
**Surface chemical analysis — Auger
electron spectroscopy and X-ray
photoelectron spectroscopy —
Methods used to determine peak
intensities and information required
when reporting results**

*Analyse chimique des surfaces — Spectroscopie des électrons Auger
et spectroscopie de photoélectrons par rayons X — Méthodes utilisées
pour la détermination de l'intensité des pics et informations requises
pour l'expression des résultats*

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Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Symbols and abbreviated terms	1
5 Methods for peak-intensity determination — direct spectrum	2
5.1 General	2
5.2 Selection and subtraction of an inelastic background	3
5.3 Measurement of peak intensity	4
5.3.1 Measurement of peak height	4
5.3.2 Measurement of peak area	4
5.4 Measurement of a peak intensity with computer software	5
5.5 Measurement of peak intensities for a spectrum with overlapping peaks	5
5.6 Uncertainty in measurement of peak area	6
6 Methods for peak intensity determination — Auger-electron differential spectrum	7
6.1 General	7
6.2 Measurement of Auger-electron differential intensity	7
6.3 Uncertainties in measurement of Auger-electron differential intensity	8
7 Reporting of methods used to measure peak intensities	10
7.1 General requirements	10
7.2 Methods used to determine peak intensities in direct spectra	10
7.2.1 Intensity measurement for a single peak, as described in 5.2 and 5.3	10
7.2.2 Intensity measurements from peak fitting, as described in 5.4 and 5.5	11
7.3 Methods used to obtain and determine peak intensities in Auger-electron differential spectra	11
7.3.1 Method used to obtain differential spectra	11
7.3.2 Method used to determine peak intensities, as described in 6.2	11
Annex A (informative) Instrumental effects on measured intensities	12
Annex B (informative) Useful integration limits for determination of peak intensities in XPS spectra	13
Bibliography	15

Foreword

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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).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 7, *Electron spectroscopies*.

This third edition cancels and replaces the second edition (ISO 20903:2011), which has been technically revised. The main changes compared to the previous edition are as follows:

- subclause 6.3 has been replaced to include modern methods for dealing with co-existing chemical states;
- minor editorial changes have been introduced for clarity.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

An important feature of Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) is the ability to obtain a quantitative analysis of the surface region (≈ 1 nm to 10 nm) of a solid sample. Such an analysis requires the determination of the intensities of spectral components.

There are several methods of peak-intensity measurement that are applicable to AES and XPS. In practice, the choice of method will depend upon the type of sample being analysed, the capabilities of the instrumentation used, and the methods of data acquisition and treatment available.

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Surface chemical analysis — Auger electron spectroscopy and X-ray photoelectron spectroscopy — Methods used to determine peak intensities and information required when reporting results

1 Scope

This document specifies the necessary information required in a report of analytical results based on measurements of the intensities of peaks in Auger electron and X-ray photoelectron spectra. Information on methods for the measurement of peak intensities and on uncertainties of derived peak areas is also provided.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 18115-1, *Surface chemical analysis — Vocabulary — Part 1: General terms and terms used in spectroscopy*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115-1 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

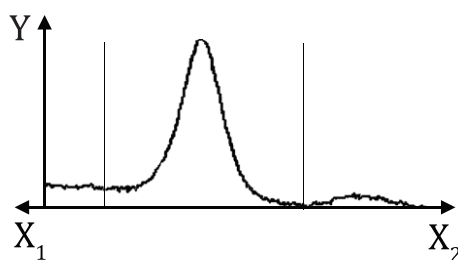
4 Symbols and abbreviated terms

A	peak area
AES	Auger electron spectroscopy
b	number of channels over which intensities are averaged to obtain a baseline
eV	electron volts
n	number of channels in a spectrum
XPS	X-ray photoelectron spectroscopy
y_i	number of counts in the i th channel of a spectrum
ΔE	channel width (in electron volts)
Δt	dwell time per channel (in seconds)
$\sigma(A)$	standard deviation of calculated peak area

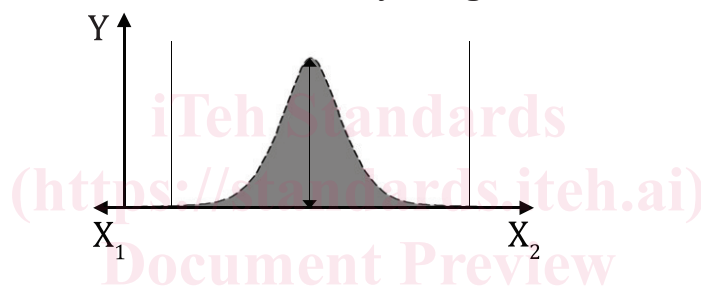
5 Methods for peak-intensity determination — direct spectrum

5.1 General

Figure 1 a) shows a portion of an X-ray photoelectron spectrum in which intensity is plotted as a function of kinetic energy increasing to the right or of binding energy increasing to the left. The intensity is plotted usually in units of counts or sometimes in units of counts per second. Intensities may also be plotted as a digitized voltage; this procedure is often used when the intensity of an Auger differential spectrum is obtained from an analogue detection system. Energies are commonly expressed in electron volts.



a) XPS peak whose intensity is to be measured, the vertical lines indicate suitable limits for the construction of a Shirley background



b) XPS peak shown in a) following the subtraction of the inelastic background (the shaded area indicates the peak area to be measured)

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 X_1 binding energy (eV)
 X_2 kinetic energy (eV)
 Y intensity

Figure 1 — Illustration of procedure involved in the determination of the intensity of a single peak in an X-ray photoelectron spectrum (as described in 5.2 and 5.3).

The intensity of a single peak in an X-ray photoelectron spectrum can be measured by using the procedure described in 5.2 and 5.3 or by using computer software as described in 5.4. The measurement of peak intensities for a spectrum containing overlapping peaks is described in 5.5. Information on the uncertainty of a measured peak area for a single peak is given in 5.6.

The intensity of a single peak in a direct Auger-electron spectrum can be measured by following the procedure described in 5.2 and 5.3, although it may be necessary first to subtract a secondary-electron background[1][2]. Alternatively, computer software can be used to measure the peak intensity as described in 5.4.

In some cases, the peak of interest may be superimposed on a sloping background. This background could arise from multiple inelastic scattering of Auger electrons or photoelectrons of initially high energy, from multiple inelastic scattering of primary electrons (in AES), or from photoemission by bremsstrahlung radiation (for XPS with an unmonochromated X-ray source). It may be necessary (e.g. with use of the Tougaard inelastic background described in 5.2) or desirable to subtract this background from the spectrum in the vicinity of the peak before proceeding with the peak-intensity measurements described in 5.2 to 5.5. This subtraction can usually be performed by fitting a straight line to the sloping

background at energies between about 10 eV and 30 eV above the peak of interest, extrapolating this line to lower energies, and subtracting the spectral intensities from this linear background. If a linear function is judged to be invalid for describing the sloping background over the spectral range of interest (e.g. for modelling the background of scattered primary electrons in AES), an exponential function can be utilized[3].

5.2 Selection and subtraction of an inelastic background

An appropriate inelastic background shall be selected and subtracted from the measured spectrum. Three types of inelastic background are in common use:

- a) linear background;
- b) integral or Shirley background[4];
- c) Tougaard background[5][6][7] and Werner background[8][9], based on physical models describing inelastic electron scattering in solids.

Information on procedures and software for determining the Shirley, Tougaard and Werner backgrounds is given in the scientific literature[4]–[13] and ISO/TR 18392[14].

From a practical viewpoint, the selection of a particular background will depend on (a) whether the relevant software is conveniently available and (b) the type of sample analysed. For insulators, the linear background is often satisfactory, while the Shirley background is often employed for metals. While these two backgrounds are simple and convenient to apply, the limits of these two backgrounds (the starting and ending points on the energy scale) should be chosen carefully so that the background is as nearly continuous as possible with the spectrum in the region of overlap.

Tougaard's approach, in particular, for background determination and subtraction[5][6][7] has found favour over the Shirley background because it describes the physics of the inelastic-scattering process more accurately[15][16]. The Tougaard and the Werner approaches have a further advantage in that they are insensitive to the precise positions of the starting and ending energy points providing they are clearly in the spectral region well away from the main peak of interest (typically starting at an energy at least 10 eV higher than that of the peak of interest and ending at an energy at least 50 eV lower). This requirement is a disadvantage in that spectra have to be recorded over a larger energy range than if the linear or Shirley background is used.

As an example, [Figure 1 a\)](#) shows an XPS peak whose intensity is to be measured. Vertical lines have been drawn to indicate suitable limits for use of the Shirley background. The spectrum after subtraction of this background is shown on an expanded energy scale in [Figure 1 b\)](#). For clarity of display, the zero of the intensity scale in [Figure 1 b\)](#) has been placed at 2 % of the ordinate axis. The end points in [Figure 1 b\)](#) are at the same positions as those in [Figure 1 a\)](#).

Averaging over neighbouring channels may be helpful in defining the signal level at the selected end points, thus improving the precision of peak-height or peak-area measurement. The sets of points to be averaged may be located inside or outside of the chosen end points or may be symmetrically placed about the end points. The end points shall be chosen to be sufficiently far from the peak so that the averaging process does not include significant peak intensity. Harrison and Hazell[17] have derived an expression for the estimated uncertainty in a peak-area measurement (see [5.6](#)) and have shown that a large contribution to this uncertainty comes from uncertainties arising from the choice of end points and the intensities at these end points.

Smoothing of a spectrum, using a Savitzky-Golay[18] convolution with a width less than 50 % of the full width at half-maximum intensity of the peak, may improve the precision of a peak-height determination. However, smoothing should be avoided for peak-area determination since it cannot improve the precision and, if over-done, will distort the spectrum.

[Annex B](#) gives information on the choice of suitable energy limits for the determination of peak intensities or areas in XPS spectra.

5.3 Measurement of peak intensity

5.3.1 Measurement of peak height

A peak height is determined (i) by direct measurement from a chart output using a ruler, (ii) by using computer software to obtain the intensity difference from the baseline to the peak maximum or (iii) by using computer software to fit an appropriate analytical peak shape (Gaussian, Lorentzian or a mixture of the two^{[10][11][12]}) to the experimental spectrum (that is, the group of data points defining the peak of interest). The length of the vertical line with arrows in [Figure 1 b](#) is a measure of the peak height in units defined by the intensity scale (either counts or counts per second).

The use of peak heights in subsequent data processing has advantages arising from the speed of processing and the ease with which this method can be applied with many instruments. However, using peak height as a measurement of intensity has several disadvantages: (i) it is insensitive to peak-shape changes arising from the complex chemistry of an element, (ii) it ignores spectral intensity from secondary features in the spectrum (such as satellite peaks) and (iii) the measured height is very dependent on the choice of inelastic background.

Instruments should be operated with settings chosen to avoid significant nonlinearities in the intensity scales^[19]; alternatively, corrections should be made for counting losses due to the finite dead time of the counting electronics^[19]. Spectra should be corrected for the intensity-energy response function of the instrument before peak heights are measured^[20]. Further information is provided in [Annex A](#).

5.3.2 Measurement of peak area

All modern AES and XPS instruments have computer software that can be used to determine the peak area (e.g. by summing the counts above the inelastic background or by numerical integration). Alternatively, the peak area can be calculated from the parameters obtained after fitting the peak with an appropriate analytical function^{[10][11][12]}. The shaded area in [Figure 1 b](#) illustrates the peak area obtained from integration of the peak defined by the end points and subtraction of the inelastic background in [Figure 1 a](#).

The measured intensity in each channel of an AES or XPS spectrum depends on a number of instrumental parameters and settings^[20]. For specified instrumental conditions, the measured intensity for each channel can be simply expressed as a number of counts (or counts/second) per eV; [Annex A](#) provides further information. A peak area (or peak intensity) is then expressed as the total number of counts (or counts/second) for a specified energy region of summation or integration.

Instruments should be operated with settings chosen to avoid significant nonlinearities in the intensity scales^[19]; alternatively, corrections should be made for counting losses due to the finite dead time of the counting electronics^[19]. Spectra should be corrected for the intensity-energy response function of the instrument before peak areas are measured^[20]. Further information is provided in [Annex A](#).

In practical AES and XPS, an analyst generally wishes to compare intensities of peaks that were measured with identical instrumental settings [e.g. analyser mode, pass energy (for the constant-analyser-energy mode) and retarding ratio (for the constant-retarding-ratio mode)] but differences in certain other settings (e.g. different energy channel widths or different dwell times). The analyst often will not know certain parameters that affect the absolute intensities of measured peaks (see [Annex A](#)) since only relative intensities are needed for practical analyses. In such cases, peak intensities can be determined from simple summations or integrations of measured spectra for the particular conditions, and these intensities are often expressed in units of counts·eV or counts·eV/second. Corrections of peak areas can then be made as needed for different channel widths and dwell times. [Annex A](#) provides further information.

The use of peak intensities derived from measurements of peak areas has some clear advantages over the use of measurements of peak heights. First, account can be taken in the measurement of peak areas of any chemical changes that result in reduced peak height and increased peak width (compared to the corresponding values for the elemental solid). Second, any satellite intensity can be easily included in the measurement of peak area. However, the uncertainty of a peak-area measurement may increase for