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Standard Guide for Addressing Variability in Exposure Testing of Nonmetallic Materials[1]

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INTRODUCTION

No experimental procedure is exactly repeatable or reproducible. Exposure testing is susceptible to poor test reproducibility because of many contributing factors. These include the type of material and its homogeneity, the complexity and variability of the outdoor environment, difficulty in precisely controlling the laboratory testing environment, and the variability in the measurement of performance. It is extremely difficult to compare "absolute data," that is, color shift, gloss, tensile, and elongation, and so forth, from different exposure tests. This is true for natural and accelerated exposures conducted outdoors or for accelerated exposure tests conducted at different times in one laboratory or comparing results between laboratories. The purpose of this guide is to provide the user with background information on test variability and guidance to conduct an exposure test that will provide valid and useful durability information.

1. Scope*

1.1 This guide covers information on sources of variability and strategies for its reduction in exposure testing, and for taking variability into consideration in the design, execution, and data analysis of both exterior and laboratory accelerated exposure tests.

1.2 The values stated in SI units are to be regarded separately as the standard. The inch-pound values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D4853 Guide for Reducing Test Variability (Withdrawn 2008)[3]
D6631 Guide for Committee D01 for Conducting an Interlaboratory Study for the Purpose of Determining the Precision of a Test Method
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
G7 Practice for Atmospheric Environmental Exposure Testing of Nonmetallic Materials
G24 Practice for Conducting Exposures to Daylight Filtered Through Glass
G90 Practice for Performing Accelerated Outdoor Weathering of Materials Using Concentrated Natural Sunlight
G113 Terminology Relating to Natural and Artificial Weathering Tests of Nonmetallic Materials
G147 Practice for Conditioning and Handling of Nonmetallic Materials for Natural and Artificial Weathering Tests

[1] This guide is under the jurisdiction of ASTM Committee G03 on Weathering and Durability and is the direct responsibility of Subcommittee G03.93 on Statistics.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

*A Summary of Changes section appears at the end of this standard

G151 Practice for Exposing Nonmetallic Materials in Accelerated Test Devices that Use Laboratory Light Sources
G152 Practice for Operating Open Flame Carbon Arc Light Apparatus for Exposure of Nonmetallic Materials
G153 Practice for Operating Enclosed Carbon Arc Light Apparatus for Exposure of Nonmetallic Materials
G154 Practice for Operating Fluorescent Ultraviolet (UV) Lamp Apparatus for Exposure of Nonmetallic Materials
G155 Practice for Operating Xenon Arc Light Apparatus for Exposure of Non-Metallic Materials
G166 Guide for Statistical Analysis of Service Life Data
G169 Guide for Application of Basic Statistical Methods to Weathering Tests
G172 Guide for Statistical Analysis of Accelerated Service Life Data
G183 Practice for Field Use of Pyranometers, Pyrheliometers and UV Radiometers

3. Terminology

3.1 *Definitions:*

3.1.1 Terminology G113 is generally applicable to this guide.

4. Significance and Use

4.1 Many standards and specifications reference exposure tests performed according to standards that are the responsibility of Committee G03 on Durability of Nonmetallic Materials. In many cases, use of the data generated in these tests fails to consider the ramifications of variability in the exposure test practices. This variability can have a profound effect on the interpretation of results from the exposure tests, and if not taken into consideration in test design and data analysis, can lead to erroneous or misleading conclusions. This guide lists some of the sources for test variability and recommends strategies for executing successful weathering studies. Not all sources of variability in weathering testing are addressed in this guide. Specific materials, sampling procedures, specimen preparation, specimen conditioning, and material property measurements can contribute significantly to variability in weathering test results. Many of these concerns are addressed in Guide G147. To reduce the contribution of an instrumental method to test variability, it is essential to follow appropriate calibration procedures and ASTM standards associated with the particular property measurement. Additional sources of variability in test results are listed in Guide D4853, along with methods for identifying probable causes.

5. Variability in Outdoor Exposure Tests

5.1 *Variability Due to Climate*—Climate at the test site location can significantly affect the material failure rates and modes. Typical climatological categories are; arctic, temperate, subtropical, and tropical (that are primarily functions of latitude). Subcategories may be of more importance as being dictated by geographic, meteorological, terrain, ecological, and land-use factors, and include such categories as desert, forested, (numerous classifications), open, marine, industrial, and so forth. Because different climates, or even different locations or orientation in the same climate, produce different rates of degradation or different degradation mechanisms, it is extremely important to know the characteristics of the exposure sites used and to evaluate materials at sites that produce intensification of important climate stresses. Typically, exposures are conducted in "hot/wet" and "hot/dry" climates to provide intensification of important factors such as solar radiation and temperature, and to determine possible effects of moisture. Different exposure sites in one climate (even those in close proximity) can cause significantly different results, depending on material.

NOTE 1—Exposures in a tropical summer rain climate (for example, Miami, Florida) and in a hot desert climate (for example, Phoenix, AZ) are recognized as benchmarks for evaluating the durability of many different materials.

5.2 *Variability Due to Time of Year*—Solar-ultraviolet radiation, temperature, and time of wetness vary considerably with time of year. This can cause significant differences in the rate of degradation in many materials. Therefore, comparison of results between short-term exposure studies (less than one full year) will be subject to greater variability. If exposures of less than a full year are required, consider using times when climatological stress is maximized so a worst case test result is obtained. It may also be valuable to make several exposure tests with varying start dates in order to provide more representative data. This is especially true when the material's response to the environment cannot be predetermined, or when materials with different environmental responses are to be compared. Often exposure periods are timed by total solar or solar-ultraviolet dose, or both. This approach may reduce variability in certain instances. However, an inherent limitation in solar-radiation measurements is that they do not reflect the effects of variation in temperature and moisture, which are often as important as solar radiation. Temperature and time of wetness are highly dependent on time of year, especially in temperate climates. With materials that are sensitive to heat or moisture, or both, the same solar-ultraviolet radiation dose may not give the same degree of change unless the heat and moisture levels are also identical.

5.2.1 Another problem related to timing exposures by broad-band radiation measurements is that solar radiation in the 290 to 310-nm band pass exhibits the most seasonal variability. Some polymer systems are extremely sensitive to radiation in this band pass. Variations in irradiance in this critical region (because of their relatively small magnitude) are not adequately reflected in total solar radiation or broad-band solar ultraviolet (UV) measurements.

5.2.2 The time of year (season) that an exposure test is initiated has, in certain instances, led to different failure rates for identical materials (**1**).[4]

5.3 *Variability Due to Year-to-Year Climatological Variations*—Even the comparison of test results of full-year exposure increments may show variability. Average temperature, hours of sunshine, and precipitation can vary considerably from year to year at any given location. The microclimate for the test specimens can be affected by yearly

[4] The boldface numbers in parentheses refer to the list of references at the end of this standard.

differences in pollution levels, airborne particulates, mold, and mildew. These differences can impact material failure rates. Results from a single-exposure test cannot be used to predict the absolute rate at which a material degrades. Several years of repeat exposures are needed to get an "average" test result for any given test site.

5.4 *Variability Due to Test Design*—Every exposure test has some variability inherent in its structure and design. Specimen placement on an exposure rack (**2**), and type or color of adjacent specimens can also affect specimen temperature and time of wetness. Sample backing or insulation as well as rack location in an exposure site field can affect specimen temperature and time of wetness.

5.5 *Variability in Glass-filtered Daylight Exposures*—Glass-filtered daylight exposures as described by Practice G24 are subject to many of the test variables previously described. Recent studies conducted by ASTM Subcommittee G03.02 on Natural Environmental Testing has demonstrated that the glass used in these exposures can be highly variable in its light transmission characteristics between 300 and 320 nm that can significantly impact exposure results (**3**). In addition, solarization processes can alter these transmission characteristics during the first few months of exposure. Specimen temperature can also vary depending on location within an under glass test rack (**4**).

6. Variability in Accelerated Outdoor Exposures Using Concentrated Sunlight

6.1 Accelerated outdoor exposures using Fresnel concentrators are described in Practice G90. Test results are subject to normal climatological and seasonal variations. Exposure periods are described by a radiant energy dose, most often in the UV region of sunlight. The UV content of the concentrated sunlight is reduced during winter exposures and is also subject to normal year-to-year variations. As mentioned in 5.2, current radiant energy band passes, both total solar and broad-band UV, used in reporting solar dose do not adequately reflect variations in the critical 290 to 310-nm range. Because of the time of year differences in the amount of available ultraviolet, timing exposures based on accumulated ultraviolet dose can improve test-to-test variability, but may not account for the substantial specimen temperature differences that exist between summer and winter.

6.2 When test conditions specify water spray, water quality is extremely critical. Water contaminants or impurities can cause specimen spotting that will give misleading durability results.

7. Variability in Laboratory Exposure Tests

7.1 Practices G151, G152, G153, G154, and G155 describe laboratory accelerated weathering tests and are referenced in many ASTM standards describing tests for particular products. A round-robin evaluation of filtered open flame carbon-arc, fluorescent UV, and xenon-arc exposures was performed between 1985 and 1992 comparing the gloss retention of various vinyl tapes (**5**). Although the variability reported is specific to the materials tested and the participating laboratories, these referenced round-robin studies serve as a warning to users of durability test standards that high levels of variability may be possible with any test or material.

7.1.1 *Repeatability*—In general, test precision within laboratories (a single test period in a test device) will always be better than precision between laboratories. By testing replicate specimens, statistically significant performance differences among materials can be readily established.

7.1.2 *Reproducibility*—The G03.03 round-robin studies found that between laboratory comparisons of absolute gloss values after a fixed exposure time is, in a practical sense, impossible. Replicates specimens exposed to seemingly identical test conditions gave highly variable results from laboratory to laboratory. Other round-robin weathering studies have demonstrated varying degrees of variability with different materials and property measurements (**6-8**) Precise control of critical exposure parameters may not be feasible when devices are located in differing ambient laboratory conditions and operated by a diverse user group.

NOTE 2—Indices of precision and related statistical terms are defined in Practice E177.

7.2 *Specific Factors Responsible for Variability in Accelerated Laboratory Exposure Tests:*

7.2.1 Light sources for all test devices are subject to normal manufacturing variation in peak irradiance and spectral power distribution (SPD). In many instances, the filter glasses associated with certain devices and light sources also demonstrate significant variation in their initial UV transmission characteristics. As the light source and filter glasses age during normal use, the irradiance and SPD can also change significantly. Instruments that monitor irradiance at 340 nm or broad-band radiometers (300 to 400 nm) may not detect or compensate for these changes.

7.2.2 Irradiance and specimen temperatures can vary significantly throughout the allowed specimen exposure area, especially in older test equipment.

7.2.3 Water contaminants or impurities and poor spray quality, that is, clogged spray nozzles, can cause specimen spotting that will give misleading durability results by impacting visual observations, reducing specular gloss values, causing unnatural color shifts, or by impacting other optical properties.

7.2.4 Ambient temperature and humidity conditions in the testing laboratory can affect test chamber conditions and device operation. In fluorescent UV condensation devices, high ambient temperatures can reduce the amount of condensate that forms on the test specimens. If the device does not have an irradiance control system, ambient temperature can also affect irradiance at the specimen plane.

8. Addressing Variability in All Exposure Tests

8.1 Extreme caution must be used when comparing test results between different laboratories or from different time periods. This applies equally to laboratory accelerated tests, outdoor exposure tests, and outdoor accelerated tests. The safest approach is to treat each exposure test as a separate entity and make durability comparisons for materials exposed at the same time in the same device or at the same outdoor exposure site.

8.2 The proper use of experimental design and data analysis techniques can cope with the variability inherent to weathering testing. Guide G169 describes how basic statistical methods can be applied to weathering tests.

8.3 *General Considerations:*

8.3.1 Round-robin studies (**5**) conducted by Committee G03 and others (**9**) indicate that nominally similar tests can cause significantly differing failure rates, but rank performance for a series of materials is quite reproducible between devices running the same test cycle in different laboratories. In these cases, differing stress levels do not affect the ranking of materials, just the time required to achieve the same level of degradation. This same response is often true for outdoor exposures as well. Year-to-year meteorological variations can significantly impact the failure rate of materials, but the weathering performance ranking of a series of materials is quite reproducible.

8.3.2 The use of replicate specimens of each material *for all exposure studies* is essential. This allows the use of statistical data treatments, such as analysis of variance, in order to meaningfully assess performance differences between materials. If only one specimen from each material is exposed, performance of differences among materials can never be determined to be statistically significant.

8.3.2.1 Use of two replicate specimens per material is acceptable, however using more replicates provides for better statistical analysis and may help to identify possible outliers. When destructive tests are used to characterize material properties, the number of replicates is higher and is often dictated by the standard describing the property measurement procedure.

8.3.3 Weathering reference materials or standard weathering reference materials are sometimes used to monitor exposure conditions in exposure devices used in different laboratories or in the same laboratory. The use of absolute property levels after specific exposure periods for these materials is acceptable only if the variability has been statistically determined through appropriate round-robin evaluations.

8.3.4 Measurements or observations should be repeated throughout the exposure test duration to determine optimum times for comparison to control specimens or for ranking the performance of a series of specimens. Optimum times are when performance differences between test specimens or between test specimens and the control specimen are the greatest and are statistically significant.

8.3.5 The equipment used to measure material properties during exposure testing should be maintained in proper calibration and operating condition.

8.3.6 Follow the procedures described in Practice G147 for selection, handling, and conditioning of test specimens to reduce their contribution to variability in test results.

8.4 *Material Specification Testing:*

8.4.1 Test specifications that list an absolute property level after a specific exposure period without setting appropriate statistical confidence intervals are not technically valid. A material specification that requires an absolute property level after a specific exposure period may be acceptable if test variability (reproducibility) has been quantified in statistical terms for the specific material type. This requires that appropriate round-robin experiments be conducted with a representative selection of exposure laboratories (follow procedures outlined in Practice E691). Once generated, this data cannot be extrapolated to other material types or exposure test conditions.

8.4.2 The proper use of control materials permits valid test information even with the highly variable nature of weathering testing. Comparisons are made relative to control specimens. The absolute amount of change in a performance property is not necessarily important. Only a statistically significant difference in performance between the control and test specimens is required. When this is achieved, the test specimen can be judged "better" or "worse" than the control. Validity can be added to these comparisons by choosing a control material that is similar in composition to the test specimens, that is, polymer type, color, or construction.

8.4.3 Material specifications requiring a specific number of exposure hours or radiant dose without any failure occurring provide very limited durability information. Two specimens with highly differing durability levels could pass this type of specification. Test to failure or until significant differences in performance are established.

8.5 *Service Life Prediction And Relating Laboratory Exposure Test Results To Outdoor Exposure Results:*

8.5.1 Because of variability inherent in exposure tests, results from a single exposure test cannot be used to determine the absolute rate at which a nonmetallic material degrades. Several replicate tests are required to estimate the mean failure rate of a material.

8.5.2 Because of the variability involved in most aspects of durability testing, direct comparisons of property retention versus time plots to obtain an acceleration factor, that is, *X* hours in the accelerated test equals *Y* year(s) outdoors, is highly questionable unless many replicate tests are run. In addition, acceleration factors are highly variable, among different material types and formulations, that limits their general applicability and usefulness. Practice G151 gives more information on problems with use of acceleration factors.

8.5.3 Nonparametric statistics, specifically Spearman rank correlation, have proven useful in quantifying how well an accelerated test relates to a long-term natural exposure test. The nonparametric approach does not assign an absolute level of performance (or failure) to a single material, but ranks the performance of a series of materials. In correlating accelerated and real-time exposure tests, the rank performance of a series of materials exposed to both environments is compared, and the strength of the association between the tests is established. Examples of nonparametric methods for analyzing weathering results are described in Guide G169. A method to evaluate correlation results to determine whether a specific rank correlation coefficient is adequate is now available (**10**).

8.5.4 Valid service-life predictions for a material may be achieved even with highly variable test results by using reliability analysis where variability is treated as a distribution of time to failure (**11**). This approach has been used successfully in the electronics and aerospace industries for several years. Work is currently underway to adapt reliability methods