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Surface chemical analysis — Auger electron spectroscopy and X-ray photoelectron spectroscopy — Guide to the use of experimentally determined relative sensitivity factors for the quantitative analysis of homogeneous materials

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National foreword

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INTERNATIONAL STANDARD

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Surface chemical analysis — Auger electron spectroscopy and X-ray photoelectron spectroscopy — Guide to the use of experimentally determined relative sensitivity factors for the quantitative analysis of homogeneous materials

Analyse chimique des surfaces — Spectroscopie des électrons Auger et spectroscopie de photoélectrons — Lignes directrices pour l'utilisation de facteurs expérimentaux de sensibilité relative pour l'analyse quantitative de matériaux homogènes

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

The committee responsible for this document is ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 7, *Electron spectroscopies*.

This second edition cancels and replaces the first edition (ISO 18118:2004), which has been technically revised.

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Introduction

Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) are surface-analytical techniques that are sensitive to the composition in the surface region of a material to depths of, typically, a few nanometres (nm). Both techniques yield a surface-weighted signal, averaged over the analysis volume. Most samples have compositional variations, both laterally and with depth, and quantification is often performed with approximate methods since it can be difficult to determine the magnitude of any compositional variations and the distance scale over which they might occur. The simplest sample for analysis is one that is homogeneous. Although this situation occurs infrequently, it is often assumed, for simplicity in the analysis, that the sample material of interest is homogeneous. This International Standard provides guidance on the measurement and use of experimentally determined relative sensitivity factors for the quantitative analysis of homogeneous materials by AES and XPS.

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INTERNATIONAL STANDARD ISO 18118:2015(E)

Surface chemical analysis — Auger electron spectroscopy and X-ray photoelectron spectroscopy — Guide to the use of experimentally determined relative sensitivity factors for the quantitative analysis of homogeneous materials

1 Scope

This International Standard gives guidance on the measurement and use of experimentally determined relative sensitivity factors for the quantitative analysis of homogeneous materials by Auger electron spectroscopy and X-ray photoelectron spectroscopy.

2 Normative references

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

ISO 18115, *Surface chemical analysis — Vocabulary*

ISO 21270, *Surface chemical analysis — X-ray photoelectron and Auger electron spectrometers — Linearity of intensity scale*

3 Terms and definitions

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

3.1
absolute elemental sensitivity factor
coefficient for an element by which the measured intensity for that element is divided to yield the atomic concentration or atomic fraction of the element present in the sample

Note 1 to entry: The choice of use of atomic concentration or atomic fraction should be made clear.

Note 2 to entry: The type of sensitivity factor used should be appropriate for the equations used in the quantification process and for the type of sample analysed, for example, of homogeneous samples or segregated layers.

Note 3 to entry: The source of the sensitivity factors should be given in order that the correct matrix factors or other parameters have been used.

Note 4 to entry: Sensitivity factors depend on parameters of the excitation source, the spectrometer, and the orientation of the sample to these parts of the instrument. Sensitivity factors also depend on the matrix being analysed and in SIMS, this has a dominating influence.

[SOURCE: ISO 18115:2013, modified]

3.2
relative elemental sensitivity factor
coefficient proportional to the *absolute elemental sensitivity factor* (3.1), where the constant of proportionality is chosen such that the value for a selected element and transition is unity

Note 1 to entry: Elements and transitions commonly used are C 1s or F 1s for XPS and Ag $M_{4,5}VV$ for AES.

Note 2 to entry: The type of sensitivity factor used should be appropriate for the analysis, for example, of homogeneous samples or segregated layers.

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Note 3 to entry: The source of the sensitivity factors should be given in order that the correct matrix factors or other parameters have been used.

Note 4 to entry: Sensitivity factors depend on parameters of the excitation source, the spectrometer, and the orientation of the sample to these parts of the instrument. Sensitivity factors also depend on the matrix being analysed and in SIMS, this has a dominating influence.

[SOURCE: ISO 18115:2013]

3.3
average matrix relative sensitivity factor
coefficient proportional to the intensity calculated for a pure element in an average matrix with which the measured intensity for that element is divided in calculations to yield the atomic concentration or atomic fraction of the element present in the sample

Note 1 to entry: The choice of use of atomic concentration or atomic fraction should be made clear.

Note 2 to entry: The type of sensitivity factor used should be appropriate for the equations used in the quantification process and for the type of sample analysed, for example, of homogeneous samples or segregated layers.

Note 3 to entry: The source of the sensitivity factors should be given. Matrix factors are taken to be unity for average matrix relative sensitivity factors.

Note 4 to entry: Sensitivity factors depend on parameters of the excitation source, the spectrometer, and the orientation of the sample to these parts of the instrument.

[SOURCE: ISO 18115:2013, modified]

3.4
pure-element relative sensitivity factor
coefficient proportional to the intensity measured for a pure sample of an element with which the measured intensity for that element is divided in calculations to yield the atomic concentration or atomic fraction of the element present in the sample

Note 1 to entry: The choice of use of atomic concentration or atomic fraction should be made clear.

Note 2 to entry: The type of sensitivity factor used should be appropriate for the equations used in the quantification process and for the type of sample analysed, for example, of homogeneous samples or segregated layers.

Note 3 to entry: The source of the sensitivity factors should be given in order that the correct matrix factors or other parameters have been used. Matrix factors are significant and should be used with pure-element relative sensitivity factors.

Note 4 to entry: Sensitivity factors depend on parameters of the excitation source, the spectrometer, and the orientation of the sample to these parts of the instrument.

[SOURCE: ISO 18115:2013, modified]

4 Symbols and abbreviated terms

AES Auger electron spectroscopy

AMRSF Average matrix relative sensitivity factor

ARSF Atomic relative sensitivity factor

ERSF Elemental relative sensitivity factor

IERF Intensity-energy response function

S_i^{At} Atomic relative sensitivity factor for element i

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S_i^{Av}	Average matrix relative sensitivity factor for element *i*
S_i^{E}	Elemental relative sensitivity factor for element *i*
RSF	Relative sensitivity factor
XPS	X-ray photoelectron spectroscopy

5 General information

It is convenient in many quantitative applications of AES and XPS to utilize relative sensitivity factors (RSFs) for quantitative analyses. Three types of RSF have been used for this purpose: elemental relative sensitivity factors (ERSFs), atomic relative sensitivity factors (ARSFs), and average matrix relative sensitivity factors (AMRSFs). Formulae defining these three types of RSF are given in A.3 and the principles on which these formulae are based on are given in A.2.

While the ERSFs are the simplest and easiest to apply, they are the least accurate because no account is taken of matrix correction factors (as described in A.3). The matrix correction factors for AES can vary between 0,1 and 8[1] while they can vary between 0,3 and 3[2] for XPS. The ARSFs are more accurate than ERSFs in that they take account of differences in atomic densities, generally the largest single matrix correction. The AMRSFs are the most reliable RSFs in that there is almost complete correction of matrix effects. It is recommended that ERSFs be used only for semi-quantitative analyses (that is, rough estimates of composition) and that ARSFs or preferably, AMRSFs be used for quantitative analyses. For the latter applications, ARSFs shall be used only in situations for which it is not possible to make use of AMRSFs (for example, measurements involving Auger electrons or photoelectrons at energies for which inelastic mean free paths cannot be reliably determined).

In analytical applications of AES and XPS, it is essential that Auger-electron and photoelectron intensities be measured using exactly the same procedure as that used for measurement of the RSFs. For some applications of AES (e.g. sputter depth profiles), it is convenient to use peak-to-peak heights of Auger-electron signals in the differential mode as measures of Auger-electron intensities. For other applications of AES (e.g. scanning Auger microscopy), the Auger-electron intensity can be determined from the difference between the intensity at a peak maximum in the direct spectrum and the intensity of a nearby background signal. Finally, for many applications in XPS and for some applications of AES, areas of peaks in direct spectra are used as measures of photoelectron or Auger-electron intensities.

Relative sensitivity factors depend on the parameters of the excitation source (for example, the incident electron energy in AES and the choice of X-ray energy in XPS), the spectrometer configuration (for example, the angle of incidence of the electron beam in AES, the angle between the X-ray source and the analyser axis in XPS, the sample area viewed by the analyser, and the acceptance solid angle of the analyser), and the orientation of the sample to these parts of the instrument.[3] The sample area viewed by the analyser and the analyser acceptance solid angle can depend on analyser settings (for example, selection of apertures, whether the analyser is operated in the constant analyser energy mode or the constant retardation ratio mode, and the corresponding choices of analyser pass energy or retardation ratio). Finally, the measured Auger-electron or photoelectron intensities can depend on the instrumental parameters described in Clause 6. Therefore, it is essential that Auger-electron and photoelectron intensities be determined using exactly the same instrumental settings and the same sample orientation as those employed for the ERSF measurements. It is also essential that the same data-analysis procedures (described in Clause 7) be used in measurements of signal-electron intensities for the unknown sample as those used in the ERSF measurements.

Commercial AES and XPS instruments are generally supplied with a set of ERSFs for one or more common operating conditions. These ERSFs were typically determined on an instrument of the same type or, in some cases, on similar instruments. It is recommended that an analyst check the ERSFs supplied with the instrument for those elements expected to be of analytical interest to ensure that the supplied ERSFs are correct. In addition, the intensity-energy response function (IERF) of the instrument can change with time, as described in Clause 8. Such changes can be detected and corrective actions be taken using

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calibration software available from the UK National Physical Laboratory.[4] Alternatively, an analyst can check for possible changes in IERF with time by measuring selected ERSFs as described in Clause 8.

6 Measurement conditions

6.1 General

The same measurement conditions (for example, instrumental configuration, sample orientation, and instrumental settings) shall be used for the measurement with the unknown sample as those chosen for the ERSF measurements. Particular attention shall be given to the following parameters.

6.2 Excitation source

The incident-electron energy in AES and the X-ray source in XPS shall be the same for the measurement of the unknown sample as that chosen for the measurement of the ERSFs.

6.3 Energy resolution

Unless peak areas are used to measure the signal intensities, the energy resolution of the electron-energy analyser (that is determined by choice of aperture sizes, pass energy, or retardation ratio) shall be the same for the unknown-sample measurement as for the measurement used to generate the ERSFs.[5]

6.4 Energy step and scan rate

The size of the energy step (energy per channel) used to acquire spectral data and the spectral scan rate shall be chosen so that there is negligible spectral distortion in the acquired data for the selected energy resolution.

6.5 Signal intensity

The incident-electron current (in AES) or the X-ray intensity (in XPS) shall be adjusted together with the voltage applied to the detector so that the measured signal intensity is proportional to the incident current or X-ray intensity to within 1 % as described in ISO 21270. Alternatively, the measured signal intensity that is corrected for counting losses as described in ISO 21270 shall be proportional to the incident current or X-ray intensity to within 1 %.

6.6 Gain and time constant (for AES instruments with analogue detection systems)

The settings of the detector system shall be the same in the unknown-sample measurement as in the measurement used to generate the ERSFs. The time constant[6] in the measurements shall be sufficiently short so that shapes of spectral features are not significantly distorted during data acquisition. The gain of the detector system shall be adjusted so that the intensities measured for the relevant peaks are within the range for linear detector response.

Procedures to check for linear detector response in pulse-counting systems are described in ISO 21270. The first method described there may be used for analogue AES systems if there are sufficient instrumental controls.

6.7 Modulation to generate a derivative spectrum

It is often convenient in AES to utilize the differential spectrum. The derivative spectrum can be acquired by applying a modulation energy to the analyser[7][8] or by numerical processing of a measured direct spectrum.[9][10] For this purpose, a modulation or numerical differential of between 2 eV and 10 eV

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