	Accelerated Test	
Hot, Wet, UV	Xenon	
Very Hot, Dry, UV	Fluorescent UV	
Temperate	Solar Concentrator	
Pollution	Combined UV/Salt	
Seashore		
Very Cold		

The different outdoor locations are used to ensure that all potential failure modes are examined. When you have found which test method is most suitable, you can conduct future testing with that procedure.

Note: You should re-verify the test exposure conditions if there are any changes to the formulation of the material. When the material changes, so does its response to the exposure conditions. Use a control specimen and re-expose along with the new material.

Table 8 shows one possible consolidated outdoor and accelerated test program. In this case the following assumptions have been made. The material is an automotive exterior paint which exhibits gloss loss and color change. The manufacturer wishes to have a 5 year warranty on the performance of the material. Early examination of the material has shown that the failure modes can be recreated in a modified SAE J2527 test. There is a new directive to reduce the cost of producing the coating and the testing laboratory is to determine if the newer cheaper formulation has the same durability as the existing product.

Table 8: Testing Program Example		
	Outdoor	Accelerated
Exposure Location	Florida	Xenon Arc
Light Source	5 degrees South	Daylight Filters
Duration	60 months duration	3,000 hours
Intervals	Measure at 3 months	Measure at 250 hrs
Evaluations	Color, Gloss, Visual	Color, Gloss, Visual
Specimen Size	300 x 150 mm panels	75 x 50 mm panels

Putting it all Together

Outdoor testing is essential for the validation of the accelerated testing. The outdoor testing can be done simultaneously with the accelerated exposures, and with the use of the correct evaluations and statistical analysis will increase the confidence of conclusions taken from the accelerated tests. Laboratory accelerated tests are one of the most useful tools available to researchers in developing new products, but in order to minimize the risk of wrong conclusions, realistic baseline data is required. Nobody wants to be surprised when specimens don't perform as predicted by the accelerated test. Outdoor testing must verify the accelerated weathering.

References

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