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Designation: D 4141 – 9501

Standard Practice for Conducting Accelerated Outdoor Exposure Tests Black Box and Solar Concentrating Exposures of Coatings¹

This standard is issued under the fixed designation D 4141; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers three two accelerated outdoor exposure procedures for evaluating the exterior durability of coatings applied to metal substrates.

1.2 The-three_two procedures are as follows:

1.2.1 Procedure A—Black Box Exposure,

1.2.2 Procedure B-Heated Black Box Exposure, and has been deleted from this practice.

1.2.3 Procedure C—Fresnel Reflector Rack Exposure.

1.3 The durability rankings of coatings provided by these three procedures may not agree when coatings differing widely in composition are compared.

1.4 The acceleration of degradation rates of coatings produced by the three procedures of exposure are discussed.

NOTE 1-OthProcedure B described a Heated Black Box procedure that is no longer in common use.

<u>1.3 This standard does not cover all the procedures may provide accelerated results that are available to the user for some types accelerating the outdoor exposure of products.</u>

1.5 The values stated <u>coatings</u>. Other procedures have been used in <u>either inch-pound or SI units are</u> <u>order</u> to <u>be regarded</u> separately as provide a particular effect; however, the standard. The values given in parentheses two procedures described here are for information only.

1.6 widely used.

<u>1.4</u> 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. A specific precautionary statement is given in Note 5.

2. Referenced Documents

2.1 ASTM Standards:

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¹ This practice is under the jurisdiction of ASTM Committee <u>D-1</u> <u>D01</u> on Paint and Related Coatings, Materials, and Applications, and is the direct responsibility of Subcommittee D01.27 on Accelerated Tests for Protective Coatings.

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D 523 Test Method for Specular Gloss²

D 660 Test Method for Evaluating Degree of Checking of Exterior Paints²

D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints²

D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints²

D714 Test Method for Evaluating Degree of Blistering of Paints²

D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints²

D 823 Practices for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²

D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²

D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base²

D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates²

D 4214 Test Methods for Evaluating Degree of Chalking of Exterior Paint Films²

G 7 Practice for Atmospheric Environmental Exposure Testing of Nonmetallic Materials³

G 90 Practice for Performing Accelerated Outdoor Weathering of Nonmetallic Materials Using Concentrated Natural Sunlight³ G 113 Terminology Relating to Natural and Artificial Weathering Tests of Nonmetallic Materials³

G 141 Guide for Addressing Variability in Exposure Testing of Nonmetallic Materials³

G 147 Practice for Conditioning and Handling of Nonmetallic Materials for Natural and Artificial Weathering Tests³

3. Terminology

3.1 The definitions given in Terminology G 113 are applicable to this practice.

4. Summary of Practice

<u>34.1</u> Several procedures are described that provide acceleration of the degradation that coatings <u>experience evidence</u> during natural weathering <u>when exposed on an open rack at a fixed angle</u>. The procedures appear in the following order:

34.1.1 Procedure A—Exposure on a black box-panel rack facing the equator at 5° from-horizontal.

3.1.2 Procedure B-Exposure on a heated black box panel rack facing the equator at 5° from horizontal.

3.1.3

<u>4.1.2</u> *Procedure C*—Exposure on a Fresnel reflector-panel rack that provides a high intensity of sunlight irradiation irradiance by following the sun and focusing the reflecting sunlight on the test-panels specimens by means of mirrors. The panels specimens are wet periodically by deionized high purity water spray.

4.2 Each of these procedures requires that coated test panels be placed on devices of specified design and be exposed under specified conditions of weathering.

4.3 The selection of Procedure A or C is dependent on several factors.

4.3.1 Procedure A is designed to simulate the weathering that occurs on horizontal automotive surfaces, and is specified in standards used by the automotive industry. Specimens are typically flat-coated metal panels measuring 10 by 30 cm (4 by 12 in.) or 15 by 30 cm (6 by 12 in.).

4.3.2 Procedure C is designed to simulate weathering on both automotive and nonautomotive products. Procedure C typically provides faster results than Procedure A on a calendar basis.⁴

3.2 Each of these procedures requires that coated test panels be placed on racks of specified design and be exposed under specified conditions of weathering.

3.3 The selection of a procedure for producing accelerated degradation is dependent on the intended end use of the coatings, limitations on the time available for the exposure tests, and the degree of reliability required for predictions of durability performance.

4.

5. Significance and Use

45.1 As with any accelerated test, the difference in rate of weathering is material dependent and no single exposure factor can be used to compare two different weathering exposures. The durability rankings of coatings provided by these two procedures may not agree when coatings differing widely in composition are compared. These two procedures should not be used interchangeably or used for absolute comparison to each other.

<u>5.2 The procedures</u> described in this practice are designed to provide greater degradation rates of coatings than those provided by fixed angle open-rack outdoor exposure racks. For many products, fixed angle exposures will produce higher degradation rates than the normal end use of the material.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vol 14.024.

⁴ Fresnel reflector panel racks are located at ITeraeus DSET Laboratories, Inc., 45601 N. 47th Avenue, Phoenix, AZ 85027-7042

⁴ Zerlaut, G.A., Rupp, M.W., and South Florida Test Service, Desert Site, 31818 N. 203rd Ave., Wittmann, AZ 85361. Anderson, T.E., "Ultraviolet Radiation as a Timing Technique for Outdoor Weathering of Materials," Paper 850378, *Proceedings*, SAE International Congress, Detroit, February 25, 1985.

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4.2 Following are comments regarding the relative rates

5.2.1 The use of degradation that can be expected for coatings exposed by the procedures described in this practice.

4.2.1 Procedure <u>Procedure</u> A (Black Box)—For many coatings, this procedure provides greater rates <u>instead</u> of degradation than those provided by 5°, equator-facing, an open-rack exposures because the black box produces higher panel temperatures during irradiation by the sun and longer time of wetness. The black box panel temperatures are comparable to those encountered on the hoods, roofs, and deck lids of automobiles parked in direct-sunlight.

4.2.2 Procedure B (Heated Black Box)— This procedure exposure is most useful a more realistic test for exposures conducted in the late fall, winter, and early spring when it produces significantly higher panel temperatures than those produced by the 5°, equator-facing, black box. Therefore, this procedure produces greater rates of degradation than those produced by Procedure A, particularly in the case of coatings materials with rates of degradation that are very higher temperature dependent. end use service conditions.

NOTE 2—<u>Procedure A (Black Box)</u>—For many coatings, this procedure provides greater rates of degradation than those provided by 5° , equator-facing, open-rack exposures because the black box produces higher specimen temperatures during irradiation by daylight and longer time of wetness. The black box specimen temperatures are comparable to those encountered on the hoods, roofs, and deck lids of automobiles parked in unfiltered daylight. The relative rates of gloss loss and color change produced in some automotive and coil coatings by exposures in accordance with Procedures A-and B are given in ASTM STP 781.⁵

4.2.3 *Procedure C (Fresnel Reflector Rack)* This method provides greater rates of degradation of coatings than those provided by Procedure A or Procedure B. This high acceleration is produced by very high sunlight intensity and high panel temperature.

4.2.4 The degradation rates produced by any of the three procedures depend on the seasons of exposure, geographical location, and type of coating. The rates are higher in late spring, summer, and early fall when the intensity of sunlight irradiation is the greatest and when panel temperatures are higher.

4.2.5 Because outdoor weather conditions vary from season to season and year to year, these procedures are not reliable for establishing absolute performance ratings for coatings. The procedures should be used only for comparing the relative performance of coatings exposed at the same time at the same location.

PROCEDURE A—BLACK BOX EXPOSURE

5. Apparatus

5.1 *Black Box*, constructed of materials in accordance with Practice G 7, or its equivalent, and positioned so that the surfaces of the test specimens are 5° from the horizontal, facing the equator.

6. Test Specimens

6.1 Each test specimen and control specimen shall consist of a uniform coating applied to the surface of a rigid panel. Suitable application procedures are given in Practices D 823.

6.2 Use surfaces of good planarity because warpage, waviness, or curvature may seriously affect the measurements of gloss and eolor and may produce a poor air seal on the black box rack.

6.3 Prepare reference specimens for inclusion in each exposure series to act as comparison standards and to provide a means for determining the severity of the exposure conditions encountered by the series. For best results, there should be at least two reference materials differing significantly in their durability performance.

6.4 Optionally, using Test Methods D 1186, measure the dry film thickness of the coatings at several different positions on the test specimens.

7. Procedure

7.1 If change in gloss is to be measured, determine the specular gloss value for each unexposed specimen using a properly calibrated glossmeter in accordance with Test Method D 523.

7.2 If change in color is to be measured, determine the color coordinates for each unexposed specimen using Test Method D 2244. Unless otherwise agreed upon, use the CIE Lab Color Scale. The color-measuring instrument shall be stable and properly calibrated.

Note 3—AProcedure C (Fresnel Reflector Rack)—The acceleration of Procedure C is produced by reflecting sunlight from ten mirrors onto an alternative procedure, reserve unexposed duplicate air-cooled specimen-panels of each coating as file specimens to determine area. In the color change ultraviolet portion of the exposed specimens. To minimize color drift, store solar spectrum, approximately 1400 MJ/m² of ultraviolet radiant exposure (295 to 385 nm) is received over a typical one-year period when these-panels devices are operated in a dark, room temperature environment.

7.3 Mount central Arizona climate. This compares with approximately 333 MJ/m² of ultraviolet radiant exposure from a central Arizona at-latitude exposure and 280 MJ/m² of ultraviolet radiant exposure from a southern Florida at-latitude exposure over the specimens on same time period. However, the test described by Procedure C reflects only direct beam radiation onto test specimens. The reflected direct beam sunlight contains a lower percentage of short wavelength ultraviolet radiation than global

⁵ Symposium on Permanence of Organic Coatings, ASTM STP 781, ASTM, 1982.

<u>daylight because short wavelength ultraviolet is more easily scattered by the atmosphere, and because mirrors are typically less</u> <u>efficient at shorter ultraviolet wavelengths. Ultraviolet radiant</u> exposure box. Cover all empty spaces levels should not be used to <u>compute acceleration factors since acceleration is material dependent.</u>

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5.3 The relative durability of coatings in outdoor use can be very different depending on the black box using black panels so location of the exposure because of differences in ultraviolet (UV) radiation, time of wetness, temperature, pollutants, and other factors. Therefore, it cannot be assumed that results from one exposure in a single location will be useful for determining relative durability in a different location. Exposures in several locations with different climates that represent a broad range of anticipated service conditions are recommended.

5.4 Because of year-to-year climatological variations, results from a single exposure test cannot be used to predict the absoluten rate at which a material degrades. Several years of repeat exposures are needed to get an "average" test result for a given location.

5.4.1 The degradation profile for many polymers is not a linear function of exposure time or radiant exposure. When short exposures are used to predict the service life or as indications of relative durability, the results obtained may not be representative of those from longer exposures.

Note 4—The predominant color 4—Guide G 141 provides information for addressing variability in exposure testing of the specimen panels on the black box as well as the temperature of nonmetallic materials. ASTM G03 Committee is developing a standard guide for application of statistics to exposure test results.

5.5 It is recommended that at least oune controld material be noted. A black panel on the box will attain a lower temperature if all the other panels part of any exposure evaluation. Control materials are white than if all used for comparing the other panels are black.

7.4 Expose performance of the test and reference specimens for a specified period of time on materials relative to the basis of controls when materials are not being ranked against one another. The control material used should be of the following:

7.4.1 Expose for a specified number of days, months, or years with respect similar composition and construction to an agreed upon starting date.

7.4.2 Expose for a specified quantity of radiant exposure either total, typically measured from 300 to 3000 nm, or ultraviolet, typically measured from 300 to 385 nm.

7.4.2.1 For Procedures A the test materials and B, when solar ultraviolet radiation be of known durability. It is measured, use a total ultraviolet radiometer that measures ultraviolet radiation in the wavelength region from 295 preferable to 385 nm.⁶ Calibrate the radiometer use two control materials, one with relatively good durability and readout system in suitable radiometric units, one with poor durability.

6. Test Specimens

<u>6.1 Each test specimen</u> and maintain in at least annual calibration against a standard source control specimen shall consist of spectral irradiance.⁶

7.4.2.2 For Procedure C, use a uniform coating applied to the measurement technique specified in Practice G 90.

7.4.3 Exposure until surface of a specified physical change has occurred rigid panel. Suitable application procedures are given in the test Practice D 823.

6.2 Use flat specimens, or

7.4.4 Exposure until a specified change has occurred in a reference specimen exposed with because warpage, waviness, or curvature may seriously affect the test specimens.

7.5 Unless otherwise agreed upon, remove the test specimens from the black box measurements of gloss and gently wash color and may produce a portion of poor air seal on the black box rack.

<u>6.3 For Procedure C, specimen-surfaces sizes are typically limited</u> to remove loose dirt. A suitable procedure consists of gentle rubbing with a sponge wet with water or a 0.05 % solution maximum of 13 cm (5 in.) in one dimension, and a nonionic detergent.⁶ Gently remove water droplets from maximum of 140 cm (55 in.) in the other dimension. However, specimensu arfe typically 7.5 by 13 cm (3 by 5 in.) or 5 by 13 cm (2 by 5 in.). Because air cooling is used to prevent-water spotting.

7.6 Unless otherwise agreed upon, perform the following on the washed portion high specimen temperatures, specimens must be flat. A thickness of less than 0.6 cm (0.25 in.) is preferred. This practice may not apply to specimens thicker than 1.3 cm (0.5 in.) because cooling may be questionable.

6.4 Prepare controls for inclusion in each-washed specimen:

7.6.1 Measure exposure series to act as comparison standards and to provide a means for determining the specular gloss severity of the exposure conditions encountered by Test Method D 523,

7.6.2 Calculate the color difference series. For best results, there should be at least two controls differing in-accordance with their durability performance.

6.5 Optionally, using Test-Method D 2244 based on instrumental measurements of color before and after exposure.

7.6.3 Checking and cracking rating by Methods D 1186 or Test Method D 660, and

7.6.4 Blistering rating by Test Method D 714.

7.7 Unless otherwise agreed upon, perform a chalk rating by Test Method D 4214 on an unwashed area D 1400, measure the dry film thickness of the coatings at several different positions on the test specimens.

6.6 Unless otherwise specified, expose at least two replicates. Larger numbers of replicates are recommended.

PROCEDURE-B-HEATED BLACK A-BLACK BOX EXPOSURE

87. Apparatus

<u>87</u>.1 *Black Box*, constructed of materials in accordance with Practice G 7, or its equivalent, equipped with a device to heat the air in the interior of the box to the specified temperature and positioned so that the surfaces of the test specimens are 5° from the horizontal, facing the equator.

8. Procedure

8.1 Use Practice G 147 for specimen handling and conditioning procedures for test specimens.

8.2 If a change in gloss is to be measured, determine the specular gloss value for each unexposed specimen using a properly calibrated glossmeter in accordance with Test Method D 523.

8.3 If a change in color is to be measured, determine the color coordinates for each unexposed specimen using Test Method D 2244. Unless otherwise agreed upon, use the CIE Lab Color Scale. The color measuring instrument shall be stable and properly calibrated.

NOTE 5—Precaution: The heated black box must be properly grounded 5—As an alternative procedure, reserve unexposed duplicate specimen panels of each coating as file specimens to prevent possible electrical shock. The heating elements in determine the heated black box must be installed so that, in normal use, a person cannot come in contact with a hot surface.

9. Procedure

9.1 If color change of the exposed specimens. To minimize color drift, store the panels in gloss a dark, room-temperature environment.

8.4 Mount and color are to be measured, follow 7.1 and 7.2, respectively.

9.2 Mount and fasten the specimens on the exposure box. Cover all empty spaces on the black box-as described in 7.3.

9.3 Set using black panels so that the temperature controller to maintain entire surface is covered.

<u>NOTE</u> 6—The predominant color of the <u>air temperature inside</u> <u>specimens on</u> the <u>heated</u> black box <u>at approximately 140°F (60°C)</u>, <u>or</u> <u>should be noted</u>. <u>A black box will attain a lower temperature if all the</u> other <u>agreed upon temperature</u>, and to turn <u>specimens are white than if</u> the <u>heater on at approximately</u> 9 a.m. and turn off at approximately 3 p.m. each day.

9.4 Expose other specimens are black.

8.5 Expose the test and control specimens for a specified period of time on the basis of one of the following:

8.5.1 Expose for a specified number of days, months, or years with respect to an agreed upon starting date.

<u>8.5.2 Expose for a specified quantity of radiant exposure either total, typically measured from 300 to 3000 nm, or ultraviolet, typically measured from 295 to 385 nm. When solar ultraviolet radiation is measured, use a total ultraviolet radiometer that measures ultraviolet in-7 the wavelength region from 295 to 385 nm. Optionally, ultraviolet can be measured in the wavelength region from 300 to 400 nm. Calibrate the radiometer and readout system in suitable radiometric units, and maintain in at least annual calibration against a standard source of spectral irradiance.</u>

98.5.3 Expose until a specified change has occurred in the test specimens.

8.5.4 Expose until a specified change has occurred in a control exposed with the test specimens.

8.5.5 In most cases, periodic evaluation of test and control materials is necessary to determine the variation in magnitude and direction of property change as a function of time or radiant exposure.

<u>8.6</u> Unless otherwise agreed upon, remove the test specimens from the black box and gently wash a portion of the specimen surfaces to remove loose dirt. The same portion of the specimen should be washed at each interval of exposure. A suitable procedure consists of gentle rubbing with a sponge wet with high purity water or a dilute solution of a nonionic detergent, followed by a high purity water rinse. The high-purity water shall meet as recommended a minimum the requirements for water purity contained in 7.5.

9.6 Unless otherwise agreed upon, Practice G 90.

8.7 If required, perform one or more of the following tests on the washed portion of each washed specimen:

8.7.1 Measure the tests outlined specular gloss in 7 accordance with D 523.6.

98.7.2 UCalculate the color difference in accordance with Test Method D 2244 based on instrumental measurements of color before and after exposure.

8.7.3 Evaluate checking and cracking rating in accordance with Test Methods D 660 and D 661.

8.7.4 Evaluate blistering rating in accordance with Test Method D 714.

8.7.5 Evalupate erosion rating in accordance with Test Method D 662.

8.7.6 Evaluate flaking rating in accordance with Test Method D 772.

<u>8.8 If required</u>, perform a chalk rating by in accordance with Test Methods D 4214 on an unwashed portion area of the each specimen.

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PROCEDURE C-FRESNEL REFLECTOR RACK EXPOSURE

10.

<u>9.</u> Apparatus

10.1

<u>9.1</u> Fresnel Reflector Exposure Rack, that follows the sun, concentrates sunlight irradiation on the test specimens by means of mirrors, and sprays-deionized high purity water on the surfaces of the test specimens at specified intervals. The rack shall have provisions for cooling the test specimens while they are irradiated. Refer to Practice G 90 for detailed descriptions of the apparatus.

110. Procedure

10.1 Use Practice G 147 for specimen handling and conditioning procedures for test specimens.

10.2 If a change in gloss and color are to be measured, follow-7.1 8.2 and 7.2, 8.3, respectively.

110.23 Mount and fasten the specimens on the exposure rack device in accordance with Practice G 90.

140.34 Set the water spray control to provide a <u>deionized high purity</u> water spray on the specimens during the night at a frequency of 4 per hour, each spray-<u>period</u> to be of 3-min duration. Refer to Cycle-C_3 of Practice G 90 for detailed operating procedures.

110.45 Expose the specimens for a specified period on the basis of either ultraviolet or total radiant exposure. Both ultraviolet and total radiant exposure must be measured and reported using the procedures described in Practice G 90.

 1 ± 0.56 Unless otherwise agreed upon, remove the specimens from the exposure <u>rack_device</u> and gently wash a portion of the specimen surfaces as recommended in 7.5.

11.6 Unless otherwise agreed upon, 8.6.

10.7 If required, perform on the washed portion of each specimen, the tests outlined in 7.6.

11.7 Unless otherwise agreed upon, 8.7.

10.8 If required, perform a chalk rating by Test Methods D 4214 on an unwashed portion of the each specimen.

121. Interpretation of Results

121.1 Express the change in gloss of each specimen either in terms of units of gloss loss or in percent gloss loss relative to the initial gloss value.

121.2 Express the change in color of each specimen in terms of total color difference, ΔE , Delta E, using one of the calculations given in Test Method D 2244.

121.3 Express the amounts of chalking, checking, and cracking cracking, or other visual appearance property as outlined in Test Methods D 4214 and D 660 (or on the 0 to 10 scale described in Section 7 of the D-1 Handbook).⁶

13. <u>D 4214, D 660, D 661, D 662, D 714, or D 772.</u>

12. Report

132.1 Report the following information:

132.1.1 The method of exposure used and its geographical location;

132.1.2 The duration of the exposure and the starting and ending dates of the beginning of the test,

13.1.3 The test.

<u>12.1.3 The</u> measured amount of ultraviolet and total radiant exposure, expressed in MJ/m^2 -(Rrequired for Procedure C, optional for Procedures A and B.),

13.1.4 The heated black box air temperature used (for Procedure-B),

13.1.5 The A). If measured, report the bandpass used for ultraviolet radiant exposure (that is, 295 to 385 nm or 300 to 400 nm).

12.1.4 If available, the type of reference specimens controls used and the severity of their degradation, and.

132.1.65 The evaluation measurements performed on each of the exposed specimens:

132.1.65.1 Units of gloss-loss or percent gloss loss,

132.1.65.2 Units of ΔE Delta E color change, noting the color space and color scales used, and

132.1.65.3 Ratings for chalking, checking, cracking, erosion, flaking, and blistering.

132.1.76 If requested, report the traceability of calibrations for all environmental measurements reported.

143. Keywords

143.1 durability; exterior exposure tests; outdoor exposure; ultraviolet/light/radiation; weathering

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