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Standard Guide for Background Subtraction Techniques in Auger Electron Spectroscopy and X-Ray Photoelectron Spectroscopy[1]

This standard is issued under the fixed designation E995; 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 The purpose of this guide is to familiarize the analyst with the principal background subtraction techniques presently in use together with the nature of their application to data acquisition and manipulation.

1.2 This guide is intended to apply to background subtraction in electron, X-ray, and ion-excited Auger electron spectroscopy (AES), and X-ray photoelectron spectroscopy (XPS).

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E673 Terminology Relating to Surface Analysis (Withdrawn 2012)[3]

2.2 *ISO Standard:*[4]

ISO 18115–1 Surface chemical analysis—Vocabulary—Part 1: General terms and terms used in spectroscopy

3. Terminology

3.1 *Definitions*—Since Terminology E673 was withdrawn in 2012, for definitions of terms used in this guide, refer to ISO 18115-1.[5]

4. Summary of Guide

4.1 *Relevance to AES and XPS:*

4.1.1 *AES*—The production of Auger electrons by bombardment of surfaces with electron beams is also accompanied by emission of secondary and backscattered electrons. These secondary and backscattered electrons create a background signal. This background signal covers the complete energy spectrum and has a maximum (near 10 eV for true secondaries), and a second maximum for elastically backscattered electrons at the energy of the incident electron beam. An additional source of background is associated with Auger electrons, which are inelastically scattered while traveling through the specimen. Auger electron excitation may also occur by X-ray and ion bombardment of surfaces.

4.1.2 *XPS*—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.

4.2 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. Relevance to a particular analytical technique (AES or XPS) will be indicated in the title of the procedure.

4.3 Implementation of any of the various background subtraction techniques that are described in this guide may depend on available instrumentation and software as well as the

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

[5] https://www.iso.org/obp/ui/#iso:std:iso:18115:-1:ed-2:v1:en.

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method of acquisition of the original signal. These subtraction methods fall into two general categories: (*1*) real-time background subtraction; and (*2*) post-acquisition background subtraction.

5. Significance and Use

5.1 Background subtraction techniques in AES were originally employed as a method of enhancement of the relatively weak Auger signals to distinguish them from the slowly varying background of secondary and backscattered electrons. Interest in obtaining useful information from the Auger peak line shape, concern for greater quantitative accuracy from Auger spectra, and improvements in data gathering techniques, have led to the development of various background subtraction techniques.

5.2 Similarly, the use of background subtraction techniques in XPS has evolved mainly from the interest in the determination of chemical states (from the binding-energy values for component peaks that may often overlap), greater quantitative accuracy from the XPS spectra, and improvements in data acquisition. Post-acquisition background subtraction is normally applied to XPS data.

5.3 The procedures outlined in Section 7 are popular in XPS and AES; less popular procedures and rarely used procedures are described in Sections 8 and 9, respectively. General reviews of background subtraction methods and curve-fitting techniques have been published elsewhere (**1-5**).[6]

5.4 Background subtraction is commonly performed prior to peak fitting, although it can be assessed (fitted) during peak fitting (*active approach* (**6, 7**)). Some commercial data analysis packages require background removal before peak fitting. Nevertheless, a measured spectral region consisting of one or more peaks and background intensities due to inelastic scattering, Bremsstrahlung (for XPS with unmonochromated X-ray sources), and scattered primary electrons (for AES) can often be satisfactorily represented by applying peak functions for each component with parameters for each one determined in a single least-squares fit. The choice of the background to be removed, if required or desired, before or during peak fitting is suggested by the experience of the analysts, the capabilities of the peak fitting software, and the peak complexity as noted above.

6. Apparatus

6.1 Most AES and XPS instruments either already use, or may be modified to use, one or more of the techniques that are described.

6.2 Background subtraction techniques typically require a digital acquisition and digital data handling capability. In earlier years, the attachment of analog instrumentation to existing equipment was usually required.

7. Common Procedures

7.1 The following background subtraction methods are widely employed. It is common for an analyst to choose one among them depending on the shape of the spectrum. As shown in a Round Robin study, different groups chose different background methods for analyzing the same spectrum (**8**). Although the purpose of this guide is to describe the common procedures employed for background subtraction, 7.3.2 provides a short guide of how to choose one or more background types depending on the shape of the spectrum.

7.2 *Commonly Employed Background Types:*

7.2.1 *Linear Background (AES and XPS)*—In this method, two arbitrarily chosen points in the spectrum are selected and joined by a straight line (**1 and 2**). This straight line is used to approximate the true background and is subtracted from the original spectrum. For Auger spectra, the two points may be chosen either on the high-energy side of the Auger peak to result in an extrapolated linear background or such that the peak is positioned between the two points. For XPS spectra, the two points are generally chosen such that the peak is positioned between the two points. The intensity values at the chosen points may be the values at those energies or the average over a defined number of data points or energy interval. The linear method can be extended to a polynomial version when the peaks are small and riding on top of a more complex (than linear) background (**7**).

7.2.2 *Shirley (or Integral) Background (AES and XPS)*—This method, proposed by Shirley (**9**), employs a mathematical algorithm to approximate the step in the background commonly found at the position of the peak. The algorithm is based on the assumption that the background is proportional to the area of the peak above the background at higher kinetic energy. This implies an iterative procedure, which was described in detail by Proctor and Sherwood (**10**), that should be employed to guarantee self-consistency (**11**). With another variant proposed by Vegh (**12**) and fully discussed by Salvi and Castle (**13**), it is possible to employ a self-consistent Shirley-type background (*SVSC-background*) without the need of an iterative process; it is especially practical for complex spectra (**7**).

7.2.2.1 The original Shirley method was modified by Bishop to include a sloping component to reproduce the decay of the background intensity (**14**). Another modification provides for a background based upon the shape of the loss spectrum from an elastically backscattered electron (**15**), and to include a band gap for insulators (**1**).

7.2.3 *2-Parameter and 3-Parameter Tougaard Backgrounds (XPS)*—This corresponds to a practical version of the approach described in 8.1. Under this method, the λK function, which enters in the algorithm, is taken from a simple universal formula which is approximately valid for some solids. Similar functions have been optimized for particular materials or material classes (**16**). The application of this background might require the acquisition of background data in a 50 to 100 eV range below (in the lower kinetic-energy side) the main peaks. Alternatively, the parameters used in the universal formula may also be permitted to vary in an optimizing algorithm so as to produce an estimate of the background (**1 and 17**). Tougaard has assessed the accuracy of structural parameters and the amount of substance derived from the analysis (**18**). A more approximate form of the Tougaard algorithm (**19**) can be used for automatic processing of XPS spectra (for example, spectra

[6] The boldface numbers in parentheses refer to the references at the end of this standard.

acquired for individual pixels of an XPS image). A simpler form of the Tougaard background, the slope-background (**20**), can be employed for spectra with a reduced (5 to 15 eV) background acquisition range below the main peaks. It is designed to reproduce the onset of the background growth due to extrinsic inelastic electron scattering, which correspond to the near-peak part of the Tougaard background (it cannot be employed to reproduce the background signal farther than ~ 15 eV from the main peaks).

7.3 *Implementation of the Various Background Subtraction Methods (XPS):*

7.3.1 *Background End-Points (XPS)*—A key choice in implementation of the methods described in 7.2 is the selection of the two end points or spectral region for background subtraction. These points are selected far enough from the peaks to assure that the intensity at those energies is only due to the background.

7.3.1.1 However, in some cases, one peak might still contribute to the signal at the chosen points, so the total intensity is not purely due to the background. This is common for spectra containing peaks with large kurtosis (large Lorentzian width) since the peak contribution at energies as far as five times the Lorentzian width from the peak center is still 1 %. In these cases it is possible to employ an *active approach* during peak fitting in which the intensity of the background is not tied to the intensity of the signal at the chosen points but calculated during peak-fitting (**6, 7**). The advantages of an active approach are discussed in various reports (**12, 13**); an early example can be found in Figure A3.7 of Ref (**21**).

7.3.2 *Choosing the Background Type Based on the Shape of the Spectrum (XPS)*—The linear background is recommended when the background at both sides of the peaks is a straight line, one side the continuation of the other. The polynomial background is recommended for small peaks riding on top of the background of a larger peak or on wide Auger structures. A step-shaped increment on the background intensity from the low to the high binding energy side of the main features could be treated with the (iterative) Shirley or with the SVSC method. Besides the plasmon features, the Tougaard-type backgrounds also reproduce an increment on the slope of the background signal near the peak on the high binding energy side.

7.3.2.1 The high binding-energy side of a photoelectron peak commonly shows both a step-shaped increment and an increment on the slope of the background signal. In these and other cases, the total background might consist of the sum of various types. The simultaneous application of various background types can be done under the active approach (**7**). Some examples are discussed in References (**7 and 20**).

7.4 *Signal Differentiation, dN(E)/dE or dEN(E)/dE (AES)* (**22 and 23**)—Signal differentiation is among the earliest methods employed to remove the background from an Auger spectrum and to enhance the Auger features. It may be employed in real time or in post-acquisition. In real time, differentiation is usually accomplished by superposition of a small (1 to 6 eV peak-to-peak) sinusoidal modulation on the analyzer used to obtain the Auger spectrum. The output signal is then processed by a lock-in amplifier and displayed as the derivative of the original energy distribution *N(E)* or *EN(E)*. In post-acquisition background subtraction, the already acquired *N(E)* or *EN(E)* signal may be mathematically differentiated by digital or other methods. The digital method commonly used is that of the cubic/quadratic derivative as proposed by Savitzky and Golay (**24**).

7.5 *X-Ray Satellite Subtraction (for Non-Monochromated X-Ray Sources) (XPS)* (**25**)—In this method, photoelectron intensity from the satellite X-rays associated with the K X-ray spectrum from an aluminum or magnesium X-ray source is subtracted. Intensity is removed from higher kinetic energy channels at the spacing of the $K\alpha_{3,4}$, $K\beta$, etc. satellite positions from the $K\alpha_{1,2}$ main peak and with the corresponding intensity ratios (**25**) to remove their contributions to the XPS spectrum. This subtraction can proceed through the spectrum but not if there is an Auger peak in the region of interest because it would erroneously remove an equivalent intensity from any Auger peaks present in the spectrum.

7.6 *Reporting*—For consistent determination of a peak area, the region over which background subtraction needs to be applied will vary with the peak width, peak shape, and the background-subtraction method applied. The consistent application of a background-subtraction process can produce precise determination of peak areas. In many circumstances, electrons appropriately associated with the photoelectron peaks can occur outside of the integration limits; therefore the accuracy of any resulting quantification will depend on the method by which the sensitivity factors were determined. Analytical errors can also occur if there are changes in AES or XPS lineshapes or shakeup fractions with changes of chemical state. Uncertainties in X-ray photoelectron spectroscopy intensities associated with different methods and procedures for background subtraction have been evaluated for both monochromatic aluminum X-rays (**8**) and for unmonochromated aluminum and magnesium X-rays (**26**). Since the peak area will depend on the chosen background and how it is applied, the analyst should specify the background type or types and the chosen end points when reporting peak areas and the derived analytical results.

8. Less Common Procedures

8.1 *Inelastic Electron Scattering Correction (AES and XPS)*—This method, proposed by Tougaard (**27**), uses an algorithm which is based on a description of the inelastic scattering processes as the electrons travel within the specimen before leaving it. The energy loss function (or scattering cross section) multiplied by the inelastic mean free path (the λK function) is iteratively convolved with the primary signal to reproduce the background in a large energy region. This background subtraction method also gives direct information on the in-depth concentration profile (**28 and 29**). The λK function could be assessed from reflected electron energy loss spectroscopy (REELS) measurements by applying a certain algorithm (**1, 30 and 31**).

8.2 *Deconvolution (AES and XPS)* (**32-35**)—Deconvolution may be used to reduce the effects due to inelastic scattering of electrons traveling through the specimen. This background is removed by deconvoluting the spectrum with elastically backscattered electrons (set at the energy of the main peak) and its

associated loss spectrum. The intensity of the loss spectrum, relative to that of the backscattered primary, is sometimes adjusted to optimize the background subtraction. Deconvolution is usually accomplished using Fourier transforms or iterative techniques.

8.3 *Linearized Secondary Electron Cascades (AES)*—In this method, proposed by Sickafus (**36 and 37**) the logarithm of the electron energy distribution is plotted as a function of the logarithm of the electron energy. Such plots consist of linear segments corresponding to either surface or subsurface sources of Auger electrons and are appropriate for removing the background formed by the low energy cascade electrons.

9. Rarely Used Procedures

9.1 *Secondary Electron Analog (AES)* (**38 and 39**)—In this method, a signal that is an electronic analog of the secondary electron cascade is combined with the analyzer signal output so as to counteract the secondary emission function. It is particularly useful for retarding field analyzers in which low-energy secondary emission is prominent.

9.2 *Dynamic Background Subtraction (DBS) (AES)* (**40 and 41**)—Dynamic background subtraction may be used either in real time or post acquisition. It involves multiple differentiation of an Auger spectrum to effect background removal, followed by an appropriate number of integrations to re-establish a background-free Auger spectrum. The amount of background removal depends on the number of derivatives taken, although two are usually sufficient. In real-time analysis, a first derivative of the Auger electron energy distribution obtained using a phase-sensitive detector is fed into an analog integrator, thereby obtaining the Auger electron energy distribution with the background removed.

9.3 *Tailored Modulation Techniques (TMT) (AES)* (**42 and 43**)—This is a real-time method of background subtraction that uses special modulation waveforms tailored to the analyzer and phase sensitive detection to measure the Auger signal. The *N(E)* distribution, *EN(E)* distribution, or areas under Auger peaks over specified energy ranges may be obtained directly using these techniques.

9.4 *Spline Technique (AES and XPS)* (**44**)—In this method, a structureless background is calculated from a measured spectrum using a smoothing spline algorithm. This background is then subtracted from the original spectrum.

9.5 *Digital Filtration (AES)* (**45 and 46**)—In a method borrowed from energy-dispersive X-ray spectroscopy, a "top-hat" digital frequency filter is applied to an Auger spectrum to suppress the slowly varying background continuum, while the more rapidly varying Auger peaks remain unaffected.

10. Keywords

10.1 Auger electron spectroscopy; background subtraction; surface analysis; X-ray photoelectron spectroscopy

APPENDIX

(Nonmandatory Information)

X1. COMPARISONS AVAILABLE IN THE LITERATURE

X1.1 At the present time, the most popular background subtraction method for AES is digital differentiation (see 7.4). Common methods for XPS include the straight line (see 7.2.1), Shirley-type (see 7.2.2), or variations of the Tougaard method (see 7.2.3). Comparisons of background subtraction methods mentioned here have been published in the literature. In the case of 7.2.1, 7.2.2, and 7.2.3, the effect on the peak area calculated in terms of the choice of end points is examined in 7.3.1, (**10 and 14, 8 and 26**). Further comparisons of these procedures and those in 7.3 on a number of materials are also offered (**8 and 26, 47-57**).

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