Designation: E2735 – 14

Standard Guide for Selection of Calibrations Needed for X-ray Photoelectron Spectroscopy (XPS) Experiments[1]

This standard is issued under the fixed designation E2735; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide describes an approach to enable users and analysts to determine the calibrations and standards useful to obtain meaningful surface chemistry data with X-ray photoelectron spectroscopy (XPS) and to optimize the instrument for specific analysis objectives and data collection time.

1.2 This guide offers an organized collection of information or a series of options and does not recommend a specific course of action. This guide cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this guide will be applicable in all circumstances.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.*

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E995 Guide for Background Subtraction Techniques in Auger Electron Spectroscopy and X-Ray Photoelectron Spectroscopy
E996 Practice for Reporting Data in Auger Electron Spectroscopy and X-ray Photoelectron Spectroscopy
E1078 Guide for Specimen Preparation and Mounting in Surface Analysis
E1127 Guide for Depth Profiling in Auger Electron Spectroscopy
E1217 Practice for Determination of the Specimen Area Contributing to the Detected Signal in Auger Electron Spectrometers and Some X-Ray Photoelectron Spectrometers
E1523 Guide to Charge Control and Charge Referencing Techniques in X-Ray Photoelectron Spectroscopy
E1577 Guide for Reporting of Ion Beam Parameters Used in Surface Analysis
E1634 Guide for Performing Sputter Crater Depth Measurements
E1636 Practice for Analytically Describing Depth-Profile and Linescan-Profile Data by an Extended Logistic Function
E1829 Guide for Handling Specimens Prior to Surface Analysis
E2108 Practice for Calibration of the Electron Binding-Energy Scale of an X-Ray Photoelectron Spectrometer

2.2 *ISO Standards:*[3]

ISO 10810 Surface Chemical Analysis—Depth Profiling—Measurement of Sputtered Depth
ISO 14606 Surface Chemical Analysis—Sputter Depth Profiling—Optimisation Using Layered Systems as Reference Materials
ISO 14701 Surface Chemical Analysis—X-ray Photoelectron Spectroscopy—Measurement of Silicon Oxide Thickness
ISO 14976 Surface Chemical Analysis—Data Transfer Format
ISO 15470 Surface Chemical Analysis—X-ray Photoelectron Spectroscopy—Description of Selected Instrumental Performance Parameters

---

[1] This guide is under the jurisdiction of ASTM Committee E42 on Surface Analysis and is the direct responsibility of Subcommittee E42.03 on Auger Electron Spectroscopy and X-Ray Photoelectron Spectroscopy.

Current edition approved Feb. 1, 2014. Published February 2014. Originally approved in 2013. Last previous edition approved in 2013 as E2735–13. DOI: 10.1520/E2735-14.

[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 American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959. United States

ISO 15472 Surface Chemical Analysis—X-ray Photoelectron Spectrometers—Calibration of Energy Scales
ISO/TR 15969 Surface Chemical Analysis—Depth Profiling—Measurement of Sputtered Depth
ISO 18115-1 Surface Chemical Analysis—Vocabulary—Part 1: General Terms and Terms Used in Spectroscopy
ISO 18115-2 Surface Chemical Analysis—Vocabulary—Part 2: Terms Used in Scanning-Probe Microscopy
ISO 18116 Surface Chemical Analysis—Guidelines for Preparation and Mounting of Specimens for Analysis
ISO 18117 Surface Chemical Analysis—Handling of Specimens Prior to Analysis
ISO 18118 Surface Chemical Analysis—Auger Electron Spectroscopy and X-ray Photoelectron Spectroscopy—Guide to the Use of Experimentally Determined Relative Sensitivity Factors for the Quantitative Analysis of Homogeneous Materials
ISO/TR 18392 Surface Chemical Analysis—X-ray Photoelectron Spectroscopy—Procedures for Determining Backgrounds
ISO 18516 Surface Chemical Analysis—Auger Electron Spectroscopy and X-ray Photoelectron Spectroscopy—Determination of Lateral Resolution
ISO 19318 Surface Chemical Analysis—X-ray Photoelectron Spectroscopy—Reporting of Methods Used for Charge Control and Charge Correction
ISO/TR 19319 Surface Chemical Analysis—Auger Electron Spectroscopy and X-ray Photoelectron Spectroscopy—Determination of Lateral Resolution, Analysis Area and Sample Area Viewed by the Analyser
ISO 20903 Surface Chemical Analysis—Auger Electron Spectroscopy and X-ray Photoelectron Spectroscopy—Methods Used to Determine Peak Intensities and Information Required when Reporting Results
ISO 21270 Surface Chemical Analysis—X-ray Photoelectron and Auger Electron Spectrometers—Linearity of Intensity Scale
ISO 22335 Surface Chemical Analysis—Depth Profiling—Measurement of Sputtering Rate: Mesh-Replica Method Using a Mechanical Stylus Profilometer
ISO 24237 Surface Chemical Analysis—X-ray Photoelectron Spectroscopy—Repeatability and Constancy of Intensity Scale

3. Terminology

3.1 *Definitions*—For definitions of surface analysis terms used in this guide, see ISO 18115-1 and ISO 18115-2.

4. Significance and Use

4.1 The purpose of this guide is assist users and analysts in selecting the standardization procedures relevant to a defined XPS experiment. These experiments may be based, for example, upon material failure analysis, the determination of surface chemistry of a solid, or the composition profile of a thin film or coating. A series of options will be summarized giving the standards that are related to specific information requirements. ISO 15470 and ISO 10810 also aid XPS users in experiment design for typical samples. ASTM Committee E42 and ISO TC201 are in a continuous process of updating and adding standards and guides. It is recommended to refer to the ASTM and ISO websites for a current list of standards.

5. Procedure

5.1 *General Sample Characterization:*

5.1.1 *Sample History*—The analyst should obtain a summary of the background information of the sample, including descriptors, history, sample cleaning and handling, existing application data, bulk composition, and any prior analysis that has been conducted. The sample history, especially handling, packing and storage, can impact the approach needed to obtain the desired information. Because inadequate sample collection can sometimes destroy or minimize the ability to collect the desired information, it is often necessary to identify the needed information and establish the procedures to be used (5.2) before the surface analysis is performed.

5.1.2 *Vacuum Compatibility*—The compatibility of sample with instrument vacuum should be considered. Although some samples have inappropriately high vapor pressures for ambient temperature operation, some instruments may include a sample cooling stage, which allows these types of materials to be analyzed. Additionally, newer XPS systems often have improved vacuum pumps coupled with monochromatic X-ray sources (that do not heat the sample) and a small X-ray spot size (requiring less sample for analysis). As a result, strongly outgassing or subliming samples can often be examined.

5.2 *Design of Experiment:*

5.2.1 The goal of the experiment should be defined. Experimental goals may include data relating to the surface chemical composition and chemical state, surface segregation, quantification, layer thickness, nanostructures, and so forth. The identification of the specific analysis objectives influences sample handling, instrument setup, the approach to data collection, and finally the methods of data analysis.

5.2.1.1 Table 1 is a summary of possible experiments along with different calibrations required to obtain meaningful data or to optimize the instrument for the best data in the time available.[4] Also included are the ASTM and ISO standards for checking the parameter. In the table, an X indicates applications where a calibration is required. Additionally, the calibrations are ranked with X = generally important and XX = generally very important calibrations for a given task.

5.2.2 *General System Check*—The analyst should perform a general system health check (including mechanical components, sample holders, vacuum level, and performance check) as recommended by the instrument manufacturer. Many analysts have also developed their own methods to verify the general operational health of an instrument. This might be done, for example, by testing a specimen commonly analyzed by the instrument to quickly verify the binding energies of few photoelectron peaks and overall count rates. Based upon the experiment to be performed, the relative importance of the parameters in Table 1 should be assessed, including calibration of the binding-energy scale, intensity repeatability and

[4] Castle, J. E., Powell, C. J., Report on the 34th IUVSTA Workshop, XPS: From Spectra to Results—Towards an Expert System, *Surface and Interface Analysis*, Vol 26, 2004, pp. 225–237.

TABLE 1 Recommended XPS Calibrations for Defined Experiments

NOTE 1—The X indicates applications where a calibration is required. X = generally important, XX = generally very important. Local methods[A] are procedures developed by an individual laboratory or the instrument manufacturer. NPL software for the calibration of the intensity scales of XPS instruments is available from the UK National Physical Laboratory.[B] BCR 261[C] is a certified reference tantalum oxide/tantalum foil calibration sample and NIST SRM 2135c[D] is a certified nickel/chromium thin-film depth profile standard.

Instrument Calibrations and Checks	ASTM Standard	ISO Standard	Additional Sources	Elemental Composition	Chemical State	Low Level Detection	Quantification	Layer Thickness	Nano-structures
General System Check			Local Method	XX	XX	XX	XX	XX	XX
Sample Preparation	E1829 E1078	18116 18117		X	X	X	X	XX	X
Binding Energy	E2108 E1523	15472 19318		XX	XX		X		X
Intensity Repeatability and Constancy		24237		X	X	XX	XX	X	X
Intensity/Energy Response Function			NPL Software				XX	X	XX
Linearity of Intensity Scale		21270 18118		X	X		XX	X	XX
Peak Intensities	E995	18392 20903	Local Method	X	X	XX	X	X	X
Ion Gun and Sputter Rate	E1577 E1127 E1634	15969 22335 14606 14701	BCR 261					XX	XX
Depth Resolution	E1577 E1127 E1634 E1636	14606	BCR 261 NIST SRM 2135c					XX	XX
Analysis Area	E1217	19319		X			X	X	XX
Lateral Resolution		18516		X			X	X	X
Data Reporting	E996	14979		X	X	X	X	X	X

[A] Manual from the system manufacturer.
[B] National Physical Laboratory (NPL), http://www.npl.co.uk/science-technology/surface-and-nanoanalysis/services/calibration-software-and-reference-materials-for-electron-spectrometers.
[C] European Institute for Reference Materials and Measurements, BCR261, certified reference material.
[D] National Institute of Standards and Technology, NIST-SRM 2135c Ni/Cr Thin Film Depth Profile Standard, http://www.nist.gov.

constancy, intensity/energy response function (IERF), linearity test of the intensity scale, energy resolution for the desired intensity, lateral resolution, charge compensation, depth resolution, and depth profile rate calibration.

5.2.3 *Sample Transport and Preparation*—As a surface analysis technique, X-ray photoelectron spectroscopy (XPS) is sensitive to the outermost few atomic layers of the sample being characterized. Specimens should be transported to the analyst in a container that does not come into direct contact with the surface of interest. In most cases, the analysis will be performed on the as-received specimen; therefore, the goal must be to preserve the state of the surface so that the analysis remains representative of the original surface. Care must then be taken to ensure that no outside agents come in contact with the surface to be investigated. These agents include: fingers, solvents or cleaning solutions, gases (including compressed air) or vapors, metals, tissue or other wrapping materials, tape, cloth, tools, packing materials or the walls of containers. Handling of the surface to be analyzed should be eliminated or minimized whenever possible.

5.2.3.1 Proper preparation and mounting of specimens is particularly critical for surface analysis. Improper preparation of specimens can result in alteration of the surface composition and unreliable data. In addition, specimen mounting techniques have the potential to affect the intended analysis. Guides E1078 and E1829 and/or ISO 18116 and ISO 18117 describe methods the surface analyst may need to minimize the effects of specimen preparation when using any surface-sensitive analytical technique. Also described are methods to mount specimens so as to ensure that the desired information is not compromised. Because of the wide range of types of specimens and desired information, only broad guidelines and general examples are included in the standards. The optimum handling procedures will be dependent on the particular specimen and the needed information. It is recommended that the specimen supplier consult the surface analyst as soon as possible with

regard to specimen history, the specific problem to be solved or information needed, and the particular specimen preparation or handling procedures required.

5.2.4 *Binding Energy, Energy Scale Linearity, and Charge Compensation*—Calibrations of the binding energy (BE) scales of XPS instruments are required for four principal reasons. First, meaningful comparison of BE measurements from two or more XPS instruments requires that the BE scales be calibrated, often with an uncertainty of about 0.1 to 0.2 eV. Second, identification of chemical state is based on measurement of chemical shifts of photoelectron and Auger-electron features, again with an uncertainty of typically about 0.1 to 0.2 eV; individual measurements, therefore, should be made and literature sources need to be available with comparable or better accuracies. Third, the availability of databases of measured BEs for reliable identification of elements and determination of chemical states by computer software requires that published data and local measurements be made with uncertainties of about 0.1 to 0.2 eV. Finally, the growing adoption of quality management systems, such as ISO 9001, in many analytical laboratories has led to requirements that the measuring and test equipment be calibrated and that the relevant measurement uncertainties be known. As described in the ASTM and ISO procedures below, the accuracy of the BE calibration can be determined by the analyst and depends on the specific analysis objectives. If, for example, changes to a BE of 0.1 eV are important, the degree of instrument calibration needs to be more carefully adjusted than if changes of 0.2 eV or greater are adequate. Annex A of ISO 19318 also describes methods of charge control and charge correction.

5.2.4.1 A procedure is given in Practice E2108 and ISO 15472 for calibrating the BE scale of an X-ray photoelectron spectrometer equipped with one or more of the following sources of characteristic Kα X-rays: magnesium (Mg) source, unmonochromated aluminum (Al) source, or monochromated Al source. In a first calibration for particular operating conditions of the instrument, or after the instrument has been modified, measurements are made of the binding energies of specified core levels of copper and gold, and these values are then compared with corresponding reference energies. The linearity of the BE scale is checked at a single point on the scale using a measurement of the position of either a specified core level of silver (monochromated Al source) or a specified Auger-electron transition of copper (Mg source or unmonochromated Al source). Additional checks can be made, if desired, with secondary standards. Procedures are given for determining the uncertainties of BE measurements (at the 95 % confidence level) at various times following a calibration. The analyst can therefore establish tolerance limits at the same level of confidence, based on the instrument stability and the analyst's needs. Then, BE measurements statistically are likely to be made within these limits during specified time intervals following a calibration. The instrument is then adjusted or subsequent BE measurements are corrected. For a routine check of the instrumental calibration, either one or two measurements are made each of the same core levels of copper and gold. In Practice E2108 and ISO 15472, a flow chart is given that summarizes the steps of the calibration procedure; references are given to relevant sections of this standard. An optional procedure is provided for determining the average energy of the X-rays from a monochromated Al X-ray source, using a measured position of a copper Auger peak in order to determine modified Auger parameters.

5.2.4.2 As discussed in Guide E1523 and ISO 19318 and ISO 21270, the emission of photoelectrons following X-ray excitation may result in a buildup of a positive surface charge on insulating specimen surfaces. This positive surface charge changes the surface potential, thereby shifting the measured energies of the photoelectron peaks to higher binding energy. This binding energy shift may reach a nearly steady-state value of between 2 and 5 eV for spectrometers equipped with nonmonochromatic X-ray sources. The surface potential charge and the resulting binding energy shift are generally larger for spectrometers equipped with monochromatic X-ray sources because of the often lower flux of low-energy electrons impinging on the specimen surface. The amount of induced surface charge, its distribution across the specimen surface, and its dependence on experimental conditions are determined by several factors, including specimen composition, homogeneity, magnitude of surface conductivity, total photoionization cross-section, surface topography, spatial distribution of the exciting X-rays, and availability of neutralizing electrons. Methods of charge control include using an electron flood gun, an ultraviolet flood lamp, specimen heating, improving the electrical connection (grounding and enhanced conduction path, isolation from ground, and biasing), a low-energy ion source or a source utilizing combined low energy electrons and low energy inert ions such as argon. Methods of charge correction include adventitious hydrocarbon referencing, gold deposition, implantation with inert gases, internal referencing, and substrate referencing.

5.2.5 *Intensity Energy Response Function (IERF, Transmission Function)*—Because spectrometers of different designs have responses that vary differently with electron energy and settings, it is essential to know the instrument-energy response function (or transmission function) for quantitative analyses. Some instruments have methods included in the software that allows the response function to be tested and appropriate parameters linked to a specific set of data. The National Physical Laboratory (NPL, United Kingdom) has developed software[5] for intensity calibration of cylindrical mirror analyzer (CMA) or hemispherical sector analyzer (HSA) spectrometers. With spectra acquired from copper, silver, and gold reference materials, the software can be used to calculate the intensity response function (or transmission function) of the spectrometer. The calibration gives the transmission function over the entire Al or Mg binding energy range in XPS. The calibrated response function is then available as a digital output data file of values at 1 eV intervals and as an analytical function. Spectra acquired subsequently can be divided by the Intensity/Energy Response Function to obtain spectra which are intercomparable with those from other calibrated

[5] National Physical Laboratory (NPL), http://www.npl.co.uk/science-technology/surface-and-nanoanalysis/services/calibration-software-and-reference-materials-for-electron-spectrometers.

instruments, typically to an accuracy of a few percent. Intensity repeatability and constancy calculations are included in ISO 24237. Using Cu for relatively rapid check measurements enables the intensity repeatability and constancy to be tracked for each mode used. This is essential for consistent quantification and can provide optimum conditions for the detection of an element.

5.2.6 *Intensity Scale Linearity*—Quantitative analysis can be compromised if the count rate for photoelectron peaks exceeds the range of linearity. ISO 21270 allows the linearity of the intensity scale to be defined, relevant corrections to be applied and the maximum counting rate established for a given, chosen, linearity level.

5.2.6.1 ISO 18118 describes the use of experimentally determined relative sensitivity factors for quantitative analysis.

5.2.7 *Peak Intensities*—The determination of peak intensity (usually area) requires removing the background portion of the spectrum from the signal intensity as described in ISO 20903. This can be accomplished by several methods involving differing levels of information related to the physical processes which produce the background signals. The production of electrons from X-ray excitation of surfaces may be grouped into two categories—photoemission of electrons and the production of Auger electrons from the decay of the resultant core-hole states. The source of the background signal observed in the XPS spectrum includes a contribution from inelastic scattering processes and, for non-monochromatic X-ray sources, electrons produced by bremsstrahlung radiation. Various background subtraction techniques have been employed to diminish or remove the influence of these background electrons from the shape and intensity of Auger electron and photoelectron features. Implementation of any of the various background techniques that are described in Guide E995 and ISO 18392 and ISO 20903 may depend on available instrumentation and software as well as the method of acquisition of the original signal.

5.2.7.1 Common background-subtraction techniques include linear background subtraction, integral (or Shirley) background subtraction, inelastic electron-scattering correction (or Tougaard), signal differentiation, and X-ray satellite subtraction.

5.2.8 *Ion Gun and Sputter Rate*—Guides E1577, E1127, and E1634 and ISO 15969, ISO 22335, and ISO 14606 address issues of depth resolution and profile rate calibration. Ion beams are utilized in surface analysis in two ways. First, they can generate signals from the specimen, for example, in SIMS and ISS and occasionally in AES. Second, they can remove material from the specimen surface while a surface analytical technique determines the composition of the freshly exposed surface. This process is called sputter depth profiling. Sputter depth profiling is used in conjunction with X-ray photoelectron spectroscopy to determine the chemical composition and atomic concentration as a function of distance from the original surface of a specimen. Guide E1577 requires reporting all characteristics of the ion beam that can possibly affect the results so that the measurement can be reproduced. Sputter crater depth measurements are performed in order to determine a sputter rate (depth/time) for each matrix sputtered during a sputter depth profile or similar in-depth type analyses. From sputter rate values, a linear depth scale can be calculated and displayed for the sputter depth profile. Data obtained from surface profilometry are useful in monitoring instrumental parameters (for example, raster size, shape, and any irregularities in topography of the sputtered crater) used for depth profiles.

5.2.8.1 Evaluation should be conducted for requirements for depth resolution versus speed of profiling, optimizing settings and calibration of the sputtered area for choice of ion beam energy and angle of incidence for best depth resolution or for fastest profile or a compromise. Other issues involve calibration of alignment and sputtered area for each energy needed and selection of other parameters including sample rotation speed. The diameter of the ion beam used for sputtering must be specified.

5.2.9 *Depth Resolution*—Guides E1577, E1127, and E1634 discuss aspects of depth resolution in sputter depth profiling, along with ISO 14606. XPS yields information concerning the chemical and physical state of a solid surface in the near surface region. The standards listed above evaluate requirements for depth resolution versus speed of profiling, optimization of settings, and calibration of the sputtered area for choice of ion beam energy and angle of incidence for best depth resolution, fastest profile, or a compromise calibration of these two objectives. Techniques for measuring the crater depths and film thicknesses are given in Guide E1634. Ion sputtering is primarily used for depths of less than the order of 1 µm. Angle lapping or mechanical cratering is primarily used for depths greater than the order of 1 µm. The choice of depth profiling methods for investigating an interface depends on surface roughness, interface roughness, and film thickness.

5.2.10 *Analysis Area*—X-ray photoelectron spectroscopy is used extensively for the surface analysis of materials. Determining the specimen area contributing to the detected signal is relevant for experiments for which the signals arising from specific regions of the sample are of importance. If, as an example, the composition of a small defect is of analytical importance, it is useful to know how much signal may arise from other portions of the sample adjacent to the region of interest. The observed specimen area depends on selected conditions of operation (that is, the size of selected slits or apertures) and also can depend on the adequacy of alignment of the specimen with respect to the electron energy analyzer. Practice E1217 and ISO/TR 19319 describe how an electron beam (if available on the XPS instrument) with a selected energy can be scanned across the surface of a test specimen. The beam may be scanned once, that is, in a line scan, or in a pattern, that is, rastered. As the electron beam is deflected across the specimen surface, measurements are made of the intensities detected by the electron energy analyzer as a function of the beam position for selected conditions of analyzer operation. The measured intensities may be due to electrons elastically scattered by the specimen surface, to electrons inelastically scattered by the specimen, or to Auger electrons emitted by the specimen. The intensity distributions for a particular detected electron energy can be plotted as a function of beam position in several ways and can be utilized

to obtain information on the specimen area contributing to the detected signal and on analyzer performance for the particular conditions of operation.

5.2.10.1 In XPS instruments where imaging is available, the analysis area can also be determined after taking images, and then selecting the analysis area from the images.

5.2.11 *Lateral Resolution*—ISO 18516 describes three methods for determining the lateral resolution achievable in Auger electron spectrometers and X-ray photoelectron spectrometers. A straight-edge method is suitable for instruments where the lateral resolution is expected to be larger than 1 µm. A grid method is suitable if the lateral resolution is expected to be less than 1 µm but more than 20 nm. A gold-island method is suitable for instruments where the lateral resolution is expected to be less than 50 nm. The first two methods are likely to be applicable to most commercial XPS instruments. ISO 18516 describes the test samples to be used, how the measurements should be made, and how the results should be analyzed. Examples are given for each method.

5.2.12 *Data Reporting*—Practice E996 and ISO 14976 describe the reporting of all experimental conditions that affect X-ray photoelectron spectra so spectra can be reproduced in other laboratories or be compared with other spectra. Minimum reporting parameters should include (as appropriate) instrument manufacturer and model, excitation source type, energy, strength (power), angle of incidence, analyzer and lens type, nominal energy resolution (as percent for fixed retardation ratio or as eV for fixed analyzer transmission), angle of emission, calibration energies (at least two); sampled area on the specimen, step scan interval, data acquisition time, sputter conditions and sputter rate calibration. The standards include expanded discussion on each of the above parameters.

6. Keywords

6.1 binding energy; calibration; data reporting; design of experiment; linearity; repeatability; resolution; sample preparation; XPS; X-ray photoelectron spectroscopy

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the ASTM website (www.astm.org/COPYRIGHT/).