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Standard Practice for Color Measurement of Fluorescent Specimens Using the One-Monochromator Method¹

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INTRODUCTION

The fundamental procedure for evaluating the color of a fluorescent object is to obtain spectrometric data for specified illuminating and viewing conditions, and then use this data to compute tristimulus values based on an International Commission on Illumination (CIE) standard observer and a CIE standard illuminant. For a fluorescent object-color specimen, the spectral radiance factors used to calculate tristimulus values are made up of two components — an ordinary reflectance factor and a fluorescence factor ($\beta = \beta_S + \beta_F$). The magnitude of the fluorescent radiance factors, and consequently the measured total radiance factors and derived color values, vary directly with the spectral distribution of the instrument source illuminating the specimen. Consequently, the colorimetry of fluorescent object-color specimens requires greater control of the measurement parameters in order to obtain precise spectrometric and colorimetric data. In order to obtain repeatable and reproducible color values for fluorescent objects it is necessary that the illumination at the specimen surface closely duplicate the standard illuminant used in the color calculations. The considerations involved and the procedures used to obtain spectrometric data and compute colorimetric values for fluorescent specimens using a one-monochromator spectrometer are contained in this practice.

1. Scope <u>ASTM E991-2</u>

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- 1.1 This practice applies to the instrumental color measurement of fluorescent specimens excited by near ultraviolet and visible radiation that results in fluorescent emission within the visible range. It is not intended for other types of photoluminescent materials such as phosphorescent, chemiluminescent, or electroluminescent, nor is this practice intended for the measurement of the fluorescent properties for chemical analysis.
- 1.2 This practice describes the instrumental measurement requirements, calibration procedures, and material standards needed for the color measurement of fluorescent specimens when illuminated by simulated daylight approximating CIE Standard Illuminant D65 (CIE D65).
- 1.3 This practice is limited in scope to colorimetric spectrometers providing continuous broadband polychromatic illumination of the specimen and employing only a viewing monochromator for analyzing the radiation leaving the specimen.
- 1.4 This practice can be used for calculating total tristimulus values and total chromaticity coordinates for fluorescent colors in the CIE Color System for either the CIE 1931 Standard Colorimetric Observer or the CIE 1964 Supplementary Standard Colorimetric Observer.

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- 1.5 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 safety, health, and healthenvironmental practices and determine the applicability of regulatory limitations prior to use.
- 1.6 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.

2. Referenced Documents

2.1 ASTM Standards:²

D985 Test Method for Brightness of Pulp, Paper, and Paperboard (Directional Reflectance at 457 nm) (Withdrawn 2010)³

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

E179 Guide for Selection of Geometric Conditions for Measurement of Reflection and Transmission Properties of Materials

E284 Terminology of Appearance

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

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E1164 Practice for Obtaining Spectrometric Data for Object-Color Evaluation

E1247 Practice for Detecting Fluorescence in Object-Color Specimens by Spectrophotometry

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

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

E2152 Practice for Computing the Colors of Fluorescent Objects from Bispectral Photometric Data

E2153 Practice for Obtaining Bispectral Photometric Data for Evaluation of Fluorescent Color

E2214 Practice for Specifying and Verifying the Performance of Color-Measuring Instruments

E2301 Test Method for Daytime Colorimetric Properties of Fluorescent Retroreflective Sheeting and Marking Materials for High Visibility Traffic Control and Personal Safety Applications Using 45°:Normal Geometry

2.2 CIE Publications and Standards:⁴

CIE Publication CIE15:2004 CIE Publication CIE15:2018 Colorimetry, 3rd4th Edition

CIE Publication No: 51.2 A Method for Assessing the Quality of Daylight Simulators for Colorimetry

CIE Publication No. 76 Intercomparison on Measurement of (Total) Spectral Radiance Factor of Luminescent Specimens

2.3 TAPPI Standards:⁵

T 571om-03 Diffuse brightness of paper and paperboard (d/0)

2.4 ISO Standards:⁶

ISO 10526:1999ISO 11664-2:2007(E)/CIE S005/E-1998S14/E:2006 CIE Standard Illuminants for Colorimetry

ISO 11475:2004ISO 11475:2017 Paper and board — Determination of CIE whiteness, D65/10 degrees (outdoor daylight)

ISO 2469:1994ISO 2469:2014 Paper, board and pulps — Measurement of diffuse reflectance factor

3. Terminology

- 3.1 *Definitions*—The definitions contained in Guide E179, Terminology E284, Practice E1164, Practice E1767, and Practice E2153 are applicable to this test method:practice.
 - 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 *fluorescence*, n—this standard uses the term "fluorescence" as a general term, including both true fluorescence (with a luminescent decay time of less than 10^{-8} s) and phosphorescence with a delay time short enough to be indistinguishable from fluorescence for the purpose of colorimetry (see Practice E2153).
- 3.2.2 fluorescent white, n—white and near white specimens containing fluorescent whitening agents.
- 3.2.3 near ultraviolet radiation, n—optical radiation within the wavelength range from 300 to 380 nm.

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 The last approved version of this historical standard is referenced on www.astm.org.

⁴ Available in hard copy or on CD-ROM at CIE/USA c/o TLA, 7 Pond St., Salem, MA 01970 TMLatTLA@aol.com or electronically downloadable via the website from the CIE Webshop (www.cie.co.at) or from the National Committees of the CIE Central Bureau (www.cie.co.at). (for the USA cie-usnc.org).

⁵ Available from Technical Association of the Pulp and Paper Industry (TAPPI), 15 Technology Parkway South, Norcross, GA 30092, http://www.tappi.org.

⁶ Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.<u>ISO</u> Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, https://www.iso.org.



3.2.4 referee procedure, n—a mutually agree upon testing procedure utilized to resolve disputes over instrumentally tested material properties that are expressed numerically.

4. Summary of Practice

- 4.1 This practice applies to the instrumental color measurement of fluorescent specimens that are excited by near ultraviolet and visible radiation and emit within the visible range. For methods to determine whether specimens exhibit fluorescence see Practice E1247. This practice provides procedures for measuring the total spectral radiance factors of fluorescent object-color specimens under simulated daylight approximating CIE D65 using a one-monochromator colorimetric spectrometer and calculating total tristimulus values (XYZ) and total chromaticity coordinates (x,y) in the CIE Color System for either the CIE 1931 Standard Colorimetric Observer or the CIE 1964 Supplementary Standard Colorimetric Observer (see CIE Publication 15).
- 4.2 The instrument source should provide broadband illumination of the specimen from 300 to 780 nm and the spectral distribution of the illumination on the specimen should closely duplicate CIE D65 (see ISO 10526:1999ISO/CIE S005/E-1998). 11664-2:2007(E)/CIE S14/E:2006). When highest measurement precision and reproducibility are required, the wavelength range should extend from 300 to 830 nm. Precise colorimetry of ultraviolet-activated fluorescent specimens requires the instrument provide significant illumination intensity below 380 nm. For the measurement of visible-activated fluorescent specimens, which have negligible excitation below 380 nm, it is only required that the illumination on the specimen provide a close match to CIE D65 over the wavelength range 380 to 780 nm.
- 4.3 The colorimetric spectrometer should employ a bidirectional optical measuring system with 45:0 or 0:45 illuminating and viewing geometry. The wavelength dispersive element (monochromator) shall be positioned between the specimen and the detector system (see CIE Pub. 76). The instrument may employ annular, circumferential, or uniplanar influx or efflux optics. The use of Practice E1767 functional notation is recommended for the complete description of instrumentation geometry including cone angles, aperture size, etc. When the specimen exhibits directionality, and an instrument with uniplanar geometry is used, information on directionality may be obtained by measuring the specimens at two or more rotation angles. If information on directionality is not required, then multiple uniplanar measurements may be averaged, or an instrument with annular or circumferential geometry may be used. However, even with annular or circumferential influx or efflux optics, some of the variability induced by specimen-optical system interactions may remain and the application of the methods in Practice E1345 may help to reduce measurement variability.
- 4.4 The important steps in the calibration of such instruments, and the material standards required for these steps, are described. Guidelines are given for the selection of specimens to minimize the specimen's contribution to the measurement imprecision. Parameters are identified that must be specified when spectrometric measurements are required in specific test methods or other documents.
- 4.5 Most modern colorimetric spectrometers have the capacity to compute the color coordinates of the specimen immediately following the measurement. When this is the case, the user shall select the CIE Color System and CIE D65, then chose either the CIE 1931 (2°) Standard Observer or CIE 1964 (10°) Supplementary Observer (see Practice E308).

5. Significance and Use

- 5.1 The most general method for obtaining CIE tristimulus values or, through their transformation, other coordinates for describing the colors of fluorescent objects is by the use of spectrometric data obtained under defined and controlled conditions of illumination and viewing. This practice describes the instrumental measurement requirements, calibration procedures, and material standards needed for measuring the total spectral radiance factors of fluorescent specimens illuminated by simulated daylight approximating CIE D65 and calculating total tristimulus values and total chromaticity coordinates for either the CIE 1931 or 1964 observers.
- 5.2 The precise colorimetry of fluorescent specimens requires the spectral distribution of the instrument light source illuminating the specimen closely duplicate the colorimetric illuminant used for the calculation of tristimulus values, which is CIE D65 in this practice. The fundamental basis for this requirement follows from the defining property of a fluorescent specimen: instantaneous light emission resulting from electronic excitation by absorption of radiant energy (η) where the wavelengths of emission (λ) are



- as a rule longer than the excitation wavelengths (1). For a fluorescent specimen, the total spectral radiance factors used to calculate tristimulus values are the sum of two components an ordinary reflectance factor, $\beta(\lambda)_S$, and a fluorescence factor, $\beta(\eta,\lambda)_F$: $\beta(\lambda) = \beta(\lambda)_S + \beta(\eta,\lambda)_F$. Ordinary spectral reflectance factors are solely a function of the specimen's reflected radiance efficiency at the viewing wavelength (λ) and independent of the spectral distribution of the illumination. The values of the spectral fluorescent radiance factors at the viewing wavelength (λ) vary directly with the absolute spectral distribution of illumination within the excitation range (η), and consequently so will the total spectral radiance factors and derived colorimetric values. One-monochromator colorimetric spectrometers used in this practice are generally designed for the color measurement of ordinary (non-fluorescent) specimens and the precision with which they can measure the color of fluorescent specimens is directly dependent on how well the instrument illumination simulates CIE D65.
- 5.3 CIE D65 is a virtual illuminant that numerically defines a standardized spectral illumination distribution for daylight and not a physical light source_(2). There is no CIE recommendation for a standard source corresponding to CIE D65 nor is there a standardized method for rating the quality (or adequacy) of an instrument's simulation of CIE D65 for the general instrumental colorimetry of fluorescent specimens. The requirement that the instrument simulation of CIE D65 shall have a rating not worse than BB (CIELAB) as determined by the method of CIE Publication 51 has often been referenced. However, the method of CIE 51 is only suitable for ultraviolet-excited specimens evaluated for the CIE 1964 (10°) observer. The methods described in CIE 51 were developed for UV activated fluorescent whites and have not been proven to be applicable to visible-activated fluorescent specimens.

Note 1—Aging of the instrument lamp will occur with normal usage resulting in changes in the spectral distribution and intensity of the illumination on the specimen over time. Measurement of the spectral distribution of the illumination at the sample port and evaluation of the adequacy of the CIE D65 simulation at regular intervals are recommended.

- 5.4 Differences in the absolute spectral irradiance distribution on the specimen between instrument models can produce significant variation in the measured color values of fluorescent specimens and result in poor reproducibility (3). In order to reproduce adequately the spectral irradiance on the specimen required for maximum measurement reproducibility, it may be necessary for a single model of instrument to be specified for use by both buyer and seller.
- 5.5 This practice is primarily for the instrumental color measurement of chromatic fluorescent specimens. While use of this practice for the color measurement of fluorescent whites is not precluded, other standards are more commonly used for measurement of these types of specimens (4, 5, 6) (see Test Methods D985, ISO 11475, ISO 2469, and TAPPI T 571).

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5.6 For geometrically sensitive fluorescent specimens angular tolerances on the axes and the angular aperture sizes must be well defined by the user to ensure adequate repeatability and reproducibility. Significant variation in measurement results for engineered surfaces and optical materials, for example retroreflective sheeting, can result from differences in the absolute axis angles of illumination and viewing and absolute size of the apertures between instruments (7). In order to replicate the measurement geometry, absolute angles and angular tolerances between instruments that is required for maximum measurement reproducibility, it may be necessary for a single model of instrument to be specified for use by both buyer and seller.

Note 2—To ensure inter-instrument agreement in the measurement of specimens with intermediate gloss, for formulation, or retroreflective specimens, tight geometric tolerances are required of the instrument axis angles and the instrument aperture angles.

- 5.7 Bidirectional (45:0 or 0:45) geometry is recommended for this practice.
- 5.7.1 Hemispherical geometry using an integrating sphere is not recommended because of the spectral sphere error resulting from radiation emitted by the fluorescent specimen reflecting off the sphere wall and re-illuminating the specimen, thereby changing the spectral illuminance distribution on the specimen from that of the original instrument source (8).

Note 3—The spectral sphere error associated with hemispherical geometry decreases as the ratio of the internal area of the sphere to the measurement area increases. When the spectral sphere error is negligible, results obtained using hemispherical geometry may for some specimens under specific measurement conditions approach those obtained using 45:0 geometry (9).

⁷ The boldface numbers in parentheses refer to the list of references at the end of this practice.

- 5.8 This practice provides procedures for selecting the operating parameters of spectrometers used for providing data of the desired precision. It also provides for instrument calibration by means of artifact standards and selection of suitable specimens for obtaining precision in the measurements.
- 5.9 Bispectral colorimetry using a bidirectional optical measuring system with a 45:0 or 0:45 illuminating and viewing geometry should be used when a high level of repeatability and reproducibility are required. The bispectral, or two-monochromator, method is the definitive method for the determination of the general radiation-transfer properties of fluorescent specimens. The bispectral method is accepted as the referee procedure for obtaining illuminant-independent photometric data on a fluorescent specimen that can be used to calculate its color for any desired illuminant and observer. The advantage of the bispectral method is that it avoids the inaccuracies associated with source simulation and various methods of approximation (10, 11)) (see Practices E2152, E2153, and Test Method E2301).

6. Apparatus

- 6.1 One-monochromator colorimetric spectrometer providing continuous broadband polychromatic illumination of the specimen intended to simulate CIE D65 and having the monochromator positioned between the specimen and the detector system for analyzing the radiation leaving the specimen. Instruments suitable for this practice are typically those designed for the measurement of color coordinates of ordinary (non-fluorescent) reflecting specimens.
- 6.1.1 Bidirectional 45:0 or 0:45 geometry is recommended.
- 6.1.2 The instrument may employ annular, circumferential, or uniplanar influx (illumination) or efflux (viewing) optics. Annular optics is recommended unless information on specimen directionality is required in which case uniplanar optics should be used.
- 6.1.3 Hemispherical geometry using an integrating sphere is not generally recommended, but may be permissible where it can be shown that the spectral sphere error of the instrument is negligible.
- 6.2 The spectrometer should provide continuous broadband illumination of the specimen at a minimum from 340 to 700 nm, preferably from 300 to 830 nm, and the spectral distribution of the illumination should closely duplicate CIE D65.
- 6.2.1 For the measurement of ultraviolet-activated fluorescent specimens the instrument should provide illumination on the specimen at a minimum from 340 to 380 nm, preferably from 300 to 380 nm, and the spectral distribution of that illumination should closely duplicate CIE D65.
- 6.2.2 For the measurement of visible-activated fluorescent specimens the instrument should provide illumination on the specimen at a minimum from 380 to 700 nm, preferably from 380 to 780 nm, and the spectral distribution of that illumination should closely duplicate CIE D65.
- 6.3 The wavelength measurement interval should be 10 nm or less. See Practice E308 and Practice E1164 for spectral bandpass recommendations.
- 6.4 The instrument should be capable of reporting total spectral radiance factor values as a function of wavelength over the range from 400 to 700 nm in increments of 10 nm or less. The preferred range is from 380 to 780 nm.
- 6.5 The instrument or an attached computer should have the capacity to compute the color coordinates of the specimen immediately following the measurement. When this is the case, the instrument should provide for user selection of the CIE Color System, CIE D65 illuminant and either the CIE 1931 (2°) Standard Observer or CIE 1964 (10°) Supplementary Standard Observer (see Practice E308).
- 6.6 Standardization Materials, either supplied by the instrument manufacturer or obtained separately, as follows (see Practice E1164):
- 6.6.1 White Reflectance Standard (mandatory) verified to be non-photoluminescent and calibrated for the appropriate instrument geometry.



- 6.6.2 System Verification Materials: (1) for setting or verifying zero on the reflectance scale; (2) for verifying the wavelength scale; and (3) for evaluating stray light (optional).
- 6.6.3 Calibrated Verification Artifact Standards (recommended)—(see [recommended]—(see Practice E1164]—Verification of the precision and bias of the entire measurement system, including calculation of tristimulus values, should be conducted on a regular basis using both non-fluorescent and fluorescent color standards with calibration values traceable to an accredited National Standards Laboratory.

Note 4—Stable fluorescent color artifact standards are not widely available as Standard Reference Materials (SRMs). However, measurement services are available from Independent Testing Laboratories and National Standards Laboratories to calibrate artifacts for use as Verification Standards. If materials with engineered surfaces or optical materials, such as retroreflective sheeting, are to be measured, then calibrated artifact standards of these materials should be included in the set of verification standards.

7. Test Specimen

- 7.1 Measurement results will not be better than the test specimens used in the measurements.
- 7.1.1 Test specimens should be representative of the material being tested.
- 7.1.2 The user in accordance with relevant industry practice or the recommendations of the material's manufacturer should define the protocols and procedures to prepare and condition the test specimen prior to testing.
- 7.2 For highest precision and accuracy, select specimens with the following properties:
- 7.2.1 Specimens should be uniform in optical properties and free from obvious defects over the area illuminated and measured.
- 7.2.2 Test specimens should be tested mounted on the substrate utilized for the intended application as defined by the user.
- 7.2.3 When specimens are not completely opaque, the spectral reflectance properties of the material behind the specimen should be specified.
- 7.3 If specimens exhibit directionality, use appropriate procedures and calculations (see Practices E1164 and E1345).
- 7.4 Handle the specimen carefully; prevent touching the area to be measured to avoid contamination. When necessary, clean the specimen by using an agreed procedure. Care should be taken not to touch the area to be measured except for application of a suitable cleaning procedure. The condition of the specimens before and after measurement should be noted and reported.

Note 5—If cleaning is required, a mild nonfluorescent, nonionic detergent that does not leave a film can be used with a soft cloth, wipe, or bristle brush. It is very important that neither the cleaning solution nor the wipe contain optical brightener.

8. Standardization and Verification

- 8.1 Standardization and its verification are essential steps in ensuring that accurate results are obtained by spectrometric measurement (see Practice E1164). Standardization and verification may require the use of material standards not normally supplied by the instrument manufacturer. The instrument user must assume the responsibility for obtaining the necessary material standards.
- 8.2 Operate the instrument in accordance with the manufacturer's established procedures and Practice E1164.
- 8.3 Where provided for by the instrument design, verify and, if necessary, adjust the UV content of the illumination. Follow the instrument manufacturer's instructions.
- 8.4 The accuracy with which the illuminating source simulates CIE Illuminant D65 should be determined periodically by measurement of the spectral distribution of irradiance at the specimen port of the instrument.



- 8.5 Standardize or verify the calibration of the following at the time of measurement:
- 8.5.1 Zero setting of the reflectance scale (mandatory).
- 8.5.1.1 Use either a highly polished black glass standard with an assigned reflectance factor of zero, or a black-cavity light trap, placed flush against the specimen measurement port, with an assigned reflectance factor of zero in accordance with the instrument manufacturer's established procedures.
- 8.5.2 Full-scale value of the reflectance scale of the instrument by use of the white reflectance standard (mandatory). Follow the instrument manufacturer's instructions.
- 8.5.2.1 Express spectral radiances obtained as radiance factors relative to the perfect reflecting diffuser assigned a value of 1.000 (100.0%) at each wavelength.
- 8.5.3 Verify the calibration of the wavelength scale (recommended).
- 8.5.4 Verify the level of stray light in the instrument is adequately low (optional),
- 8.6 The precision and bias of the entire measurement system, including verification of total spectral radiance factors and calculation of CIE tristimulus values, should be determined periodically by measurement of calibrated fluorescent reference materials (recommended). The calibration of these reference materials should be traceable to national standardizing laboratories.

Note 6—When fluorescent retroreflective specimens are to be measured, the set of calibrated reference materials should include appropriate retroreflective product standards.

9. Procedure

- (https://standards.iteh.ai)
- 9.1 Operate the instrument in accordance with the manufacturer's established procedures and Practice E1164.
- 9.2 When required, select the CIE XYZ and CIE Yxy color scales, the CIE 1931 Standard Observer or CIE 1964 Supplementary Standard Observer, and CIE Standard Illuminant D65 for the computation of color coordinates.
- 9.2.1 Select other options, such as wavelength range and interval, when required. Follow the recommendation of this practice and instrument manufacturer's instructions or specified procedures.
- 9.3 Place the specimen, with backing material or mounted on a substrate as required, against the measurement port of the instrument.
- 9.4 Measure the specimen, following the instrument manufacturer's instructions.
- 9.5 Transcribe the data required for the report, when not printed by the instrument.
- 9.6 Perform calculations of CIE total tristimulus values (XYZ) and total chromaticity coordinates (x,y) for CIE D65 and either the CIE 1931 (2°) Standard Observer or CIE 1964 (10°) Supplementary Standard Observer that are not made automatically by the instrument (see Practices D2244 and E308).

10. Report

- 10.1 Report the following information (see Practices E691 and E1164):
- 10.2 Specimen description (see Practice E1164).
- 10.2.1 The description should include information on the source of the specimen and type of material, for example whether the sample is opaque, translucent or transparent, a textile or plastic, retroreflective, ultraviolet-activated or visible-activated.