

Designation: D 4303 - 99

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

This standard is issued under the fixed designation D 4303; 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 ( $\epsilon$ ) 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' paints are described as follows:
- 1.1.1 *Test Method A*—Exposure to natural daylight filtered through glass,
- 1.1.2 *Test Method B*—Exposure to irradiance from daylight fluorescent lamps,
- 1.1.3 *Test Method C*—Exposure in xenon-arc irradiance simulating daylight filtered through glass, and
- 1.1.4 *Test Method D*—Exposure to irradiance from cool white fluorescent lamps and soda-lime glass filtered fluorescent UV sunlamps.
- 1.2 These test methods are used to approximate the color change that can be expected over time in pigments used in artists' paints in normal indoor exposure.
- Note 1—The color changes that result from accelerated exposure may not duplicate the results of normal indoor exposure in a home or gallery. The relative resistance to change, however, can be established so pigments can be assigned to categories of relative lightfastness.
- 1.3 Lightfastness categories are established to which pigments 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 artists' oil, resin-oil, acrylic emulsion, alkyd, watercolor paints, and gouache paints.
- 1.6 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:
- D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates<sup>2</sup>
- <sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.57 on Artists' Paints and Related Materials.
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  - <sup>2</sup> Annual Book of ASTM Standards, Vol 06.01.

- D 4302 Specification for Artists' Oil, Resin–Oil, and Alkyd Paints<sup>3</sup>
- D 4674 Test Method for Accelerated Testing for Color Stability of Plastics Exposed to Indoor Fluorescent Lighting and Window Filtered Daylight<sup>4</sup>
- D 5067 Specification for Artists' Watercolor Paints<sup>3</sup>
- D 5098 Specification for Artists' Acrylic Emulsion Paints<sup>3</sup>
- D 5724 Specification for Gouache Paints<sup>3</sup>
- E 284 Terminology of Appearance<sup>2</sup>
- E 1347 Test Method for Color and Color–Difference Measurement by Tristimulus (Filter) Colorimetry<sup>2</sup>
- E 1348 Test Method for Transmittance and Color by Spectrophotometry Using Hemispherical Geometry<sup>2</sup>
- E 1349 Test Method for Reflectance Factor and Color by Spectrophotometry Using Bidirectional Geometry<sup>2</sup>
- G 24 Practice for Conducting Natural Light Exposures Under Glass<sup>5</sup>
- G 113 Terminology Relating to Natural and Artificial Weathering Tests of Nonmetallic Materials<sup>5</sup>
- G 151 Practice for Exposing Nonmetallic Materials in Accelerated Test Devices that Use Laboratory Light Sources<sup>5</sup>
- G 155 Practice for Operating Xenon Arc Light Apparatus for Exposure of Nonmetallic Materials<sup>5</sup>

#### 3. Terminology

- 3.1 *Definitions*—Appearance terms used in these test methods are defined in Definitions E 284. Terms relating to natural and artificial lightfastness tests are defined in Definitions G 113
- 3.1.1 *glass*—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 specimens that have been prepared as directed in Specification D 4302, D 5067, D 5098 or D 5724 depending on the vehicle. The measurements are recorded for comparison with readings made after the specimens have been exposed.

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol 06.02.

<sup>&</sup>lt;sup>4</sup> Annual Book of ASTM Standards, Vol 08.03.

<sup>&</sup>lt;sup>5</sup> Annual Book of ASTM Standards, Vol 14.02.

- 4.2 Specimens are exposed to daylight filtered through glass or to laboratory test sources to simulate and accelerate the effects of indoor illumination using at least two of the four test methods described in Section 7.
- 4.3 The pigments are classified by color difference calculated in accordance with Test Method D 2244.

### 5. Significance and Use

- 5.1 The retention of chromatic properties by a pigment over a long period of years is essential in an artistic painting. Accelerated exposure simulates color changes that may reasonably be expected. The producer and the user of artists' paints, therefore, can be apprised of suitable pigments.
- 5.2 There can be significant variations in results between the different exposure procedures used in these test methods. In addition, there can also be significant differences in results between exposures conducted in accordance with a single procedure because of variability inherent within the procedures themselves.

#### 6. Apparatus

- 6.1 Sun Exposure Facilities as described in Practice G 24, using an exposure angle of 45°, facing the equator.
- 6.2 Xenon-Arc Lightfastness Apparatus as described in Practice G 155.
- 6.3 Daylight Fluorescent Lamp Apparatus, <sup>6</sup> as described in Annex A1.
- 6.4 Fluorescent Cool White Lamp/Filtered Sunlamp Light-fastness Apparatus, using very high output cool white fluorescent lamps and soda-lime glass filtered fluorescent UV sunlamps as described in Test Method D 4674.
- 6.5 *Spectrophotometer*, abridged spectrophotometer or colorimeter capable of excluding specular reflectance in its measurement.

#### 7. Procedure

- 7.1 Specimens of pigments to be tested in oil, resin-oil, or alkyd panels shall be prepared as directed in Specification D 4302. Specimens of pigments to be tested in acrylic emulsion paints shall be prepared as directed in Specification D 5098. Specimens of pigments to be tested in watercolors shall be prepared as directed in Specification D 5067.
- 7.2 Immediately before exposure, measure all test specimens on a spectrophotometer or spectrocolorimeter (see Test Method E 1348 or E 1349) or colorimeter (see Test Method E 1347) using Illuminant C and the 1931 2° observer and excluding specular reflection from the measurement. Record the measurements.
- $^6$  The Verlilux lamps F48T12VLX/HO, manufactured by Verilux, Inc., 9 Viaduct Rd., Stamford, CT 06906, used in the apparatus described in Annex A1 are not available. Other fluorescent lamps that closely simulate daylight may be suitable, and Subcommittee D01.57 seeks information that will allow these fluorescent lamps to be recommended. Lamps should have a related color temperature of 6000  $\pm$  500 K and a color rendenng index of at least 90 as calculated by Method of Measuring and Specifying Colour-Rendering Properties of Light Sources, International Commission of Illumination (CIE) publication No. 13.2 (TC-3.2) 1974. Available from the U.S. National Committee of the CIE, c/o Director of Marketing, OEM Division, North American Philips Lighting Corp., Philip Square CN8800, Somerset, NJ 08873.

- 7.2.1 Read specimen panels always with any brush marks in the same direction and with panels in the same position so that the same area of the panel is measured 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.
- 7.2.2 If it is necessary to store specimens in the dark for a period of time, prior to measurement and exposure, those that contain oil in the vehicle shall be placed in subdued light for seven days to eliminate any yellowing of the oil due to storage.
- 7.2.3 Compare these pre-exposure measurements with subsequent measurements of the test specimens made at the end of exposure to calculate the amount of any color change. If feasible, measure the test specimens at regular, frequent intervals during exposure to calculate the rate of any color change.
- 7.2.4 Retain a specimen of each paint in the dark. After the test is complete, measure these retained specimens and compare with the pre-exposure measurement of the test specimens to verify that the retained specimens have not changed color significantly during storage.
- 7.2.5 Compare visually the retained, unexposed specimens with the exposed test specimens to verify that the measured color difference agrees with the perceived color change between the exposed and unexposed specimens. These retained specimens may also be needed for an additional test as described in 7.7.1 and 7.7.2.
- 7.3 Expose specimen panels in at least two of the four test methods described. Oil, resin-oil alkyd, and acrylic emulsion paint specimens shall be exposed by Test Method A and either Test Method B, Test Method C or Test Method D. Watercolors, gouache, and other paints sensitive to moisture shall be exposed by Test Methods C and either Test Method B or Test Method D.
- 7.3.1 Test Method A—Exposure to Natural Daylight Filtered Through Glass:
- 7.3.1.1 Mount the specimens on an open-type rack and expose at  $45^{\circ}$  angle to the vertical to a total radiation dose of 1260 MJ/m  $^2$  in accordance with Practice G 24.
- 7.3.1.2 For tests in southern Florida expose panels during the months of October through May.
- 7.3.2 Test Method B— Exposure to Irradiance from Daylight Fluorescent Lamps:
- 7.3.2.1 Mount the specimens with the test face 3 in. (75 mm) from the plane of the lamps.
- 7.3.2.2 Unless specified otherwise, expose the specimens to a repeating cycle of 8 h light followed by 4 h dark until the specimen has been exposed to a total radiant energy dose of 1260 MJ/m<sup>2</sup>. Rotate the specimen panels two positions to the right after each 100-MJ/m<sup>2</sup> dose. This test method takes approximately nine months to complete.
- 7.3.2.3 Maintain the ambient room temperature at  $24 \pm 3$  °C and prevent the test chamber from exceeding room temperature by more than 6°C.
- 7.3.2.4 Monitor irradiance at the specimen location for total radiation for each rotation period. Measure radiation 1 h after



the lamps are turned on and at the end of the period. The mean of these readings in joules per square centimetre (J/cm<sup>2</sup>) per hour multiplied by the number of hours of radiation gives the J/cm<sup>2</sup> for that period.

- 7.3.3 Test Method C— Exposure to Xenoc-Arc Irradiance Simulating Daylight Filtered Through Glass:
- 7.3.3.1 Mount specimens in unbacked holders avoiding positions that place specimens at the extreme top or bottom of the specimen rack.

7.3.3.2 Unless agreed otherwise, expose specimens in accordance with Practices G 151and G 155 to total radiation from the filtered xenon arc to reach a radiant exposure of 510 kJ/m² measured at 340 nm. Expose the specimens to 100 % light from the xenon arc apparatus equipped with window glass filters. Set the machine to maintain an irradiance level at 0.35 W/m²/nm at 340 nm and maintain the relative humidity in the test chamber at 55  $\pm$  5 % RH. The black panel temperature shall be 63  $\pm$  2°C. This radiant exposure measured at 340 nm has been calculated to provide total UV radiant exposure equivalent to the total UV behind glass-filtered daylight when total spectral radiant exposure to glass-filtered daylight is 1260 MJ/m². See Appendix X1.

NOTE 2—When mutually agreed upon, the following alternative light and dark cycle may be employed in accordance with Practices G 151 and G 155. Expose test specimens to the following cycle:

Set the xenon arc apparatus equipped with window glass filters to maintain an irradiance level of 0.35 W/m<sup>2</sup>/nm at 340 nm.

*Light Cycle:* 3.8 h light, at  $35 \pm 5$  % RH. The black panel temperature shall be  $63 \pm 2$  °C. Followed by:

*Dark Cycle:* 1 h dark, at 90  $\pm$  5 % RH. The black panel temperature shall be 35  $\pm$  3°C.

It has been found that Alizarin Crimson, and possibly other pigments, are affected by a light and dark cycle, owing to the oxidation-reduction effect of titanium dioxide changing valence with the changes from light to dark and vice-versa, characteristic of daylight and indoor light.

Any variance from the specified test cycle must be detailed in the Report section.

7.3.4 Test Method D—Exposure to Irradiance From Very High Output Cool White Fluorescent Lamps and Intermittent Exposure to Soda-Lime Glass Filtered Fluorescent UV Sunlamps Representative of Illumination in an Indoor Environment:

7.3.4.1 Two suitable reference pigments must be determined and agreed to for each vehicle type  $^7$ . To establish reference pigments, formulate two suitable pigments in the relevant vehicle, prepare test specimens and expose them using both Method A and Method C. Both test specimens must show a color difference before and after exposure that places them in Lightfastness Category II ( $\Delta$ E\*>4 and >8) in both the Method A and Method C tests. To conform to Test Method D, these reference pigments, purchased from the same manufacturer(s) and dispersed in the vehicle used to establish their lightfastness, must be included as controls when testing colors in that vehicle. Test Method D is complete when test specimens of

both or the two reference paints show a color difference greater than four and less than 8 CIELAB units (CIELAB  $\Delta E^*>4.0$  and >8).

7.3.4.2 Verify proper lamp function before starting test. Pre-age the lamps by leaving them on for a minimum of 48 h prior to the initial test. Radiometer readings at the start of the test shall not be less than  $8.0 \text{ W/m}^2$  for the 1500-mA lamps and  $1.0 \text{ W/m}^2$  for the 430-mA lamps.

7.3.4.3 Adjust the specimens or specimen table so that the surface of all test specimens are within 3 mm of being coplanar with the cosine receptor.

7.3.4.4 Close the specimen drawer and verify lamp irradiance in accordance with 7.3.4.2.

7.3.4.5 Turn on both sets of lamps. After 20 min turn off fluorescent sunlamps (FS) and record the cool white (CW) irradiance (radiometer reading) in W/m<sup>2</sup>.

7.3.4.6 Turn off the CW lamps and turn on the FS lamps. Record the radiometer reading (W/m<sup>2</sup>). Calculate the off-time internal cycle of the FS lamp so the average nominal sunlamp UV actinic exposure is set at 12 % of the value of the VHO lamps. An example calculation is as follows: Radiometer readings:

$$CW = 10.3 \text{ W/m}^2, FS = 2.4 \text{ W/m}^2$$
 (1)

In 1 h:

CW radiation = 
$$10.3 \text{ W/m}^2 \times 3600$$
 (2)

$$=$$
 s 37 080 J/m<sup>2</sup>  $\times$  0.12 = 4450

FS radiation =  $2.4 \text{ W/m}^2 \times 3600 \text{ s} = 8640 \text{ J/m}^2$ 

The On-Time required for the FS lamps is 4450/8640 = 0.515 h.

The Off-Time interval is 0.485 h/h total operation of the unit. 0.47.3.4.7 Program the FS lamp cycling to a 1-h time on interval/cycle.

7.3.4.8 Program the FS lamp timer to the off-time interval calculated in the example in 7.3.4.6.

7.3.4.9 Start the test. Do not add specimens once the test has begun.

7.3.4.10 Rotate specimens every 200 h by moving those in the innermost row (adjacent to median) to the outermost row of the same specimen tray, move all remaining rows one row closer to the median.

7.3.4.11 Test is complete when reference pigment specimens show a CIELAB  $\Delta E^*>4.0$ .

7.4 Measure test specimens immediately after exposure with specular reflection excluded in the measurement and record.

7.5 Calculate the color difference between the specimen before exposure *and* after exposure in accordance with the CIE 1976 L\*a\*b\* color difference equation in Test Method D 2244. State the color change in total color difference units ( $\Delta E^*$ <sub>ab</sub>).

7.6 Visually check the exposed specimen against the unexposed specimen of the same paint kept in subdued light, in order to verify that the color difference stated in  $\Delta E^*_{ab}$  units agrees with visual assessment. Make this check also following any subsequent exposures.

7.7 Since all test methods can produce aberrant results in pigments that are sensitive to some aspect of a particular type

 $<sup>^7\,</sup>PR$  170 (F5RK)-Novaperm Red F5RK, manufactured by Clariant Corp., and PY 3-Arylide Yellow 272-1007, manufactured by Sun Chemical Corp., have been found satisfactory in both oil and acrylic vehicles.



of exposure, assign pigments to lightfastness categories based on results from a minimum of two of the test methods described in 7.3.1-7.3.4:

7.7.1 Unless specified otherwise, expose one specimen inland outdoors under glass to daylight in southern Florida below 27° latitude following Test Method A and expose the second specimen indoors to simulated daylight behind glass following either Test Method B, Test Method C, or Test Method D. Place pigment in the appropriate category if both tests indicate the same category and neither result is within  $\pm 0.5~\Delta E^*$   $_{ab}$  of the borderline between categories.

7.7.2 If the results from the first two tests place the pigment in different lightfastness categories, or if either of the test results fall within  $\pm 0.5~\Delta E^*_{ab}$  of the dividing line between two categories, either assign the pigment to the poorer of the two categories involved or for a more accurate rating, proceed with the third exposure. The third exposure may use either the test method not used in the two previous exposures, or may repeat the exposure which gave the poorer result, providing the two test results are within 4  $\Delta E^*_{ab}$  of one another. To guard against accepting an aberrant test result, if there is more than 4  $\Delta E^*_{ab}$  difference between the first two test results, use the test method not employed in the first two tests for the third exposure.

7.7.3 Calculate the mean of the color differences from the three exposures to determine the appropriate lightfastness category unless one of them differs from the nearest test result by more than 4  $\Delta E^*$   $_{ab}$ , or the mean is within  $\pm 0.5$   $\Delta E^*$   $_{ab}$  of the dividing line between categories. In these cases make a fourth exposure.

7.7.4 For the fourth exposure use the test method not previously employed, or repeat the test method with poorest result, unless the results from that test method differ from the nearest other test result by more than 4  $\Delta E^*_{ab}$ . In this case, discard this one test result. Calculate the mean of the test results from the four exposures, or three exposures if one has been discarded, to determine the lightfastness category.

#### 8. Interpretation of Results

- 8.1 Lightfastness I—Assign pigments that exhibit a mean color change of 4 or less  $\Delta E^*_{ab}$  to Lightfastness Category I.
- 8.2 *Lightfastness II*—Assign pigments that exhibit a mean color change of more than 4.0 but not more than 8.0  $\Delta E^*$ <sub>ab</sub> to Lightfastness Category II.
- 8.3 *Lightfastness III*—Assign pigments that exhibit a mean color change of more than 8.0 but not more than 16.0 to Lightfastness Category III.
- 8.4 *Lightfastness IV*—Assign pigments that exhibit a mean color change of more than 16.0 but not more than 24.0 to Lightfastness Category IV.
- 8.5 *Lightfastness V*—Assign pigments that exhibit a mean color change of more than 24.0 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 ASTM test methods used,
- 9.1.3 Vehicle used.
- 9.1.4 Colour Index Names and Constitution Numbers for all pigments tested,

- 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 rated,
- 9.1.9 Give the color difference in CIELAB  $\Delta E^*$  for reference and test specimens prior to and following exposure, and
- 9.1.10 Lightfastness category for all test specimens as determined in 7.7.
- 9.2 The following is specific information required for each of the test methods:
  - 9.2.1 Test Method A:
  - 9.2.1.1 Total spectral radiant exposure, MJ/m<sup>2</sup>.
  - 9.2.2 Test Method B:
  - 9.2.2.1 Total spectral radiant exposure, MJ/m<sup>2</sup>, and
- 9.2.2.2 If the program includes a dark period, specify the light/dark periods.
  - 9.2.3 Test Method C:
  - 9.2.3.1 Name and model of apparatus used,
- 9.2.3.2 Irradiance level,  $W/m^2$ , for the control point or spectral range being measured,
  - 9.2.3.3 Radiant exposure at 340 nm, kJ/m<sup>2</sup>,
  - 9.2.3.4 Relative humidity,
  - 9.2.3.5 Black panel temperature,
  - 9.2.3.6 Dry bulb temperature, and
  - 9.2.3.7 Panel rotation schedule.
  - 9.2.4 Test Method D:
  - 9.2.4.1 Initial CW UV irradiance, W/m<sup>2</sup>,
  - 9.2.4.2 Final CW UV irradiance, W/m<sup>2</sup>,
  - 9.2.4.3 Initial FS UV irradiance, W/m<sup>2</sup>,
  - 9.2.4.4 Final FS UV irradiance, W/m<sup>2</sup>, astm-d4303-99
  - 9.2.4.5 FS On Time, and
  - 9.2.4.6 Panel rotation schedule.

## 10. Precision and Bias

10.1 Precision—Variation in test results can result from differences in pigment manufacture from time to time within a company, different varieties of a pigment 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 oil and 82 in acrylic emulsion vehicle, were made at the same time by one person and exposed in four sets of lightfastness tests: southern Florida sun filtered through glass, Kansas sun filtered through glass, fluorescent apparatus, and xenon-arc apparatus.<sup>8</sup>

 $<sup>^8\,\</sup>mathrm{Supporting}$  data are available from ASTM Headquarters. Request RR: D01–1036.