



Designation: **E2539 – 14** **E2539 – 14 (Reapproved 2017)**

Standard Test Method for Multiangle Color Measurement of Interference Pigments¹

This standard is issued under the fixed designation E2539; 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.

INTRODUCTION

Objects that exhibit a change in color with different angles of illumination and view are said to be “gonioapparent.” The tristimulus colorimetric values of gonioapparent objects are derived using the spectral reflectance factors obtained from spectrometric measurements or colorimetric measurements at various angles of illumination and detection. The tristimulus colorimetric values are computed using the spectral reflectance factors of the object, the CIE Standard Observer, and the spectral power distribution of the illuminant, as described in Practice E308. This Test Method, E2539, specifies the color measurement of interference pigments at various illumination and detection angles.

1. Scope

1.1 This test method covers the instrumental requirements and required parameters needed to make instrumental color measurements of thin film interference pigments. This test method is designed to encompass interference pigments used in architectural applications, automobiles, coatings, cosmetics, inks, packaging, paints, plastics, printing, security, and other applications.

1.2 Characterization of the optical behavior of materials colored with interference pigments requires measurement at multiple angles of illumination and detection.

1.3 Data taken utilizing this test method are quantitative and are appropriate for quality control of interference pigment color.

1.4 The measurement results are usually expressed as reflectance factors, tristimulus color values, or as CIE L*a*b* color coordinates and color difference.

1.5 The totality of data taken may not be necessary for evaluating mixtures also containing non-interference pigments. The committee is investigating and evaluating the appropriateness of this test method for those materials. It is the responsibility of the users to determine the applicability of this test method for their specific applications.

1.6 Interference pigments are typically evaluated for color and color appearance in a medium, such as paint or ink. The gonioapparent effect depends strongly on the physical and chemical properties of the medium. Some of the properties affecting color and color appearance include vehicle viscosity, thickness, transparency, and volume solids. As a general rule, for quality control purposes, interference pigments are best evaluated in a masstone product form. In some cases this product form may be the final product form, or more typically a qualified simulation of the intended product form (such as a paint drawdown) that in terms of color and appearance correlates to final product application.

1.7 This standard does not address the requirements for characterizing materials containing metal flake pigments. Measurements of the optical characteristics of materials containing metal flake pigments are described in Test Method E2194.

1.8 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.9 *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.*

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

¹ This test method is under the jurisdiction of ASTM Committee E12 on Color and Appearance and is the direct responsibility of Subcommittee E12.12 on Gonioapparent Color.

Current edition approved Nov. 1, 2014; June 1, 2017. Published November 2014; June 2017. Originally approved in 2008. Last previous edition approved in 2012 as E2539 – 12:14. DOI: 10.1520/E2539-14.10.1520/E2539-14R17.

2. Referenced Documents

2.1 ASTM Standards:²

E284 Terminology of Appearance

E308 Practice for Computing the Colors of Objects by Using the CIE System

E805 Practice for Identification of Instrumental Methods of Color or Color-Difference Measurement of Materials

E1164 Practice for Obtaining Spectrometric Data for Object-Color Evaluation

E1345 Practice for Reducing the Effect of Variability of Color Measurement by Use of Multiple Measurements

E1708 Practice for Electronic Interchange of Color and Appearance Data

E1767 Practice for Specifying the Geometries of Observation and Measurement to Characterize the Appearance of Materials

E2194 Test Method for Multiangle Color Measurement of Metal Flake Pigmented Materials

E2480 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method with Multi-Valued Measurands

2.2 ISCC Publications:³

Technical Report 2003–1 Guide to Material Standards and Their Use in Color Measurement

3. Terminology

3.1 Terms and definitions in Terminology E284, and Practice E1767 and Test Method E2194 are applicable to this test method. See Section 5 of E284 for “Specialized Terminology on Gonioapparent Phenomena.”

4. Summary of Test Method

4.1 This test method describes the instrumental geometries, including abridged goniospectrometry, used to measure interference pigments. Optical characterization requires color measurement at multiple illumination and multiple detection angles specified in this procedure. These sets of illumination and detection angles are specified in the test method. Standardization and verification of the instrument used to measure these materials are defined. The requirements for selection of specimens and measurement procedures are provided. The results are reported in terms of reflectance factors, CIE tristimulus values, and other color coordinate systems that define the color of the object. Expected values of precision are presented.

5. Significance and Use

5.1 This test method is designed to provide color data obtained from spectral reflectance factors at specific illumination and detection angles for interference pigments. Information presented in this test method is based upon data taken on materials exclusively pigmented with interference pigments.

5.2 These data can be used for acceptance testing, design purposes, research, manufacturing control, and quality control.

5.3 Specimens must be statistically representative of the end use.

5.4 Applicability of this test method for other materials, including combining interference pigments with absorbing and scattering pigments should be confirmed by the user.

6. Environmental Conditions

6.1 If the standard laboratory conditions listed below change during the test or from test to test by an appreciable amount, these conditions may reduce accuracy and precision of this test method. In some cases these effects may only be observed during the performance of the test.

6.2 *Factors affecting test results*—The following factors are known to affect the test results.

6.2.1 *Extraneous radiation*—light from sources other than the illuminator(s) and any near-infrared (NIR) must be shielded from entering the test apparatus.

6.2.2 *Vibrations*—mechanical oscillations that cause components of the apparatus to move relative to one another may cause errors in test results.

6.2.3 *Thermal changes*—temperature changes occurring during a test or differences in temperature between testing locations may affect calibration.

6.2.4 *Power input fluctuations*—large changes in the line frequency or supply voltage may cause the apparatus to report erroneous results.

6.3 *Standardization*—The system must allow for successful standardization. If the system cannot be standardized, consult the manufacturer’s user guide.

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

³ Available from the Inter-Society Color Council, 1191 Sunset Hills Road, Reston, VA 20190, www.iscc.org.

6.4 *Controlling factors*—Accuracy and precision can be enhanced by controlling and regulating each factor within the constraints of the allowable experimental error. The values and limits for these factors are typically determined experimentally by the user.

7. Apparatus

7.1 *Multiangle Spectrometer*—This test method specifies the required illumination and detection angles of multiangle spectrometers. These multiangle spectrometers are designed specifically to characterize the optical behavior of materials colored with interference pigments. Geometries are specified in Section 8. The spectrometer may either be a goniospectrometer or an abridged goniospectrometer.

7.1.1 Bi-directional spectrometers or colorimeters with a single angle of measurement; such as 45°:0° or 0°:45°, and spectrometers using hemispherical geometry cannot adequately characterize the gonioapparency of these materials.

7.1.2 Multiangle spectrometers or colorimeters similar to those specified in Test Method E2194 cannot adequately characterize the gonioapparency of these materials.

7.2 *System Validation Materials*—The precision and bias of the entire measurement system, including calculation of CIE tristimulus values, should be determined by periodic measurement of known, calibrated, verification standards. These standards are supplied by instrument manufacturers or obtained separately.⁴

8. Geometric Conditions

8.1 The angles of illumination and detection are critical to multiangle measurements of materials pigmented with interference pigments.

8.2 Recommended Geometries:

8.2.1 All geometries cited here are uniplanar.

8.2.2 *Geometry Designation*—The angles of illumination and detection will be specified as illumination anormal angle, detection anormal angle, and detection aspecular angle enclosed in parenthesis. See Practice E1767. For the example of an illumination angle of 45° and a detection angle of -30° (implying an aspecular angle of 15°), the geometry should be designated as 45°:-30° (as 15°).

NOTE 1—For either illumination or detection, an anormal angle is defined as the angle subtended at the point of incidence by a given ray and the normal to the surface. An anormal angle is understood to be the smaller of the two supplementary angles defined by the ray and the normal. In a uniplanar geometry, a ray's anormal angle has a positive sign if that ray and the incident ray (illuminant ray) are on the same side of the normal.

NOTE 2—The aspecular angle is the detection angle measured away from the specular direction, in the illumination plane. Positive values of the aspecular angle are in the direction toward the illumination axis.

8.2.3 For the reflectance-factor measurements of interference pigments, the instrument's illumination and detection angles shall conform to the angles as specified in Table 1. These angles are required to measure the range of colors exhibited by interference pigments.

8.2.4 For the reflectance-factor measurement of materials pigmented with metal-flake pigments and interference pigments, additional information is provided by angles specified in Table 2. These angles are used to measure the color travel due to pigment flake-orientation effects and light scattering from the flake edges.

9. Test Specimen(s)

9.1 *Introduction*—Measured values depend on the quality of the test specimens. The specimens must be statistically representative of the lot being tested and should meet the requirements listed below. If the specimens do not meet these requirements, include this information in the report (Section 14).

⁴ ISCC Technical Report 2003-1.

TABLE 1 Specified Geometries for Measuring the Color Range due to Interference

Illumination Angle	Detection Angle	Aspecular Angle	Designation
45°	-60°	-15°	45°:-60° (as-15°)
45°	-30°	+15°	45°:-30° (as15°)
15°	-30°	-15°	15°:-30° (as-15°)
15°	0°	+15°	15°:0° (as15°)

Note—This table gives the minimum geometries for the quality control application. For other applications, additional geometries; such as 65°:-50° (as15°), may be desirable or needed.

**TABLE 2 Specified Geometries for Measuring the Color due to Scattering or Orientation**

Illumination Angle	Detection Angle	Aspecular Angle	Designation
45°	-30°	15°	45°:-30° (as15°)*
45°	-20°	25°	45°:-20° (as25°)
45°	0°	45°	45°:0° (as45°)*
45°	30°	75°	45°:30° (as75°)
45°	65°	110°	45°:65° (as110°)*

Note—The three angles designated with an asterisk (*), refer to preferred angles for critical measurements as specified in Test Method E2194.

Note—Given a geometric configuration, the reverse geometry is considered equivalent, if all other components of the instrument design are equivalent.

9.2 *Specimen Handling*—Handle the specimens carefully. Touch them by their edges only. Never lay the measurement surface of the specimen down on another surface or stack specimens without a protective medium between them as recommended by the provider.

9.3 *Specimen Cleaning*—If necessary, clean the specimens following the providers' recommended cleaning procedure.

9.4 *Specimen Conditioning*—Allow specimens to stabilize in the measurement environment for a time period agreed to by the parties concerned.

9.5 *Specimen Physical Requirements:*

9.5.1 For test specimens that will be assessed visually, the size shall be at least 8 by 8 cm (approximately 3 by 3 in.). This specimen size is well suited for both visual assessment and instrumental measurement. See also 12.2.

NOTE 3—This recommendation for specimen size corresponds to the physical size required for observation by the CIE 1964 Standard Observer (10°). The specimen must subtend at least 10° when being observed. Observation usually occurs at approximately 45 cm (17.7 in.) from the eye.

9.5.2 The surface of the specimen should be planar.

9.6 *Specimen Optical Requirements:*

9.6.1 *Uniformity*—Reference and test specimens should be uniform in color and appearance. For materials pigmented with interference or metallic pigments, measurements on different locations on the sample are necessary to assess the degree of non-uniformity. These data are also useful for determining the number of measurements necessary to achieve a value that is statistically representative of the sample. See Practice E1345. Additionally, the samples must be similar in appearance to make meaningful observations. There should be no appearance of mottling or banding in the specimens.

9.6.2 *Gloss*—Specimens should be uniform and similar in gloss when viewed in a lighting booth.

9.6.3 *Surface Texture*—The specimens being compared should have substantially similar surface textures. Orange peel is a common example of surface texture.

9.6.4 *Orientation*—Consistent orientation of the specimen for presentation to the measuring instrument must be controlled for repeatable measurements. This is necessary to minimize errors due to indiscriminate matching of the directionality of the specimen to that of the instrument.

10. Instrument Standardization

10.1 Standardization is necessary to adjust the instruments output to correspond to a previously established calibration using one or more homogeneous specimens or reference materials. For the measurement of reflectance factor, full scale and zero standardization are necessary. See Practice E1164.

10.2 *Full-Scale Standardization Plaque*—A standardization plaque with assigned spectral reflectance factors relative to the perfect reflecting diffuser, traceable to a national standardizing laboratory, for each illumination and detection angle is required to standardize the instrument. The instrument manufacturer typically supplies and assigns the standardization values to this plaque.

NOTE 4—Different instrumentation manufacturers use different international standardization laboratories, different calibration techniques, and different standard reference materials. These factors and others may influence the numerical values obtained from subsequent measurements and thus care must be exercised when comparing values obtained from different instruments.

10.3 *Zero (0) Level Standardization*—Standardization of the zero (0) level is required for every geometry. The instrument may perform an internal calibration of the zero level by taking a measurement when there is no light in the optical path or a black standardization may be required.

10.4 Follow the instrument manufacturer's guidelines for standardization carefully.

11. Instrumental Performance Validation

11.1 *Introduction*—The use of verification standards to validate spectrometer performance of an instrument is recommended. These standards are readily available from multiple sources.⁴ The instrument user should assume responsibility for obtaining these standards and their appropriate use.

11.2 *Full Scale Reflectance Factor Scale Validation*—To ascertain proper standardization, it is recommended to measure a reference plaque immediately after the standardization sequence and validate that the measured values agree with the assigned values within ±0.05 CIELAB values.

11.2.1 *Discussion*—Typically, another tile is used for this purpose.

11.3 *System Performance Validation*—The precision and bias of the entire measuring system including calculation of CIE tristimulus values should be validated periodically by using calibrated verification standards. These standards may be supplied by the manufacturer or other sources.

11.3.1 *Discussion*—A green tile is often used to validate wavelength stability. Materials containing interference pigments are often used to validate the stability of instrument geometries.

11.4 Follow the instrument ~~manufacturer's~~manufacturer's guidelines for validation carefully.

12. Measurement Procedure

12.1 *Select Measurement Variables*—Select and validate the measurement parameters before initiating the measurement sequence.

12.1.1 Select the illumination and detection geometries. See Section 8 for the specification of angles when measuring gonioapparent materials pigmented with interference pigments.

12.1.2 Select the desired standard observer function.

12.1.3 Select the desired illuminant.

12.1.4 Select the desired CIE colorimetric space such as CIELAB.

12.2 Variation in measurements of gonioapparent materials is largely due to the inherent non-uniformity of these materials and the difficulty in positioning non flat samples relative to the measurement device.

12.3 Averaging the values made from multiple measurements across the surface of the specimen will help determine the statistical value that is representative of the specimen being measured and the desired precision. Refer to Practice E1345 for a description of averaging practice to improve precision.

12.4 Measure the specimen(s) in accordance with the instrument manufacturer's instructions or other specifications agreed to between buyer and seller.

13. Calculations

13.1 Using spectral reflectance factor data obtained by measuring the specimen, compute the CIE colorimetric values in accordance with Practice E308. Report data as specified in Practice E805 and Section 14 of this test method. It is highly recommended that instrumental readings be corrected for finite bandpass by a standard method of deconvolution.

14. Reports

14.1 It is recommended that the test data be submitted in electronic form;⁵ however, written data are acceptable.

14.2 The report of the measurement must include the minimum reporting requirements. Additionally recommended reporting requirements may be included. These requirements are presented in Table 3.

TABLE 3 Reporting Parameters

Function	Required	Recommended
14.3 Logistic Data		
14.3.1 Test Operator	✓	
14.3.2 Date of Test	✓	
14.3.4 Location of Test		✓
14.3.5 Time of Test		✓
14.3.6 Temperature and Relative Humidity		✓
14.4 Specimen Description		
14.4.1 Type and Identification	✓	
14.4.2 Date of Manufacture		✓
14.4.3 Method of Specimen Preparation		✓
14.4.4 Date of Specimen Preparation		✓
14.4.5 Specimen Orientation During Measurement	✓	

⁵ Refer to Standard Practice E1708.

14.4.6 Any changes that occurred to the specimen as a result of the measurement process	✓	
14.4.7 Any relevant observations by technician	✓	
14.5 Instrument Parameters		
14.5.1 Instrument Identification	✓	
14.5.2 Instrument Manufacturer		✓
14.5.3 Model Description	✓	
14.5.4 Serial Number		✓
14.5.5 Instrument Configuration	✓	
14.5.6 Observer	✓	
14.5.7 Illuminant	✓	
14.5.8 Color Scale	✓	
14.5.9 Index		✓
14.6 Instrument Geometry		
14.6.1 Specify Angles	✓	
14.7 Instrument Spectral Parameters		
14.7.1 Wavelength Range		✓
14.7.2 Wavelength Interval		✓
14.7.3 Spectral Bandpass		✓
14.8 Standardization		
14.8.1 Full Scale Standardization Plaque		✓
14.8.2 Time and Date of Last Standardization		✓
14.9 Specimen Data		
14.9.1 Spectral data for each angle of measurement as a function of wavelength. (Note that this is not applicable for spectrophotometers or colorimeters.)		✓
14.9.2 Color Coordinates data for each designated measurement geometry	✓	

15. Precision and Bias

15.1 Repeatability (with Replacement)

15.1.1 *Material*—The data obtained and results reported here are based on different materials containing interference pigments. There were three gonioapparent specimens selected for the study. The fourth specimen is the instrument standardization plaque and is not a gonioapparent material.

15.1.1.1 A blue automotive coating containing Flex Product's ChromaFlair⁶ Cyan/Purple 230 light interference pigment prepared by DuPont Performance Coatings, Wilmington, DE.

15.1.1.2 A ChromaFlair⁶ coating designated Green/Purple 190, which is a light interference pigment, applied to the back side of a transparent polyester (plastic) substrate by Flex Products, Santa Rosa, CA. The sample is measured through the clear plastic side.

15.1.1.3 An IRIODIN⁷ coating, which is metal oxide coated mica, prepared by Merck, Darmstadt, Germany, and

15.1.1.4 The instrument standardization plaque, which is a homogeneous white material and not gonioapparent.

15.1.2 *Data Acquisition*—The repeatability data were obtained in a single laboratory during the month of May 2007. The instrument was standardized according to manufacturer's directions and the reflectance factors of the specimens were acquired. The specimen was removed and replaced for each measurement sequence; this measurement technique is called repeatability with replacement. A total of 32 consecutive measurements were gathered in the shortest possible period of time.

15.1.3 *Data Computation*—The 95 % Confidence Interval, CI, for the data were computed using the following method outlined in Practice E2480.

15.1.4 *Repeatability*—Two test results obtained under repeatability conditions, which are defined as measurements made in the same laboratory using the same test method by the same operator using the same equipment in the shortest possible period of time using specimens taken from one lot of homogeneous material, should be considered suspect to a 95 % repeatability limit if their values differ by more than the ΔE^*_{ab} as shown in Table 4.

15.2 *Reproducibility*—The reproducibility of this test method is being determined.

15.3 *Context Statement*—The precision statistics cited for this method must not be treated as exact mathematical quantities that are applicable to all spectrometers, uses, and materials. There will be times when differences occur that are greater than those predicted by the study leading to these results would imply. Sometimes these instances occur with greater or smaller frequency than the 95 % probability limit would imply. If more precise information is required in specific circumstances, those laboratories directly involved in a material comparison must conduct interlaboratory studies specifically aimed at the material of interest.

⁶ ChromaFlair is a registered trademark of Flex Products, Inc.

⁷ Iriodin is registered trademark of EMD Chemicals Inc., Darmstadt, Germany.