Designation: E996 – 10

Standard Practice for
Reporting Data in Auger Electron Spectroscopy and X-ray Photoelectron Spectroscopy[1]

This standard is issued under the fixed designation E996; 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 Auger and X-ray photoelectron spectra are obtained using a variety of excitation methods, analyzers, signal processing, and digitizing techniques.

1.2 This practice lists the desirable information that shall be reported to fully describe the experimental conditions, specimen conditions, data recording procedures, and data transformation processes.

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 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]

E673 Terminology Relating to Surface Analysis (Withdrawn 2012)[3]
E902 Practice for Checking the Operating Characteristics of X-Ray Photoelectron Spectrometers (Withdrawn 2011)[3]
E983 Guide for Minimizing Unwanted Electron Beam Effects in Auger Electron Spectroscopy
E995 Guide for Background Subtraction Techniques 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

3. Terminology

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

4. Summary of Practice

4.1 Report all experimental conditions that affect Auger and X-ray photoelectron spectra so spectra can be reproduced in other laboratories or be compared with other spectra.

5. Significance and Use

5.1 Include the experimental conditions under which spectra are taken in the "Experiment" section of all reports and publications.

5.2 Identify any parameters that are changed between different spectra in the "Experiment" section of publications and reports, and include the specific parameters applicable to each spectrum in the figure caption.

6. Information To Be Reported

6.1 *Equipment Used:*

6.1.1 If a commercial electron spectroscopy system is used, specify the manufacturer and model. Indicate the type of electron excitation source and electron analyzer as well as the model designation of other equipment used for generating the experimental data, such as a sputter ion source.

6.1.2 If a spectrometer system has been assembled from several components specify the manufacturers and model numbers of excitation source, analyzer, and auxiliary equipment.

6.1.3 Identify the model name, version number, and manufacturer of software packages used to acquire or process the data.

6.2 *Specimen Analyzed:*

6.2.1 Describe the specimen as completely as possible, for example, its bulk composition, history, any methods of cleaning or sectioning, pre-analysis treatments, and dimensions.

6.2.2 Describe the method of mounting and positioning the specimen for analysis, for example, mounted on a carousel, or mounted between strips of a particular metal. If the specimen

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[1] This practice 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.

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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.

is heated, cooled or treated in the spectrometer system, describe the method used (for example, heated by electron bombardment on the back of the specimen, or resistively heated). See Guide E1078 for more detail.

6.2.3 State the operating pressure of the vacuum system during data acquisition and the position of the vacuum gage relative to the specimen being analyzed. State if the system was backfilled with a sputter gas. Indicate the presence of active gases if they are appropriate to the measurement. If the system (and specimen) was baked-out before analysis, the time, temperature and final pressure should also be stated.

6.3 *Parameters Used for Analysis:*

6.3.1 *Excitation Source*—For electron beam excitation, state the beam energy, beam size, incident current, whether the beam is stationary or scanned (if scanned, state the area), and angle of incidence. State the method used to determine the electron beam diameter. (See Note 1.) For radiation-sensitive specimens, give the pre-analysis and analysis beam exposure times. See Guide E983 to minimize unwanted electron beam effects. For X-ray excitation, specify the anode material, characteristic radiation energy, beam size at the specimen, whether the beam is stationary or scanned (if scanned, state the area), source strength, electron emission current, acceleration voltage, window material, and whether the source X-ray was monochromatic.

NOTE 1—The common method of measuring incident electron beam current by applying a low (approximately + 100 volt) specimen bias does not account for emission of backscattered electrons. The preferred method is to use a Faraday cup bearing a small entrance aperture to limit the number of electrons escaping.

6.3.2 *Charge Correction*—For insulating specimens, it is often necessary to correct for the charging of the specimen under irradiation. When energies of lines from such specimens are quoted, the method of charge correction must also be described as well as the standard value assumed. If an electron beam or ion beam is used, its beam current, energy, and diameter or current density should also be given.

6.3.3 *Analyzer*—State the type of analyzer (and lens) used for electron collection (cylindrical mirror (single or double-pass), hemispherical, spherical, and the like). State the spectrometer's energy resolution, retardation ratio, pass energy (if pertinent), emission angle, source-to-analyzer angle, acceptance angle width, and specimen acceptance area. Describe how any of these analyzer properties vary with electron energy.

6.3.4 *Modulation*—If phase-sensitive detection is used to obtain the Auger spectrum in derivative form the peak-to-peak energy modulation should be stated. If electron beam modulation is used, the electron beam chopping frequency and duty cycle should be stated.

6.3.5 *Time Constant*—Give the system time constant if analog detection is used. The limiting time constant could be determined by that of the phase-sensitive detector, ratemeter, recorder, or digitizing system.

6.3.6 *Scan Rate*—If an analog scan is used, give the sweep rate in eV/s (electronvolt/second). If a stepped scan is used, give the step size in eV and the dwell time per step.

6.3.7 *Energy Scale Calibration*—The method for calibration of the binding energy scale shall be specified. It is recommended that the procedure described in Practice E902 be used to ensure that the spectrometer is operating in a reproducible manner.

6.3.8 *Detector Description*—Describe the detector used. If an electron multiplier is used and the front is biased, state the bias voltage. Indicate whether the output of the analyzer is measured directly, or by a voltage isolation method, by pulse counting, or by voltage-to-frequency conversion. For a multichannel detector, give the number of channels in the spectrum covered by the width of the detector.

6.3.9 *Signal Averaging*—If the spectrum is signal averaged, state the number of scans.

6.3.10 *Sputtering*—If ion sputtering was used for cleaning or sputter depth profiling, describe the ion species, ion energy, energy filtering, neutral rejection (if employed), the beam current, diameter, or maximum current density, and angle of incidence. If ion beam scanning is used, state the area and rate. State the total pressure in the vicinity of the specimen (if known) and if the sputtering source was differentially pumped. If a depth scale is given on a sputter depth profile, state the method of depth calibration. If the sputter rate is not known, it is recommended that relative sputter rates be determined using a known thickness of tantalum pentoxide or silicon dioxide. State the specimen rotation rate if rotational depth profiling was used.

6.4 *Data Handling:*

6.4.1 *Data Processing*—Describe any smoothing, differentiation, background subtraction (see Guide E995), deconvolutions, curve resolution, intensity scale correction, satellite subtraction, or other processing of the data. Specify any assumptions and approximations required for the processing, together with the data reduction algorithm. In the case of multiple processing methods, the step-by-step effect of each method should be explained.

6.4.2 *Quantification*—If the elemental concentrations or surface coverages are calculated from the data, indicate the method, type of software, and version, along with the values and source of any parameters, for example, relative sensitivity factors, elemental region end points used for peak area, or intensity determination, and instrument transmission function correction coefficients. State the signal-to-noise ratio, precision, and minimum detection limits of the data.

6.4.3 *Peak Energies*—Auger electron and photoelectron peak positions are normally reported as the energy of maximum intensity in the *N(E)*-type spectrum. For derivative Auger spectra, the maximum negative excursion in the *dN/dE*-type spectrum is reported. When peak energies are reported, also report the peak energies of any calibration materials used to check the spectrometer performance. When line energies are cited more precisely than 0.1 eV, describe the method used to determine the peak energy. For all data, give an estimate of the precision of the measurement.

6.5 *Display of Data:*

6.5.1 *Auger and XPS Spectra*—The horizontal electron energy scale shall be marked in eV. Mark the vertical axis as N(E) if the electron energy distribution is measured, or dN/dE if the first derivative is measured. With certain types of analyzers, other electron energy distributions are measured and these should be given, for example, with a single-pass cylindrical mirror analyzer E·N(E), or dE·N(E)/dE, are usually measured. The units used for the vertical axis can be "arbitrary units." If pulse counting is used, report the units as "counts" or (preferred) "counts per second."

6.5.2 *Sputter Depth Profiles*—The signal intensity (in arbitrary units) or the atomic percent concentration are given on the vertical axis. If signal intensity is used, label the axis "peak height" or "peak area" as applicable, or in the case of derivative spectra "peak-to-peak height." Label the horizontal axis "depth," if this is known, otherwise use "sputter time." Report sputtering conditions as in 6.3.10. More detail on depth profiling is provided in Guide E1127.

6.5.3 *Line Scans*—The vertical axis of the data should be labeled similarly to that for sputter depth profiles, in 6.5.2. Note the kinetic energy used for making the measurement. State if one energy is used, or if intensity is calculated as $P - B$ (or a linear background intensity interpolated between two background values, or some other means). Note if the effects of electron current drift and specimen topography have been minimized by plotting such functions as:

$$(P - B)/B \text{ or } (P - B)/(P + B) \tag{1}$$

where:

P = a measure of the signal intensity, and
B = the background intensity at an energy offset from the peak.[4]

Label the horizontal axis "position" with the appropriate units in micrometres, millimetres, etc.

6.5.4 *Maps*—Describe the Auger or XPS signal used for obtaining a map of an element or chemical state (see 6.5.3). Mark the magnification scale on the map by including a dimension marker (µm or nm). Indicate the type of signal (see 6.5.1) used for determining the brightness of the map. Also, describe and display the intensity scale (dot intensity, gray levels, or false colors) used to produce the map. Indicate if topography correction was used (6.5.3) or other mathematical processing techniques, such as smoothing. If digital images are being presented, indicate the number of picture elements (pixels) being used in the horizontal and vertical direction. Also indicate the mapping time, beam current, and number of intensity levels. Also indicate if thresholding or non-linear processing has been applied.

7. Abbreviated Reporting of Data

7.1 For some publications and reports, space does not allow for the full reporting of all information necessary to describe the measurement and data. While the analyst needs to have the full measurement description available, reporting the following minimum parameters may satisfy many requirements:

7.1.1 Instrument manufacturer and model:

7.1.2 Excitation source type, energy, strength, and angle of incidence,

7.1.3 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 (if any);

7.1.4 Sampling area on the specimen, and

7.1.5 Step scan interval, data acquisition time, and modulation amplitude (for phase-sensitive detection).

8. Keywords

8.1 Auger electron spectroscopy; surface analysis ; X-ray photoelectron spectroscopy

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[4] Prutton, M., Larson, L. A., and Poppa, H., *Journal of Applied Physics*, Vol 54, 1983, p. 374.