
**Surface chemical analysis — Auger
electron spectroscopy — Reporting of
methods used for charge control and
charge correction**

*Analyse chimique des surfaces — Spectroscopie des électrons
Auger — Indication des méthodes mises en œuvre pour le contrôle et la
correction de la charge*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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Introduction

Auger electron spectroscopy (AES) is widely used for characterization of surfaces of materials. Elements in the sample (with the exception of hydrogen and helium) are identified from comparisons of Auger transition energies, determined from measured Auger spectra, with tabulations of these energies for the various elements. Although Auger electrons are observed during X-ray irradiation of specimens (X-ray photoelectron spectroscopy), AES, as used in this document, is associated with electron irradiation of a specimen. Because the incident electron beam can be focused to sizes approximating 10 nm, AES is an important tool for characterization of small surface features and of nanostructured materials. Information on the elements present, and sometimes the chemical state of the detected elements, can frequently be obtained from examination of the line shape and energies of the peaks (see ISO/TR 18394^[43]). Reliable determination of elements present requires appropriate calibration of the energy scale (as described in ISO 17973 and ISO 17974).

The surface potential of an insulating specimen may change during an AES measurement due to the build-up of surface and near-surface electrical charge, and this charge can shift the energy of Auger electrons, thus complicating elemental (and chemical state) identification, especially when a negative surface potential moves the Auger spectrum above the energy interval selected by the electron analyser. The build-up of surface potential can also move the location of the electron beam, effectively shifting the region on the specimen or even off the specimen that is being analysed. Similar changes occur for metals during electron irradiation if they are not connected to ground. This would occur, for example, if small metal particles are incorporated in an insulating matrix. Depending on the secondary-electron yield, the surface potential may shift positively or negatively. In some circumstances, these two shifts (energy and position) create an unstable feedback system, rendering the collection of AES spectra nearly impossible. In addition to changes in the Auger-electron peak energy and intensity, the specimen surface composition might be altered (specimen damage) directly by the incident electron beam or due to electric-field-induced diffusion when a field is set up in the surface region of the specimen. A variety of methods and approaches have been developed to control and minimize charging effects in AES. The application of a particular method can be highly dependent on the details of the instrument being used, the size and shape of the specimen being examined, the specimen morphology and composition, and the information to be collected. Although the build-up of surface charge can complicate analysis, in some circumstances it can also be used creatively as a tool to gain information about the specimen.

The amount of induced charge near the surface, its distribution across the specimen surface, and its dependence on experimental conditions are determined by many factors, including those associated with the specimen and the characteristics of the spectrometer. Charge build-up is a well-studied^[1] three-dimensional phenomenon that occurs along the specimen surface and into the material. Charge build-up may also occur at phase boundaries or interface regions within the depth of a specimen that is irradiated by electrons. Some specimens undergo time-dependent changes in charge build-up due to charge trapping, chemical changes or component diffusion or volatilization induced by heating or by incident or secondary electrons. Such specimens may never achieve steady-state potentials.

There is, at present, no universally applicable method or set of methods for charge control or for charge correction in AES^{[2],[3]}. This International Standard specifies the information that has to be provided to document the method of charge control during data acquisition and/or the method of charge correction during data analysis of insulating specimens. Information is given in Annex A on common methods for charge control that can be useful for many applications. The particular charge-control method that may be chosen in practice depends on the type of specimen (e.g. powder, thin film or thick specimen), the nature of the instrumentation, the size of the specimen and the extent to which the specimen surface might be modified by a particular procedure. To assist an analyst, a summary table lists the common charge-control methods in approximate order of simplicity of application.

This International Standard has two main areas of application. First, it identifies information on methods of charge control and/or charge correction to be included in reports of AES measurements (e.g. from an analyst to a customer or in publications) in order to evaluate and reproduce data on insulating materials and to ensure that measurements on similar materials can be meaningfully compared. Second, adherence to the International Standard will enable published AES spectra to be used with confidence by other analysts.

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Surface chemical analysis — Auger electron spectroscopy — Reporting of methods used for charge control and charge correction

1 Scope

This International Standard specifies the minimum amount of information required for describing the methods of charge control in measurements of Auger electron transitions from insulating specimens by electron-stimulated Auger electron spectroscopy and to be reported with the analytical results. Information is provided in Annex A on methods that have been found useful for charge control prior to or during AES analysis. This annex also contains a table summarizing the methods or approaches, ordered by simplicity of approach. Some methods will be applicable to most instruments, others require special hardware, others might involve remounting the specimen or changing it. A similar International Standard has been published for X-ray photoelectron spectroscopy (ISO 19318^[44]).

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 17973, *Surface chemical analysis — Medium-resolution Auger electron spectrometers — Calibration of energy scales for elemental analysis*

ISO 17974, *Surface chemical analysis — High-resolution Auger electron spectrometers — Calibration of energy scales for elemental and chemical-state analysis*

ISO 18115, *Surface chemical analysis — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115 apply.

4 Symbols and abbreviated terms

AES Auger electron spectroscopy

E_p primary-electron energy, in keV

$E_{p(max)}$ energy at which the TSEEY is a maximum

E_{p1}^0 energy at which the secondary-electron emission yield rises above unity

E_{p2}^0 energy at which the secondary-electron emission yield drops below unity

E_{p2}^c	energy at which the range of the incident electrons is approximately equal to the maximum escape depth of the secondary electrons
FIB	focused ion beam
FWHM	full width at half maximum, in eV
I_p	primary-electron current
I_s	secondary-electron current
j_p	current density of the primary-electron beam on the specimen surface
KE_{corr}	corrected kinetic energy, in eV
KE_{meas}	measured kinetic energy, in eV
KE_{ref}	reference kinetic energy, in eV
N	charging index
R	range of primary electrons
SEM	scanning electron microscopy
t	electron irradiation time
TSEY	total secondary-electron emission yield
U_s	surface potential
V_e	electron interaction volume
z	specimen thickness
ρ	electrical resistivity of the specimen
σ	total secondary-electron yield
θ	angle of incidence of primary-electron beam on the specimen with respect to the surface normal, in degrees
Δ_{corr}	correction energy, to be added to measured Auger electron energies for charge correction, in eV

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5 Apparatus

5.1 Charge-control technique

One or more of the charge-control techniques described in Clause A.3 may be employed in most AES spectrometers. The AES instrument shall be operated in accordance with the manufacturer's or other documented procedures.

5.2 Special apparatus

Some of the techniques outlined in Clause A.3 require special apparatus, such as a low-energy ion source or a source for evaporative deposition of gold. Some of the referenced items may be the subject of patent rights for specific vendors. Mention of them here is for convenience and does not represent an endorsement by ISO or a member body.

5.3 Specimen mounting and preparation

Certain specimen-mounting procedures, such as mounting the specimen under a fine metal mesh^{[3],[4]}, can enhance electrical contact of the specimen with the specimen holder or reduce the amount of surface charge build-up. This and other methods of specimen mounting to reduce static charge are described in detail in ISO 18116^[5] and ISO 18117^[42] (and in ASTM E1078^[4] and ASTM E1829^[6]).

5.4 Instrument calibration

The kinetic-energy scale of the Auger electron spectrometer shall be calibrated using ISO 17973 or ISO 17974 or another documented method before using this International Standard.

6 Reporting of information related to charge control

6.1 Methods of charge control

Many of the methods commonly used to control the surface potential and to minimize surface charging are described in Clause A.3. Information on reasons for charge control, choice of a charge-control method, and critical specimen and experimental conditions, as described in 6.2, 6.3 and 6.4, shall be reported (or referenced) for individual specimens or collections of similar specimens.

6.2 Reasons for needing charge control and choice of method

The reasons for needing charge control and for choosing a particular method shall be reported.

EXAMPLE 1 The specimen was an insulating film deposited on a conducting substrate. By using an electron primary-beam energy above 10 keV, no specimen charging was observed.

EXAMPLE 2 Experience with similar specimens indicated that charging was likely. To minimize charging, the specimens were mounted under a conducting aperture and beam energies below 3 keV were used. The current was adjusted until the AES spectra obtained were reproducible and stable.

EXAMPLE 3 Spectra recorded initially without any charge control showed peak shifting and broadening. Placing a grounded fine-mesh grid above the specimen minimized these problems. Repeated analyses showed that changes in specimen composition due to charge build-up were below 10 % if the total electron dose was below 1 000 C/m².

If the components used for charge control are not standard for the AES instrument, information on the manufacturer or on the relevant design characteristics shall be provided or referenced.

NOTE A specimen does not need to be a good conductor for routine AES analysis to be accomplished without charging problems. Although it is important to be aware of potential charging issues, experimental verification that they are present is useful before great effort is spent minimizing possible difficulties.

6.3 Specimen information

6.3.1 Specimen form

The form of the specimen shall be reported. The physical nature, source, preparation method and structure of a specimen can influence its charging behaviour^{[1],[2],[3]}.

EXAMPLE 1 Powder.

EXAMPLE 2 Thin film spin-cast on silicon.

EXAMPLE 3 Macroscopic mineral specimen.

6.3.2 Specimen dimensions

The size, shape and surface roughness of a specimen can have a significant effect on the extent of specimen charging. The shape of the specimen shall be reported, together with approximate values of the dimensions of the specimen or of any relevant specimen features (e.g. particle diameters, surface roughness).

6.3.3 Specimen-mounting methods

Specimen mounting and contact with the specimen holder can significantly impact charging^[1]. The method by which a specimen is mounted, including information about special methods used to increase conductivity or isolate a specimen from ground, shall be reported.

EXAMPLE 1 Powder specimen pressed into foil, which was attached to the specimen holder by conducting tape.

EXAMPLE 2 1 ml of solution containing nanoparticles was deposited on a silicon substrate and dried prior to analysis.

EXAMPLE 3 Specimen held to holder using conductive adhesive tape (with manufacturer and type of tape specified).

EXAMPLE 4 Corroded specimen held on specimen holder by metal screw.

EXAMPLE 5 Mineral specimen and conducting aperture mounted using metal screw.

EXAMPLE 6 Specimen mounted with the primary-electron beam at glancing incidence on the specimen surface.

6.3.4 Specimen treatment prior to or during analysis

Any specimen treatment prior to or during analysis, including any physical or chemical treatment that could affect charging of the specimen during AES measurements, shall be reported.

NOTE Such treatment of the specimen can modify the surface composition as well as the electrical conductivity, and hence charging, of the surface region.

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6.4 Values of experimental parameters

Values of parameters used for AES measurements and charge control, including beam parameters (energy, nominal incident current, beam size, raster area on the specimen, angle of incidence on the specimen), irradiation time of the specimen during set-up and AES measurements, and operating parameters of ancillary components such as a low-energy ion gun shall be recorded (or referenced).

EXAMPLE A focused electron beam with energy of 10 keV and 1 nA current at 45° incidence to the specimen normal, rastered over an area of 200 nm by 200 nm, was used for the analysis.

6.5 Information on the effectiveness of methods of charge control

The adequacy of the charge-control method(s) used for the type of analysis being conducted shall be established. Auger line peak positions (or peak widths) with and without a method of charge control provide one way of determining the adequacy of the charge-control method. In some cases, the ability to determine that the AES lines are within 5 eV of the expected value and that the relative peak amplitudes are stable might be satisfactory.

EXAMPLE 1 The AES peak positions were within 5 eV of the nominal values and the peak shapes were both similar to reference data and stable on repeated scans.

EXAMPLE 2 Repeated measurements in new areas demonstrated consistent agreement regarding peak shape and relative intensity.

It is recommended that specimens be examined for the presence or absence of specimen damage and that the results be recorded.

7 Reporting of method(s) used for charge correction and the value of that correction

7.1 Methods of charge correction

In many cases, the methods used to control charge eliminate the need for further correction of the Auger peak energies. If a method of charge correction is applied to AES data, the following critical specimen and experimental parameters shall be reported.

7.2 Approach

Any method for correcting the measured kinetic energy of the Auger peaks for charging effects shall be specified in sufficient detail so that the method can be reproduced and its effectiveness judged.

7.3 Value of correction energy

Information shall be given on the magnitude of the correction energy Δ_{corr} for each spectrum and how this correction energy was determined. The corrected kinetic energies and the values of the reference energies shall be reported.

The correction energy Δ_{corr} is determined by taking the difference between the measured kinetic energy of a reference line KE_{meas} and the accepted or reference value for this kinetic energy KE_{ref} , using the following relation:

$$\Delta_{\text{corr}} = KE_{\text{ref}} - KE_{\text{meas}} \quad (1)$$

The corrected kinetic energy for another Auger peak in the same spectrum KE_{corr} can then be found from the sum of the measured kinetic energy for that peak KE_{meas} and the correction energy:

$$KE_{\text{corr}} = KE_{\text{meas}} + \Delta_{\text{corr}} \quad (2)$$

NOTE Equations (1) and (2) apply only when charge compensation has adequately removed differential charging effects.

Annex A (informative)

Description of methods of charge control for Auger electron spectroscopy

A.1 Introduction

In this annex, methods used to minimize or control changes in surface potential during electron-stimulated Auger electron spectroscopy are briefly described. The methods and approaches are dependent on the types of instrumentation available and the types of specimen being examined. To assist analysts in dealing with charging in AES, Table A.1 lists approaches in the approximate order that they might be attempted. In any laboratory, the precise order will depend on the facilities available and experience in the particular application. Short descriptions and references to the approaches in Table A.1 are provided in Clause A.3. Specific issues that might arise for non-uniform specimens and during sputter profiling, and some approaches for dealing with them, are discussed in Clause A.4. A short background to general physical considerations of charge build-up during AES analysis is provided in Clause A.5, which outlines a conceptual framework for the issues that arise for different types of specimen. It must be noted that, although specimen charging can be a complication to analysis, it can be used to obtain information about the properties of a specimen or regions of a specimen.

A major strength of AES is the high spatial resolution (in three dimensions) available for chemical analysis. However, along with the generation of Auger electron peaks, the electron beam used for AES can alter the electrical potential of the specimen by introducing charge at the surface and in the near-surface region of insulating materials and might initiate several processes that can change or damage the specimen. The amount and distribution of surface and near-surface charge for a specific experimental system (instrument and specimen) are determined by many factors, including the electron-beam energy, current density and angle of incidence of the beam on the specimen, specimen composition, specimen homogeneity, surface contamination, magnitude of bulk and surface conductivities, surface topography, vacuum environment and availability of neutralizing low-energy electrons or ions. Charge build-up occurs along the specimen surface and into the material^[1]. The presence of particles on, or of different phases in, the specimen surface may result in an uneven distribution of charge across the surface, a phenomenon known as differential charging. Charge build-up can also occur at phase boundaries or interface regions within the specimen. Many insulating specimens undergo time-dependent changes in the amount of charging because of charge build-up within the material or because of chemical and physical changes induced by primary or secondary electrons (including electron-stimulated desorption^{[2],[3]}, electron-induced sputtering^[7] and electron-induced adsorption) or specimen heating.

Many different factors, including the specimen resistivity, specimen thickness and mounting approach used, contribute to changes in the surface potential. However, it is useful to know, as pointed out by Hofmann^[3], that a specimen does not need to be a particularly good conductor to enable collection of AES data without observable charging. As one threshold, when using an electron current of approximately 1 μA , an effective resistance of $\sim 1\text{ M}\Omega$ from the specimen surface to ground is not likely to produce observable charging. Using the examples defined by Hofmann, charging might appear in AES for specimens with resistivity values between $10^{-3}\ \Omega\cdot\text{m}$ and $10^{+3}\ \Omega\cdot\text{m}$ (or $10^5\ \mu\Omega\cdot\text{cm}$ and $10^{11}\ \mu\Omega\cdot\text{cm}$). As used in Table A.1, a specimen is considered to be an insulator for AES analysis when it has a resistivity of $\sim 5\ \Omega\cdot\text{m}$ or higher. High-quality insulators have resistivity values of $> 10^5\ \Omega\cdot\text{m}$ [the resistivity of SiO_2 is approximately $10^{14}\ \Omega\cdot\text{m}$ in contrast to the resistivity of Cu of about $10^{-8}\ \Omega\cdot\text{m}$], but the actual resistivity values of importance for AES depend on specimen, instrumental and operational details.

There is no single method to overcome all charging problems during AES analysis^{[2],[3]}, and in some circumstances charging difficulties might be impossible to avoid. Due to the development of new tools and the need to characterize nanostructured materials, several new approaches for dealing with charging during AES started appearing in the literature in the late 1990s and early 2000s. These include specimen preparation methods and increased use of low-energy ions to assist charge compensation. Whereas major advances in