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Standard Guide for Addressing Variability in Exposure Testing on 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 either inch-pound or SI units are to be regarded separately as the standard. The 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 4853 Guide for Reducing Test Variability[2]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[3]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

G 7 Practice for Atmospheric Environmental Exposure Testing of Nonmetallic Materials[3]

G 23 Practice for Operating Light-Exposure Apparatus (Carbon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials[3]

G 24 Practice for Conducting Exposures to Daylight Filtered Through Glass[3]

G 26 Practice for Operating Light-Exposure Apparatus (Xenon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials[3]

G 53 Practice for Operating Light- and Water-Exposure Apparatus (Fluorescent UV-Condensation Type) for Exposure of Nonmetallic Materials[3]

G 90 Practice for Performing Accelerated Outdoor Weathering of Nonmetallic Materials Using Concentrated Natural Sunlight[3]

G 147 Practice for Conditioning and Handling of Nonmetallic Materials for Natural and Artificial Weathering Tests[3]

3. Terminology

3.1 *Definitions:*

3.1.1 Terminology G 113 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 G-3 on Durability of Nonmetallic Materials. In many cases, use of the data generated in these tests

[1] This guide is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.93 on Statistics.

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[2] *Annual Book of ASTM Standards*, Vol 07.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

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 G 147. 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 D 4853, 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 a range of sites that represent the full spectrum of anticipated service conditions. Exposure sites in one climate (even those in close proximity) can cause significantly different results, depending on material.

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 polymers. Therefore, comparison of results between short-term exposure studies (less than one or more full years) is extremely risky. 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. Many polymer systems are extremely sensitive to radiation in this band pass. Variations in radiance 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 can be difficult. 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 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 polymer 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 G 24 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 400 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 G 90. 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 significantly 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

[4] *Annual Book of ASTM Standards*, Vol 06.01.

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 Significant variability in test results can occur between laboratories conducting accelerated outdoor exposures using concentrated sunlight **(4)**. Identical materials exposed for the same time period, but at different sites within close proximity to each other, had significantly different failure rates.

6.3 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 A round-robin evaluation of Practice G 23, G 26, and G 53 exposure tests was performed between 1985 and 1992 comparing the gloss retention of various vinyl tapes **(6)**. 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 during a specific exposure period in an individual test device.

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 **(7-9)** 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 1—Indices of precision and related statistical terms are defined in Practice E 177.

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. Examples of suitable statistical methods for analyzing weathering results are described in a guide currently under development in Subcommittee G03.93.

8.3 *General Considerations*:

8.3.1 Round-robin studies **(6)** conducted by Committee G-3 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 recommended. 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.

8.3.3 Weathering reference materials or standard weathering reference materials are often used to monitor or control exposure conditions between laboratories or exposure devices. 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