

Designation: D4303 - 06

# Standard Test Methods for Lightfastness of Colorants Used in Artists' Materials<sup>1</sup>

This standard is issued under the fixed designation D4303; 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  $(\varepsilon)$  indicates an editorial change since the last revision or reapproval.

## 1. Scope

- 1.1 Four test methods to accelerate the effects of long term indoor illumination on artists' materials are described below. One of the natural daylight methods and one of the xenon-arc methods are used to categorize the lightfastness of colorants.
- 1.1.1 *Test Method A*—Exposure in southern Florida to natural daylight filtered through window glass.
- 1.1.2 *Test Method B*—Exposure in Arizona to natural daylight filtered through window glass.
- 1.1.3 *Test Method C*—Exposure in a non-humidity controlled xenon-arc device simulating daylight filtered through window glass.
- 1.1.4 *Test Method D*—Exposure in a humidity controlled xenon-arc device simulating daylight filtered through window glass.
- 1.2 These test methods are used to approximate the color change that can be expected over time in colorants used in artists' materials exposed indoors to daylight through window glass.
- Note 1—The color changes that result from accelerated exposure may not duplicate the results of normal indoor exposure in a home, art gallery, or museum. The relative resistance to change, however, can be established so colored materials can be assigned to categories of relative lightfastness.
- Note 2—Users who wish to test colored materials under fluorescent illumination should consult Practice D4674.
- 1.3 Lightfastness categories are established to which colorants are assigned based on the color difference between specimens before and after exposure.
- 1.4 Color difference units are calculated by the CIE 1976 L\*a\*b\* color difference equation.
  - 1.5 These test methods apply to colored artists' materials.
- 1.6 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.7 This standard does not purport to address 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:<sup>2</sup>

D2244 Practice for Calculation of Color Tolerances and Color Differences from Instrumentally Measured Color Coordinates

D4302 Specification for Artists' Oil, Resin-Oil, and Alkyd Paints

D4674 Practice for Accelerated Testing for Color Stability of Plastics Exposed to Indoor Office Environments

D5067 Specification for Artists' Watercolor Paints

D5098 Specification for Artists' Acrylic Dispersion Paints

D5724 Specification for Gouache Paints

D6901 Specification for Artists' Colored Pencils

E284 Terminology of Appearance

E1347 Test Method for Color and Color-Difference Measurement by Tristimulus Colorimetry

E1348 Test Method for Transmittance and Color by Spectrophotometry Using Hemispherical Geometry

E1349 Test Method for Reflectance Factor and Color by Spectrophotometry Using Bidirectional (45°:0° or 0°:45°) Geometry

G24 Practice for Conducting Exposures to Daylight Filtered Through Glass

G113 Terminology Relating to Natural and Artificial Weathering Tests of Nonmetallic Materials

G141 Guide for Addressing Variability in Exposure Testing of Nonmetallic Materials

G151 Practice for Exposing Nonmetallic Materials in Accelerated Test Devices that Use Laboratory Light Sources
 G155 Practice for Operating Xenon Arc Light Apparatus for

<sup>&</sup>lt;sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.57 on Artist Paints and Related Materials.

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<sup>&</sup>lt;sup>2</sup> 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.



## Exposure of Non-Metallic Materials

## 3. Terminology

- 3.1 *Definitions*—Appearance terms used in these test methods are defined in Terminology E284. Terms relating to natural and artificial lightfastness tests are defined in Terminology G113.
- 3.1.1 *glass*, *n*—as used in these test methods, glass refers to single-strength window glass.

## 4. Summary of Test Methods

- 4.1 Color measurements are made on duplicate specimens that have been prepared as directed in the specification for that material. Examples of specifications are: D4302, D5067, D5098, D5724, and D6901. Each contains colorants in a different vehicle. The measurements are recorded for comparison with readings made after the specimens have been exposed.
- 4.2 Lightfastness is determined by exposing the specimens to daylight filtered through glass outdoors either in southern Florida or in Arizona and also to xenon arc radiation through a window glass filter.
- 4.3 The colorants are classified by the amount of color change calculated as  $\Delta E^*$  units in accordance with Practice D2244.
- 4.4 Variations in test results can be due to differences in specimen preparation, surface irregularities, color measurements and conditions of exposure. Allowance for these variations is made by assigning a wide range of color change n each of the five lightfastness categories. Colorants are placed in one of these categories based on the mean of the  $\Delta E^*$  values obtained from two or more types of exposure. Only colorants that place in the first two categories conform to the requirements of this standard.

## 5. Significance and Use /catalog/standards/sist/5583222

- 5.1 The retention of chromatic properties by a colorant over a long period of years is essential in a work of art. Accelerated exposure simulates color changes that may reasonably be expected. The producer and the user of artists' materials, therefore, can be apprised of suitable colorants.
- 5.2 Variations in results may be expected between the test methods. Also, some variation may be expected when the same test is repeated. Variations in Methods A and B are due to differences in outdoor conditions that are not accounted for in testing to equivalent radiant exposures. Information on sources of variability and strategies for addressing variability in laboratory accelerated exposure tests is found in Guide G141.
- 5.3 This standard does not cover factors other than light-fastness that can affect the permanence of art materials.

## 6. Apparatus

- 6.1 Outdoor Exposure Facilities as described in Practice G24, using an exposure angle of 45°, facing the equator.
- 6.2 Xenon-Arc Lightfastness Apparatus as described in Practice G155.
- 6.3 Spectrophotometer, abridged spectrophotometer or colorimeter capable of excluding specular reflectance in its measurement.

#### 7. Procedure

- 7.1 Prepare seven specimens of the art materials to be tested, following the directions given in the appropriate specification. If there is no specification for the art material, seven specimens must be prepared that are as similar, uniform, and opaque as possible.
- 7.1.1 Two specimens of each color shall be exposed in each of two test methods, either A or B and either C or D. One specimen of each color shall be retained for a visual comparison with the test specimens following exposure, and two specimens shall be retained for use in a third exposure if needed.
- 7.1.2 The retained, unexposed specimens are stored in the dark unless the formulation contains oil. Store specimens that contain oil in a light level of 500 to 700 lux (50 to 75 fc) to prevent yellowing. If specimens must be stored for as long as 100 days, store all specimens in the dark, but remove those containing oil and place in the light level specified above to prevent yellowing for at least 7 days before measurement or visual evaluation.
- 7.1.3 Cut the specimen to a size that will fit the holder to be used for exposure and the port of the color measuring instrument.
- 7.1.4 Determine if test specimens are opaque. Colors mixed with white, as described in the specifications for artists' paints, are opaque. Other materials must be applied over both a black substrate and a white substrate to determine opacity. Any measured color difference between the color over black and over white indicates a lack of opacity.
- 7.1.4.1 A measurement representative of the whole specimen must be obtained if the specimen is not opaque. To get a representative measure of the color, both before and after exposure. either (1) use a large measuring port of 25 mm (1 in.) diameter, or (2) using a small port, obtain the mean of a number of measurements of various areas of the specimens, and compare it with the mean of a second set of measurements of different areas. If the means agree, use that value as the representative color. Otherwise, repeat the procedure until agreement is obtained.
- 7.2 Immediately before exposure, measure all test specimens using a spectrophotometer or spectrocolorimeter (see Test Method E1348 or E1349) or colorimeter (see Test Method E1347) using Illuminant D65 and the 1964 10° Observer and excluding specular reflection from the measurement. Record the CIELAB measurement data.
- 7.2.1 Measure specimen panels with any brush marks in the same direction and measure the same area of the panel before and after each exposure interval. If the design of the instrument allows, three readings at different locations on the panel should be made and the mean calculated. If feasible, mark on the back of the specimen the spot(s) measured, and remeasure these same spots following exposure.
- 7.3 Expose duplicate specimens in each of two test methods, outdoor Test Method A or B and xenon arc Test Method C or D, as described below:
- 7.3.1 Test Method A—Exposure in Southern Florida Below 27° Latitude to Natural Daylight Filtered Through Window Glass—Test Method A can be used for under glass outdoor

exposure if the material is an oil paint or acrylic dispersion paint on an aluminum substrate.

- 7.3.1.1 Mount duplicate specimens of each color on an open sided rack under glass and expose in southern Florida at a 45 degree angle to the horizontal facing south during October through May to a total global solar (290 to 2500 nm) radiation dose of 1260 MJ/m<sup>2</sup> incident on the glass, in accordance with Practice G24.
- 7.3.2 Test Method B—Exposure in Arizona to Natural Daylight Filtered Through Window Glass—Use Test Method B if the test specimens are prepared on a paper substrate or the vehicle is affected by the combination of high moisture content and temperature fluctuations that are characteristic of south Florida. Examples are watercolor and gouache paints, colored pencils, colored water-thinned inks, and pastels.
- 7.3.2.1 Mount duplicate specimens in an enclosed black box with a small fan to circulate the air and expose in Arizona at a 45 degree angle to the horizontal facing south during October through May to a total global solar (290 to 2500 nm) radiation dose of 1260 MJ/m<sup>2</sup> incident on the glass, in accordance with Practice G24.
- 7.3.3 Test Method C—Exposure Simulating Daylight Filtered Through Window Glass in a Xenon Arc Device That Does Not Control the Relative Humidity—This method will generally have a low relative humidity.
- 7.3.3.1 Use a xenon-arc device that conforms to the requirements defined in Practices G151 and G155. Unless otherwise specified, the spectral power distribution of the xenon-arc shall conform to the requirements in Practice G155 for xenon arc radiation through a window glass filter.
- (a) Place specimens in the test device in positions that conform with specifications in Practice G151 or use the procedures described in this practice that either ensure equal radiant exposure on all specimens or compensate for irradiance differences within the exposure chamber. To assure equal radiant exposure it may be necessary to reposition specimens during exposure.
- 7.3.3.2 Unless agreed otherwise, set the irradiance at the control point to  $0.35 \pm 0.02$  at 340 nm and expose specimens to 100 % light to reach a total radiant exposure of 510 kJ/(m²·nm) at 340 nm, the equivalent to 1260 MJ/m² of total solar radiation. For a xenon-arc device that controls exposure at 300 to 800 nm, set the irradiance at the control point to 500 W/m² and expose to 100 % light to reach a total radiant exposure of 739 MJ/m² at 300 to 800 nm. For xenon arc devices that control exposures in a different spectral region, consult the manufacturer of the device for the irradiance and radiant exposure required to produce equivalent test results.
- 7.3.3.3 The uninsulated black panel temperature shall be 63  $\pm$  2°C. For the equivalent insulated black panel temperature, consult the manufacturer of the device.
- Note 3—The set points specified for irradiance, temperature and humidity are the target conditions for the sensor programmed by the user at the control point. Therefore, when a standard calls for a particular set point, the user programs that exact number. The operational fluctuation specified with the set point does not imply that the user is allowed to program a set point higher or lower than the exact set point specified. Operational fluctuation is determined by the machine variable and is the

maximum deviation allowable from the set point of the sensor at the control point during equilibrium conditions.

Note 4—To track the rate of color change in the xenon arc exposure, the total exposure time can be divided into three or more phases and the device programmed to stop at the end of each phase so the specimens can be measured and recorded. Then specimens are returned to the test chamber and exposure continues until the total required amount of irradiation is reached.

7.3.4 Test Method D—Exposure Simulating Daylight Filtered Through Window Glass in a Humidity Controlled Xenon-Arc Device—This environment will typically have higher relative humidity than Test Method C:

7.3.4.1 Follow 7.3.3.1.

7.3.4.2 Mount specimens in unbacked holders and follow step (a) in 7.3.3.1. It is recommended that all unused spaces in the specimen exposure area be filled with blank metal panels that are not highly reflective.

7.3.4.3 Follow 7.3.3.2.

7.3.4.4 Follow 7.3.3.3.

7.3.4.5 Set the relative humidity at the control point in the test chamber to 55  $\pm$  5 % RH.

7.3.4.6 In machines that allow control of chamber air temperature, it shall be set at  $43 \pm 2$ °C.

Note 5—Duplicate specimens should not be placed near one another during the exposures.

Note 6—It has been found that Alizarin Crimson and other colorants are affected differently when exposed to a light/dark cycle rather than to continuous light. Dark periods are characteristic of exposure to daylight as well as to indoor lighting. Therefore, when mutually agreed upon, the following alternative light and dark cycle may be employed as an alternate constant light: 3.8 h light followed by 1 h dark. During the light period, the conditions of irradiance, temperature and humidity are as given in 7.3.3 or 7.3.4. During the dark period, the uninsulated black panel temperature shall be set at  $35 \pm 2^{\circ}$ C at the control point. In machines that allow control of air chamber temperature, it shall also be set at  $35 \pm 2^{\circ}$ C at the control point. In machines that allow control of relative humidity, it shall be set at  $55 \pm 5^{\circ}$  RH at the control point, during both light and dark periods. Any variance from the specified test cycle must be detailed in the Report section.

- 7.4 Following exposure, if the surface of an exposed test specimen appears excessively streaked or spotty, showing areas of white substrate, assign that colorant to Lightfastness V.
- 7.5 Shortly after exposure, measure the exposed specimens not already placed in Lightfastness V, using Illuminant D65 and the 1964  $10^{\circ}$  Observer and with specular reflection excluded. Record the measurement.
- 7.6 Calculate the color difference between the recorded measurement of the specimen before exposure and the recorded measurement of the specimen after exposure in accordance with Practice D2244 and state the color change in total color difference units  $\Delta E^*_{ab}$ .
- 7.7 Measure the retained (unexposed) specimen of each color and compare the measurement with the pre-exposure measurement of that specimen to verify that the retained specimen has not changed color significantly during storage.
- 7.7.1 Unless the color of the retained specimen has changed significantly during storage, visually compare the retained specimen of each material with the exposed specimen of that material to verify that the measured color difference agrees

with the perceived color difference. If the visual color difference is inconsistent with the color difference expressed in  $\Delta E^*_{ab}$  units, remeasure both specimens and recalculate the color difference. Make this check following any subsequent exposures.

- 7.8 Find the mean of the CIELAB color differences calculated in 7.6 for the two specimens exposed outdoors by Test Method A or B. Find the mean of the color differences calculated in 7.6 for the two specimens exposed in a xenon-arc device by Test Method C or D.
- 7.8.1 Each mean is the test result for the specimen in that type of exposure, unless there are 5  $\Delta E^*$  or more units of color change between the measurement of the two specimens. In this case compare the  $\Delta E^*$  units for the two specimens with the mean  $\Delta E^*$  units from the other type of exposure and discard the specimen data that differs more from that mean.
- 7.9 Find the combined mean of the outdoor test result and the xenon arc test result, obtained in 7.8 or 7.8.1, and use this mean to assign the colorant to a lightfastness category as described in Section 8, unless the two test results place the specimens in different lightfastness categories; or unless the mean of the two test results is within  $\pm 0.5~\Delta E*_{ab}$  of the dividing line between lightfastness categories.
- 7.9.1 If the outdoor and xenon arc test results place the colorant in different lightfastness categories, or if the mean of the two types of exposure is within  $\pm 0.5 \Delta E^*_{ab}$  of the dividing line between lightfastness categories, conduct a third test; or place the colorant in the poorer of the two relevant categories.
- 7.9.1.1 For the third test use a method not employed in the first two exposures; or if this is not possible, repeat the test method with the poorest results.
- 7.9.1.2 After the third test is complete and the mean color change determined, if there are 5  $\Delta E^*_{ab}$  or more units of color difference between one of the test results and the closest of the other two test results, discard that test result as aberrant. Find the mean of the three exposures, or two exposures if one has been discarded, and assign each colorant to a lightfastness category as described in Section 8.

## 8. Interpretation of Results

- 8.1 When a very light color loses all, or almost all color during exposure, this loss of color does not result in a large CIELAB color difference between the specimen before and after exposure. Therefore, place all very light materials whose test specimen bleached, or lost almost all color, into Lightfastness V regardless of the size of the CIELAB color change.
- 8.2 Lightfastness I—Assign colorants that exhibit a mean color change of 4 or less  $\Delta E^*_{ab}$  to Lightfastness Category I.
- 8.3 Lightfastness II—Assign colorants that exhibit a mean color change of more than 4.0 but not more than 8.0  $\Delta E^*_{ab}$  to Lightfastness Category II.
- 8.4 *Lightfastness III*—Assign colorants that exhibit a mean color change of more than 8.0 but not more than 16.0 to Lightfastness Category III.
- 8.5 *Lightfastness IV*—Assign colorants that exhibit a mean color change of more than 16.0 but not more than 24.0 to Lightfastness Category IV.

8.6 *Lightfastness V*—Assign colorants that exhibit a mean color change of more than 24.0, or have lost all but a trace of color, to Lightfastness Category V.

## 9. Report

- 9.1 The following applies to reports for all test methods:
- 9.1.1 Name of company,
- 9.1.2 Vehicle used,
- 9.1.3 Colour Index Names and Constitution Numbers for all colorants tested, when available,
  - 9.1.4 ASTM test methods used,
  - 9.1.5 Date when exposure began,
  - 9.1.6 CIELAB notation for test specimens prior to exposure,
- 9.1.7 Date when test specimens were removed from exposure and total exposure time,
- 9.1.8 CIELAB notation for test specimens following exposure. If it is not possible to measure specimens immediately after removal from exposure, give the date when measured,
- 9.1.9 The color difference in CIELAB  $\Delta E^*$  units for test specimens following exposure, and
- 9.1.10 Lightfastness category for all test specimens as determined in Section 8.
- 9.2 The following is specific information required for each of the test methods:
  - 9.2.1 Test Method A and B:
  - 9.2.1.1 Whether test was conducted in Florida or Arizona,
  - 9.2.1.2 Total solar radiant exposure, MJ/m<sup>2</sup>.
  - 9.2.2 Test Methods C and D:
  - 9.2.2.1 Name and model of device used,
- 9.2.2.2 Radiant exposure at the wavelength, or spectral range, in which measurements are made in kJ/(m<sup>2</sup>·nm) or MJ/m<sup>2</sup>, respectively,
- 3–9.2.2.3 Irradiance level at the wavelength, or spectral range, at which measurements are made.
- 9.2.2.4 Black panel temperature and type of black panel used.
- 9.2.2.5 Relative humidity and chamber air temperature, if controlled or measured, or both.
- 9.2.2.6 If the program includes a dark period, specify the light/dark periods.
  - 9.3 Panel repositioning schedule, if used.

#### 10. Precision and Bias

- 10.1 Precision—Variation in test results can result from differences in colorants manufactured from time to time within a company, different varieties of a colorant from company to company, specimen preparation, different instruments and instrumental readings, variations in the surface of the specimen, and the conditions of exposure. Allowance for these variables is made by requiring more than one test and by establishing lightfastness categories that include a range of color differences.
- 10.2 To establish the relationship between test methods, 5 sets of 172 paint specimens, 90 in linseed oil and 82 in acrylic dispersion vehicle, were made at the same time by one person and exposed in four sets of lightfastness tests: southern Florida daylight filtered through glass, Kansas daylight filtered through



glass, in a full spectrum fluorescent exposed apparatus, and in a window glass filtered xenon-arc.<sup>3</sup>

10.2.1 All four test methods placed 73 % of the colorants in the same category. When 12 aberrant test results were dropped from consideration and the third and fourth exposures conducted as required in cases where test results are near the border line between lightfastness categories (see 8.2), all combinations of the test results placed 99 % of the colorants in the same category.

10.3 *Bias*—Since there is no accepted reference material suitable for determining bias for the procedure in these test methods for measuring lightfastness, bias has not been determined.

## 11. Keywords

11.1 art materials; colorants; lightfastness; lightfastness categories; relative humidity; test specimens; window glass filtered daylight exposure; xenon arc

## **ANNEX**

(Mandatory Information)

## A1. FLUORESCENT DAYLIGHT LAMP LIGHTFASTNESS APPARATUS

A1.1 The apparatus for exposing paint specimens to fluorescent irradiation is shown in a large-size version in Fig. A1.1 and Fig. A1.2. A smaller version is shown in Fig. A1.3. Fig. A1.4 provides the dimensions and arrangement for constructing the larger size apparatus out of materials that are easily available.

A1.2 Cabinet Construction—Make framework for the apparatus out of  $1\frac{1}{2}$  by  $1\frac{1}{2}$  by  $\frac{1}{8}$ -in. (40 by 40 by 3.2-mm) aluminum angle available in hardware stores. The supports and the remainder of the enclosure are ½-in, tempered hardboard. The exposure panel supports, which are also the apparatus sides, are made removable by being fastened by reversed bolts with wing nuts. Coat them on the interior side with a non-yellowing white paint. Four reflectors, which each can hold four fluorescent lamps, are fastened together by the 1½-in. angles, back to back, to form a square in the center of the cabinet facing the specimens, which are attached to the hardboard panels support as shown in Fig. A1.4. Construct the framework so that the lamps are 3 in. (75 mm) from the test specimens. Cut holes in each side adjacent to the center and centered on a panel location to accommodate an Eppley pyranometer. With one panel space missing on each panel bank, the cabinet accommodates 108 test panels.

A1.3 *Irradiation*—The reflector fixtures hold sixteen 1.2-m (48-in.) long high-powered lamps with a correlated color temperature of 6000 K and a spectral irradiance simulating sunlight.

A1.3.1 Irradiance can be monitored by a spectral radiometer or a filter radiometer which has been calibrated to provide information on the total spectral radiant exposure.

A1.3.2 Install a timer, shown as K in Fig. A1.4, to cycle exposure for 8 h of light followed by 4 h of dark.

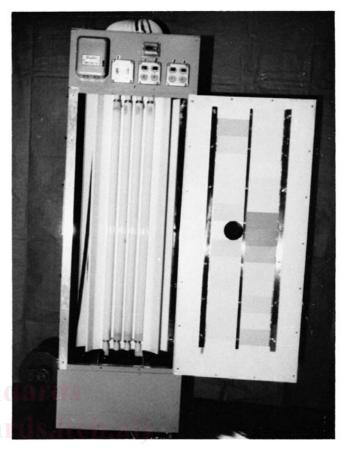
A1.3.3 Install an electric time meter in the circuit, shown as N in Fig. A1.4 to record the total amount of time the lamps are turned on.

A1.4 Temperature and Humidity Control—Install a centrifugal blower unit at the base of the cabinet, shown as I in Fig. A1.4. The air from the blower passes immediately through an air filter O and, after passing through the specimen exposure area, is vented out through the top of the cabinet. The blower furnishes the filtered air draft that keeps the temperature in the cabinet to within 6°C of the ambient temperature of the room. The small 6°C temperature rise is sufficient to limit the humidity in the cabinet to about 60 % relative humidity even when the general humidity is very high. Install a thermometer in the top of the cabinet near the vent and away from any direct illumination from the lights.

A1.5 If preferred, a smaller open apparatus holding a single bank of four daylight fluorescent lamps may be constructed as shown in Fig. A1.3. A small fan blowing through the unit from one end furnishes the air draft, but otherwise the apparatus must have the same distance between lamps and specimens and the same monitoring equipment described in A1.2 and A1.3.

 $<sup>^3</sup>$  Supporting data are available from ASTM International Headquarters. Request RR: D01-1036.





Note 1—Pyranometer, recorder, and demand readout meter in place. (a) Four-Bank Fluorescent Exposure Apparatus that Handles One Hundred and Eight 3 by 6-in. Panels

FIG. A1.1 Apparatus for Exposing Paint Specimens to Fluorescent Irradiation

Note 1—Notice hole for mounting Eppley pyranometer which is attached at right. (b) Four-Bank Verilux Fluorescent Exposure Apparatus with One Panel Open

FIG. A1.2 Apparatus for Exposing Paint Specimens to

Fluorescent Irradiation

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