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Surface chemical analysis — Electron spectroscopies — Minimum reporting requirements for peak fitting in X-ray photoelectron spectroscopy

Analyse chimique des surfaces — Spectroscopie d'électrons — Exigences minimales pour le rapport d'ajustement de pic en spectroscopie de photoélectrons X

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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 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*.

Introduction

X-ray photoelectron spectra produced from the surfaces of many materials are complex and frequently consist of overlapping or unresolved peaks. The lack of resolution can be due to instrumental parameters, the X-ray line width, the natural line width of the transition, or a contribution from all of these. It is frequently necessary, therefore, to use a mathematical procedure to fit some or all of the peaks in XPS spectra in order to establish the position and intensity of each of the component peaks contained within each peak envelope. This is often the first step in the identification of the chemical states which give rise to the overall peak envelope and the quantification of each chemical state present. The analyst must therefore have confidence in both the position (to establish the chemical state) and the peak area (to allow accurate quantification) of each peak reported following peak fitting.

The mathematical procedure applies model peak and background shapes, the defining parameters of which are varied in order to obtain the optimum fit to the experimental data. Most commonly, the model peak shapes are some combination of Gaussian and Lorentzian functions.

Many of the parameters that should be reported following peak fitting are those that define these curves. Other factors are those which are selected by the analyst to ensure that the peak-fitting process results in a chemically meaningful description of the peak envelope or to minimize the time taken for the fitting process. These include parameters that the analyst

— chooses to fix at a constant value during the fitting process,

— defines as a range of values over which the parameter can vary during the fitting process, and

— mathematically links the value of a parameter to that of another parameter

Peak fitting is a purely mathematical process from which quantitative and qualitative results are obtained which may be related to the chemistry of the surface being analysed. The results will depend upon the analyst’s choice of parameters and constraints and this choice will influence the interpretive conclusions that the analyst reaches from the peak-fitting results. For that reason, it is important that these parameters and constraints are reported. This will allow another analyst to

— assess the reliability and validity of the conclusions drawn from the peak fitting exercise,

— repeat the peak-fitting process on the same data set and obtain the same results, and

— repeat the peak-fitting process on data which has been obtained from a similar sample and be in a position to make a valid comparison of the data sets.

Most software packages which have been designed for use with XPS data contain a peak-fitting routine. These routines allow the operator to select appropriate parameters and apply the desired constraints to the fitting process. It is highly likely that the software will provide an output which reports these and usually includes the facility to copy them for use with another spectrum. Such an output will make reporting the appropriate parameters particularly convenient.

This International Standard is not intended to provide instructions for either fitting XPS peaks or for linking the outcome of a peak-fitting routine to the chemistry of the surface being analysed. Indeed, in the examples shown in this International Standard, it is not claimed that the fitting shown is the only way fitting can be done or even that the examples show the optimum peak fitting method. The examples serve to illustrate the purpose of this International Standard.

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1 Scope

The purpose of this International Standard is to define how peak fitting and the results of peak fitting in X-ray photoelectron spectroscopy shall be reported. It is applicable to the fitting of a single spectrum or to a set of related spectra, as might be acquired, for example, during a depth profile measurement. This International Standard provides a list of those parameters which shall be reported if either reproducible peak fitting is to be achieved or a number of spectra are to be fitted and the fitted spectra compared. This International Standard does not provide instructions for peak fitting nor the procedures which should be adopted.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
background, inelastic
intensity distribution in the spectrum for particles originally at one energy but which are emitted at lower energies due to one or more inelastic scattering processes

[SOURCE: ISO 18115-1, 4.50]

2.2
background, Shirley
background calculated to fit the measured spectrum at points at higher and lower kinetic energy than the peak or peaks of interest such that the background contribution at a given kinetic energy is in a fixed proportion to the total peak area above that background for higher kinetic energies.

[SOURCE: ISO 18115-1:2010, 4.54]

2.3
background, Tougaard
intensity distribution obtained from a model for the differential inelastic scattering cross section with respect to energy loss and the three-dimensional distribution of the emitting atoms in the surface region

[SOURCE: ISO 18115-1:2010, 4.57]

2.4
pass energy
mean kinetic energy of the detected particles in the energy dispersive portion of the energy analyser

[SOURCE: ISO 18115-1:2010, 4.325]

2.5
peak fitting
procedure whereby a spectrum, generated by peak synthesis, is adjusted to match a measured spectrum

2.6
peak synthesis
procedure whereby a synthetic spectrum is generated using either model or experimental peak shapes in which the number of peaks, peak shapes, peak widths, peak positions, peak intensities, and background shape and intensity are adjusted for peak fitting

[SOURCE: ISO 18115-1:2010, 4.329]

2.7
residual spectrum
difference between the experimentally acquired spectrum and the synthesized spectrum

3 Symbols and abbreviated terms

3.1 Abbreviated terms

BE	binding energy
eV	electron volt
FWHM	full width at half maximum
L/G	ratio of the intensity of the Lorentzian component to the Gaussian component of a pseudo Voigt peak consisting of the sum of a Gaussian and a Lorentzian function
PE	pass energy
XPS	X-ray photoelectron spectroscopy

3.2 Symbols

χ^2_{min}	value of χ^2 after the minimization process has been completed
χ^2	chi square
σ_a	standard deviation for the binding energy of a peak
c_i	total number of counts in channel i prior to background subtraction
i	channel number in a spectrum
M	number of independently adjustable parameters used in the fitting process
N	number of energy channels in the part of the spectrum being fitted
r_i	spectrum residual in channel i (obtained from the total number of counts, not counts per second)
Δa	energy by which a peak position is changed (from the position which results in the minimum chi square value) during the process of estimating the uncertainty in the peak position
Δh	amount by which a peak height is changed (from the value which results in the minimum chi square value) during the process of estimating the uncertainty in the peak intensity
Δw	amount by which a peak width is changed (from the value which results in the minimum chi square value) during the process of estimating the uncertainty in the peak width

4 Reporting of relevant data acquisition parameters

4.1 General

This Clause applies to the instrumental parameters that can affect the shape of a peak, peak envelope, or background in a spectrum. Such parameters will therefore affect the parameters that define the fitted spectrum and so they shall be reported.

4.2 Spectrometer

There are instrumental and acquisition parameters that influence the shape of the peaks in an XPS spectrum. These parameters can also affect the results of any peak fitting activity and shall be reported.

In addition, the relationship between instrumental parameters and the results obtained from peak fitting may depend upon the precise design of instrument used. An example of this is the relationship between the pass energy used to acquire the data and the resolution of the peaks in the spectrum. It is therefore necessary to report the manufacturer and the model of the instrumentation used. This can either be the model name (including a version identifier, if appropriate) of the complete spectrometer or the model names of the key components.

4.3 Instrument resolution

The parameters that can affect the results of a subsequent peak fitting shall be reported. These include any of the following factors which can affect the resolution of the spectrometer:

- chosen pass energy;
- if the spectrometer has adjustable slits which control instrument resolution at the entrance and/or exit of the analyser, the settings for those slits;
- if the spectrometer has a transfer lens having an adjustable angular acceptance and/or an adjustable field of view aperture, the settings of each of these can affect the resulting resolution and shall therefore be reported.

Clearly, the above parameters do not form part of the peak-fitting process and the relationship between the values reported and the shape of the spectrum may be dependent upon the spectrometer used. The value of each of these parameters shall be reported so that a meaningful comparison can be made between peak-fitted data obtained from different spectrometers can be made.

4.4 Detector

The type of detector employed in the spectrometer can have an effect upon the shape of the acquired spectrum. Common types of detector include multiple channel electron multipliers and channel plate detectors. The type of detector employed shall be reported. Instruments with channel plates or a large number of channel electron multipliers may be operated in either the “scanned mode” or the “snapshot mode” depending upon whether the median energy of the analyser changes during the spectrum acquisition (scanned) or whether it is constant (snapshot). The type of spectrum acquired shall be reported.

If the quality and reliability of the fitted spectrum is to be fully assessed, then there shall be some indication of the amplitude of the noise in the spectrum. The majority of XPS spectrometers use an electron multiplier connected to some form of pulse counting equipment. This means that the dominant form of noise in the spectrum is due to Poissonian statistics. Since this form of noise is purely statistical, it is only related to the number of counts in each of the channels of the spectrum. For the purposes of reporting peak fitting results, it is only necessary to report the number of counts in the channel that contains the maximum number of counts. It should be noted that if the data are transformed in some way (for example, by correcting for the transmission function of the instrument), then the data may not conform to Poissonian statistics.