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# Standard Practice for Identifying Elements by the Peaks in Auger Electron Spectroscopy[1]

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## 1. Scope

1.1 This practice outlines the necessary steps for the identification of elements in a given Auger spectrum obtained using conventional electron spectrometers. Spectra displayed as either the electron energy distribution (direct spectrum) or the first derivative of the electron energy distribution are considered.

1.2 This practice applies to Auger spectra generated by electron or X-ray bombardment of the specimen surface and can be extended to spectra generated by other methods such as ion bombardment.

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E673 Terminology Relating to Surface Analysis (Withdrawn 2012)[3]

E983 Guide for Minimizing Unwanted Electron Beam Effects in Auger Electron Spectroscopy

E984 Guide for Identifying Chemical Effects and Matrix Effects in Auger Electron Spectroscopy

E1523 Guide to Charge Control and Charge Referencing Techniques in X-Ray Photoelectron Spectroscopy

2.2 *ISO Standards:*[4]

ISO 17973: 2002 Surface Chemical Analysis—Medium-Resolution Auger Electron Spectrometers—Calibration of Energy Scales for Elemental Analysis

ISO 17974: 2002 Surface Chemical Analysis—High-Resolution Auger Electron Spectrometers—Calibration of Energy Scales for Elemental and Chemical-State Analysis

## 3. Terminology

3.1 *Definitions:*

3.1.1 Terms used in Auger electron spectroscopy are defined in Terminology E673.

## 4. Summary of Practice

4.1 The Auger spectrum is obtained with appropriate instrumental parameters from a low kinetic energy limit of approximately 30 eV to an upper kinetic energy limit of approximately 2000 to 3000 eV or higher to include all the principal Auger electron energies of all elements (except hydrogen and helium which do not have Auger transitions).

4.2 This practice assumes the existence of appropriate reference spectra from pure element or stoichiometric compound standards, or both, with which an unknown spectrum can be compared (**1, 2**).[5] It may be useful to note that although Auger energies in some data bases are referenced to the Fermi level, other data collections have been referenced to the vacuum level. Auger kinetic energies referenced to the Fermi level would be approximately 5 eV larger than values referenced to the vacuum level.

4.3 An element in an Auger spectrum is considered positively identified if the peak shapes, the peak energies, and the relative signal strengths of peaks from the unknown coincide with those from a standard reference spectrum of the element or compound.

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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.

[4] 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.

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

## 5. Significance and Use

5.1 Auger analysis is used to determine the elemental composition of the first several atomic layers, typically 1 to 5 nm thick, of a specimen surface. In conjunction with inert gas ion sputtering, it is used to determine the sputter depth profile to a depth of a few micrometres.

5.2 The specimen is normally a solid conductor, semiconductor, or insulator. For insulators, provisions may be required for control of charge accumulation at the surface (see Guide E1523). Typical applications include the analysis of surface contaminants, thin film deposits or segregated overlayers on metallic or alloy substrates. The specimen topography may vary from a smooth, polished specimen to a rough fracture surface.

5.3 Auger analysis of specimens with volatile species that evaporate in the ultra-high vacuum environment of the Auger chamber and substances which are susceptible to electron or X-ray beam damage, such as organic compounds, may require special techniques not covered herein. (See Guide E983.)

## 6. Apparatus

6.1 *Electron Energy Analyzers:* a retarding field analyzer, cylindrical mirror analyzer (single or double pass), or hemispherical analyzer is typically used. The calibration of the analyzer's energy scale should be checked at regular intervals (see ISO 17973: 2002 and ISO 17974: 2002) to aid in making reliable identifications.

6.2 *Standard Equipment:* typically an electron gun or X-ray source is used for excitation, an electron multiplier is used for amplification of the Auger electron signal, and a recording device is used to output the data.

6.2.1 A vacuum capability in the test chamber is required for operation of the electron gun or X-ray source and the spectrometer, and to allow analysis without contamination from the ambient gases; depending on specimen surface conditions, analysis is performed in the pressure range from $10^{-3}$ to $10^{-8}$ Pa.

## 7. Procedure

7.1 Identify the peak having the largest signal strength in the spectrum and note its peak energy and characteristic shape. Note that by convention, the peak energy is measured at the energy of the maximum intensity in the direct N(E) spectrum and at the minimum value in the derivative spectrum. These two energies will not be the same, with the derivative spectrum giving higher peak energies.

7.2 Consult a list of peak energies for elements and note the possible energy matches. The peak position can vary by up to 20 eV because of slightly differing chemistries, so include elements within a wide range of energies around the peak position noted in 7.1.

7.3 Consult the standard elemental spectrum for one of the elements identified by 7.2, and look for the presence of additional lines in the specimen spectrum that match the standard spectrum in energy and intensity (see peak overlap in 8.1.1) (**1, 2**). Direct and derivative standard spectra are available (**1, 2**). Compare the shape of the peaks as well. If a good match is found, label all lines from the standard spectrum that are visible in the specimen spectrum. If a match is not found, eliminate that element from further consideration and select another element from the list found in 7.2 and repeat 7.3.

7.4 If all of the elements from 7.2 have been exhausted, widen the energy range and choose additional elements. Remember, charging may shift the energy spectrum substantially. In this event, look for the carbon and oxygen peak shapes (or other known elements) by shape and relative position to determine the extent of charging. Correct for charging and repeat 7.2 and 7.3.

7.5 If a match is still not found, temporarily ignore the most intense peak and repeat 7.1-7.3 for the next most intense peak. Recall that relative Auger peak intensities may change because of the specimen chemistry. A weaker peak may become more intense and the primary peak may become less intense. (See Guide E984.)

7.6 Repeat 7.1-7.3 with peaks of decreasing signal strength until all peaks are positively identified.

7.7 It should be noted that since the Auger signal strength varies proportionally with the concentration of the element detected, an element present at a small concentration may register only its strongest Auger peak(s). The identification of such weak peaks should be verified by optimizing the signal-to-noise ratio in separate scans, for example, by repetitive scans of the energy range of interest.

7.8 When extending the technique to Auger transitions generated by an X-ray source, it is important to note that the kinetic energy of the Auger electrons does not change when the energy of the incident X-rays is changed. This may allow the Auger peaks to be distinguished from the photoelectron peaks.

7.9 Auger features generated by incident ions may have relative intensities and energies that differ substantially from those generated by electrons or X-rays (**3**).

## 8. Interferences

8.1 The procedure for positive elemental identification given in Section 7 is valid except when the characteristic shape of an Auger peak is subject to change (which is not caused by instrumental parameters such as different analyzer resolutions). This can arise from two situations:

8.1.1 Peak overlap occurs when Auger peak energies from two or more different elements coincide within the energy width ranges of the peaks. The spectrum of a possible component element can be subtracted from the composite data. This subtraction or spectrum stripping can be performed numerically, and the residual intensity can be compared with reference spectra for another possible element (**4**). Sometimes, for qualitative analysis, spectrum stripping can be adequately performed by an experienced eye. It may be necessary to take additional data on an expanded scale (perhaps with increased energy resolution and improved signal-to-noise ratio) to perform the spectrum stripping with the desired precision.

8.1.2 Changes in peak energy, peak shape, or relative signal strengths of peaks may be due to Auger chemical effects or matrix effects. (See Guide E984.) In this case, the reference spectrum should be that of the particular compound containing the element.

8.2 In spectra obtained using X-ray generation there will generally be photoelectron peaks in the spectrum. These may interfere with the Auger peaks. As noted above, if possible, changing the energy of the incident X-rays will change the kinetic energy of the photoelectron peaks, while the kinetic energy of the Auger peaks will be unchanged.

## 9. Discussion

9.1 The state-of-the-art in Auger electron spectroscopy now allows routine qualitative analysis without too many interpretational difficulties. It is assumed that the practitioner employs common practice in generating an Auger spectrum in order to use this procedure. Under normal circumstances, all elements (except hydrogen and helium) present on a specimen surface being analyzed can be detected with a sensitivity limit of 1 atomic % or better.

9.2 *Electron Beam Excitation*—Typical parameters used for electron beam excitation are 2 to 30 keV beam energy and $10^{-10}$ to $10^{-6}$ A beam current. The beam energy and beam current are selected to obtain sufficient Auger signal strength and an appropriate beam size and analysis depth. High electron beam current densities may cause electron beam damage in certain specimens. (See Guide E983.) If phase-sensitive detection is used for obtaining the Auger spectrum, typical modulations used are 2 to 6 $eV_{pp}$ sinusoidal or square-wave at 5 to 10 kHz.

9.3 *X-Ray Excitation*—Typical parameters used in the source for X-ray excitation are 5 to 50 mA electron emission at 10 to 15 keV energy, resulting in a power dissipation of 100 to 1000 W.

9.4 Because this practice for chemical identification relies on comparison with reference spectra, the unknown spectrum should ideally be generated using the same spectrometer type and instrumental parameters employed for the reference spectra. Practical situations do not always meet these ideal conditions; therefore, systematic spectrum differences should be taken into consideration when comparing with the standard spectra, such as the electron background shape which changes with differing spectrometer type or primary electron beam energy.

9.5 Peaks other than those arising from interferences referred to in Section 8 can arise from ionization loss peaks (electron excitation), photoelectron peaks (X-ray excitation and electron excitation) (**5**), ion-excited Auger peaks, or charging effects.

9.5.1 Ionization loss peaks from primary electrons may appear in the Auger spectrum, particularly when the primary beam energy is relatively low. Changing the primary beam energy shifts the loss peak energy by the same amount and loss peaks may thus be identified. It is usually recommended that the primary beam energy should be at least three times greater than the Auger energies of interest to minimize the occurrence of these loss peaks.

9.5.2 Photoelectron peaks from characteristic X-rays (for example, Mg $K_\alpha$) will occur in the spectrum. Changing the characteristic X-rays (for example, from Mg $K_\alpha$ to Al $K_\alpha$) will move the photoelectron peaks relative to the Auger spectrum.

9.5.3 Auger transitions can also be excited in certain materials by ion beams typically used in sputter depth profiling (for example, $Ar^+$, 1–5 keV). Such materials include magnesium, aluminum, and silicon. Their ion excited Auger signal strengths increase with beam energy.

9.5.4 Charging effects are manifested by shifts of the entire spectrum and are particularly prevalent during the analysis of insulating surfaces. Severe charging may introduce spurious peaks. Differential charging on the specimen may cause multiple spectral shifts. Unusually sharp peaks or peaks whose energy position shifts in subsequent spectra are usually indicative of charging. Instrumental adjustments are necessary to eliminate this problem. Care should be used in Auger peak identification since such effects change the Auger peak energy and shape.

## 10. Keywords

10.1 AES; Auger electron spectroscopy; spectroscopy; surface analysis

## REFERENCES

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