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Standard Guide for Literature Describing Properties of Electrostatic Electron Spectrometers¹

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1. Scope

1.1 The purpose of this guide is to familiarize the analyst with some of the relevant literature describing the physical properties of modern electrostatic electron spectrometers.

1.2 This guide is intended to apply to electron spectrometers generally used in Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS).

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

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

1.5 *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

E673 Terminology Relating to Surface Analysis (Withdrawn 2012)³

E902 Practice for Checking the Operating Characteristics of X-Ray Photoelectron Spectrometers (Withdrawn 2011)³

E1217 Practice for Determination of the Specimen Area Contributing to the Detected Signal in Auger Electron

Spectrometers and Some X-Ray Photoelectron Spectrometers

E2108 Practice for Calibration of the Electron Binding-Energy Scale of an X-Ray Photoelectron Spectrometer

2.2 *ISO Standards:*⁴

ISO 18516 Surface Chemical Analysis—Auger Electron Spectroscopy and X-Ray Photoelectron Spectroscopy—Determination of Lateral Resolution

ISO 21270 Surface Chemical Analysis—X-Ray Photoelectron and Auger Electron Spectrometers—Linearity of Intensity Scale

ISO 24236 Surface Chemical Analysis—Auger Electron Spectroscopy—Repeatability and Constancy of Intensity Scale

ISO 24237 Surface Chemical Analysis—X-Ray Photoelectron Spectroscopy—Repeatability and Constancy of Intensity Scale

3. Terminology

3.1 For definitions of terms used in this guide, refer to Terminology **E673**.

4. Summary of Guide

4.1 This guide serves as a resource for relevant literature which describes the properties of electron spectrometers commonly used in surface analysis.

5. Significance and Use

5.1 The analyst may use this document to obtain information on the properties of electron spectrometers and instrumental aspects associated with quantitative surface analysis.

6. General Description of Electron Spectrometers

6.1 An electron spectrometer is typically used to measure the energy and angular distributions of electrons emitted from a specimen, typically for energies in the range 0 to 2500 eV. In surface analysis applications, the analyzed electrons are produced from the bombardment of a sample surface with

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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 from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <http://www.iso.org>.

electrons, photons or ions. The entire spectrometer instrument may include one or more of the following: (1) apertures to define the specimen area and emission solid angle for the electrons accepted for analysis; (2) an electrostatic or magnetic lens system, or both; (3) an electrostatic (dispersing) analyzer; and (4) a detector. Methods to check the operating characteristics of X-ray photoelectron spectrometers are reported in Practice E902.

6.2 Intensity Scale Calibration and Spectrometer Transmission Function—Quantitative analysis requires the determination of the ability of the spectrometer to transmit electrons, and the resultant detector signal, throughout the spectrometer instrument. This can be described by an overall electron energy-dependent transmission function $Q(E)$ and is given by the product (1, 2),⁵ as follows:

$$Q(E) = H(E) \cdot T(E) \cdot D(E) \cdot F(E), \quad (1)$$

where:

$H(E)$ = the effect of mechanical imperfections (such as aberrations, fringing fields, etc.),

$T(E)$ = electron-optical transmission function,

$D(E)$ = detector efficiency, and

$F(E)$ = efficiency of the counting systems.

Knowledge of this transmission function permits the calibration of the spectra intensity axis (3). A detailed review of the experimental determination of the transmission function for XPS (4) and AES (5) measurements has been published.

6.3 Energy Scale Calibration—Calibration of the energy scales of AES and XPS instruments is required for (1) meaningful comparison of building-energy or kinetic-energy measurements from two or more instruments; (2) valid identification of chemical state from such comparisons; (3) effective use of databases containing reported energy values; and (4) as a component of a laboratory quality system. Suitable photon energy values for Al and Mg anode X-ray sources often used in XPS measurements are available (6) and reference binding energy values for copper (Cu), gold (Au), and silver (Ag) have been published (7). Reference kinetic-energy values for Cu, aluminium (Al), and Au are also available (8, 9). Binding energy scale calibration procedures have been described in the literature for XPS (10, 11) and kinetic energy scale calibrations for AES (8, 12-14) measurements. Practice E2108 describes a procedure for calibrating the binding energy scale of XPS instruments using Cu, Ag, and Au specimens.

6.4 Linearity of Intensity Scale—See ISO 21270 for methods to evaluate linearity of the intensity scale of AES and XPS spectrometers.

6.5 Repeatability and Constancy of Intensity Scale—See ISO 24236 and ISO 24237 for methods to evaluate the repeatability and constancy of intensity scales of AES and XPS spectrometers, respectively.

6.6 Lateral Resolution—See ISO 18516 for methods to determine the lateral resolution of AES and XPS spectrometers.

6.7 Specimen Area Contributing to the Detect Signal—See Practice E1217 for methods to determine the specimen area contributing to the detected signal in Auger electron spectrometers and some X-Ray photoelectron spectrometers.

6.8 Calibration Protocol—Recommendations have been published describing spectrometer calibration requirements and the frequency with which AES and XPS spectrometers should be calibrated (15).

7. Literature

7.1 Electrostatic Analyzers—Spectrometers commonly used on modern AES and XPS spectrometer instruments generally employ electrostatic deflection analyzers. Auger electron spectrometers often use cylindrical mirror analyzer (CMA) designs, although concentric hemispherical analyzers (CHA) (also known as spherical deflection (or sector) analyzers) are also used. The CHA design is the most common analyzer employed on modern XPS instruments, although double-pass CMA designs were also employed on earlier XPS instruments. Retarding field analyzers (RFA) have historical interest in early AES work, but are now commonly used on low energy electron diffraction apparatus.

7.1.1 Electrostatic Deflection Analyzers—A review of the general properties of deflection analyzers may be found in review articles (16, 17). More detailed reviews are also available where, in addition to the CMA and CHA designs, plane mirror, spherical mirror, cylindrical sector, and toroidal deflection analyzers are treated (18-20). As the width of typical Auger spectral features are several electron volts, the use of a CMA design in conventional AES has sufficed for routine analysis, particularly for small area analysis where a compromise between signal-to-noise and energy resolution is important. These are commonly used at a resolution defined by the full-width at half-maximum of the spectrometer energy resolution, ΔE , divided by the electron energy, E , of 0.25 to 0.6 %. The ability to incorporate an electron source concentric with the CMA axis has been extensively exploited in scanning-electron microscope instruments to give Auger data as a function of beam position (that is, images). However, analysis of the Auger spectra from some compounds and surface morphologies may be enhanced by the use of a CHA design which can provide better energy resolution (but a lower transmission) and superior angular resolution. The CHA design is most frequently employed on XPS instruments where spectral features generally have narrow energy widths of 1 eV or less and higher angular resolution is desired for the detected electrons than is possible with a CMA. The relationship between the pass energy of various spectrometer designs and the potential between their electrodes is described in detail (16).

7.1.2 Retarding Field Analyzers—The use of a retarding field analyzer (RFA), consisting of concentric, spherical-sector grids, is currently used most commonly on electron diffraction instruments where the angular distribution of the detected electrons is examined. See also a brief review of RFA designs (16) and a substantial report on resolution and sensitivity issues (21).

⁵ The boldface numbers in parentheses refer to the list of references at the end of this guide.

7.2 Apertures—The effects of the spectrometer entrance and exit slits and apertures, their associated fringing fields, as well as the effect of the divergence of the incident electron trajectories on analyzer performance, particularly energy resolution, have also been reviewed (16-20). A detailed examination of the effects of unwanted internal scattering in CHA and CMA electron spectrometers has been reported in the literature (22-24).

7.3 Lens Systems—Input lens systems are frequently employed in CHA (and cylindrical sector) designs to vary the surface analysis area (25) and to permit a convenient location of the CHA so as to allow access of complementary surface characterization techniques to the sample (26). The electrostatic lens design often consists of a coaxial series of electrodes that define the analysis area on the sample surface and determines the electron trajectories at the input to the analyzer. The lens system also determines the angular resolution and modifies the transmission characteristics of the spectrometer system (1). Reviews of electrostatic lens systems incorporated in surface analysis instruments have been published (16-20, 27). Lens systems have also been introduced at the exit of analyzers for photoelectron imaging (17, 28-30). Methods to determine the specimen area examined are described in Practice E1217.

7.4 Detectors—Detection of the analyzed electrons is generally accomplished through the use of an electron multiplier to produce usable signals. Surface analysis instruments currently use a variety of multipliers, but most are glass upon which a

resistive coating is placed. The coating is formulated to provide a substantial secondary electron yield upon primary electron impact. The multiplier has a potential placed upon it so that the secondary electrons are accelerated to adjacent coated surfaces, thus providing the electron multiplying effect. Multipliers are available in various shapes for both analog and pulse counting amplification modes of operation (31). Single-channel electron multipliers were common in early instruments, but multiple-channel (“multichannel”) electron multipliers fabricated into thin plates are now available for use in detectors. See a general review of electron multipliers (32-34). The use of position-sensitive detectors, such as resistive anodes, as well as wedge and strip anodes at the output of such electron multipliers, has afforded the ability to also record the spatial (angular) characteristics of the analyzed electrons and has thus permitted the determination of surface composition as a function of position (“chemical maps”) in XPS instruments (20, 33). A delay-line detector has recently been developed for XPS (35). The detection efficiency of single channel multipliers as a function of incident energy, angle of incidence, as well as count rate have been reported (34). In addition, the influence of the detector electronics and counting systems have also been examined (36, 37).

8. Keywords

8.1 apertures; Auger electron spectroscopy; detectors; electron spectrometers; electrostatic analyzers; lens systems; X-ray photoelectron spectroscopy

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