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Surface chemical analysis — X-ray photoelectron spectroscopy — Procedures for determining backgrounds

Analyse chimique des surfaces — Spectroscopie de photoélectrons X — Protocoles pour déterminer les fonds continus

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Foreword

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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.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

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Introduction

This Technical Report gives guidance for determining backgrounds in X-ray photoelectron spectra. The methods of background determination described in this report are applicable for quantitative evaluation of spectra of photoelectrons and Auger electrons excited by X-rays from solid surfaces and surface nanostructures.

The use of background determination in X-ray photoelectron spectroscopy (XPS) has developed from the need (i) for accurate quantitative information on chemical composition (including in-depth composition) of surface/interface layers and nanostructures, (ii) for unambiguous identification of chemical states of surface species and (iii) for extracting electronic-structure information from photoelectron spectra excited from solids. It is therefore necessary to separate the intrinsic part of a spectrum, which is associated with the photoionization or photoexcitation process by the X-radiation of interest in XPS or the Auger-electron decay process and which is needed for further analysis, from other parts of the spectrum (the background) appearing due to other processes. There are widely used procedures available for background subtraction in XPS that are reviewed in detail in References [1] to [4]. Here, the most common procedures and their use are summarized, including methods (i) commonly available in commercial software systems, (ii) available and used in more advanced laboratories and (iii) used in individual laboratories to develop understanding of the processes reflected in the XPS spectra.

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Surface chemical analysis — X-ray photoelectron spectroscopy — Procedures for determining backgrounds

1 Scope

This Technical Report gives guidance for determining backgrounds in X-ray photoelectron spectra. The methods of background determination described in this report are applicable for evaluation of spectra of photoelectrons and Auger electrons excited by X-rays from solid surfaces.

2 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115 [5] apply.

3 Symbols and abbreviated terms

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AES	Auger electron spectroscopy
PIA	Partial intensity analysis
QUASES^{TM}	Quantitative analysis of surfaces by electron spectroscopy
REELS	Reflection electron energy loss spectroscopy
XPS	X-ray photoelectron spectroscopy

4 Types of background in XPS

The electrons produced by X-ray irradiation of surfaces are either photoelectrons (as a result of the primary photoionization process) or Auger electrons (as a result of the secondary, core-hole decay process). Contributions to the measured spectra (i.e., electron energy distributions) from electrons scattered inelastically in the sample, from the secondary electron cascade, and — in the case of excitation by non-monochromatic X-ray irradiation — from photoelectrons induced by X-ray satellites and by bremsstrahlung radiation constitute the background. It is usually not necessary in practical XPS to determine the secondary-electron cascade background at low energies.

In this Technical Report, a description of methods for removing X-ray satellites is given in Clause 5 and for removing inelastic electron scattering in Clause 6. A brief comparison is given in Clause 7 of the effectiveness of procedures for removing the effects of electron inelastic scattering from electron spectra.

NOTE 1 In some cases, the intensity of the intrinsic part of a spectrum is distributed among features attributable to the "no-loss" main peak and to various electronic excitations associated with the creation of the core hole. The latter intrinsic contributions are sometimes denoted as the "intrinsic background". The identification of the intrinsic loss features and measurement of their intensities can be important for quantitative applications of XPS.

NOTE 2 Time-varying fluctuations of the analytical signal due to sources of noise [5] will lead to uncertainty in the signal intensity. Intensity contributions due to noise are not included in the types of background discussed in this Technical Report.

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5 Removal of X-ray satellites from electron spectra

For XPS with non-monochromated X-ray sources, a fixed satellite structure is associated with the exciting main X-ray line (often Al or Mg Kα radiation). These X-ray satellites lead to corresponding features in the XPS spectra.

For selected photoelectron peaks measured with Al or Mg X-ray sources, intensity is removed from higher-kinetic-energy channels corresponding to the energy differences between the K$\alpha_{3,4}$, Kβ, etc., X-ray satellite positions and the K$\alpha_{1,2}$ main peak and the corresponding intensity ratios [6] to remove the satellite contributions in the given spectral region. In such a way, scaled photoelectron peaks corresponding to the peaks excited by the X-ray satellites are subtracted. This subtraction process can be applied in turn to remove satellite peaks associated with other photoelectron peaks. The subtraction process may also erroneously remove an equivalent intensity from Auger peaks present in the spectrum if these are mistakenly identified as photoelectron peaks.

6 Estimation and removal of inelastic electron scattering from electron spectra

6.1 General Information

Various procedures have been developed for separating the part of intrinsic origin in the measured photo-excited electron spectra from the contributions due to electrons that are inelastically scattered in the sample [1-4]. These procedures (including those described in Clause 5) are usually applied to XPS data following data acquisition and require digital-data acquisition and handling capability.

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Prior to application of a procedure for removing the inelastic electron scattering, a measured spectrum normally should be corrected for the spectrometer response function [7, 8] in cases where the distortion of the spectral shape due to instrumental effects is not negligible. To remove the effect of inelastic electron scattering in the spectrum, two different strategies can be followed: either to remove (subtract) the contribution attributable to electron inelastic scattering from the spectrum, or to include a background component in the model function being used to fit the spectrum. The electron-scattering contribution can be considered either as a background for the whole spectrum or as a sum of tail contributions [9, 10] from individual photoelectron peaks. In the case of background removal/subtraction, the parameters of the background components are fixed and, after creating such a background, can be subtracted from the measured spectrum. In the case of background fitting, some or all of the parameters of the background components are allowed to vary in the fitting process.

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NOTE The methods described here for removing the contributions of electron inelastic scattering from a spectrum may not be used for some specific applications of XPS (i.e., total reflection XPS or Auger-photoelectron coincidence spectroscopy) without further consideration.

6.2 Procedures to account for inelastic electron scattering

6.2.1 Introduction

Even in the case of very thin samples, a considerable fraction of the electrons in a spectrum have been inelastically scattered; therefore, the estimation of the background for inelastic electron scattering is very important for quantitative applications. Common procedures for removing the effects of inelastic scattering are briefly described.

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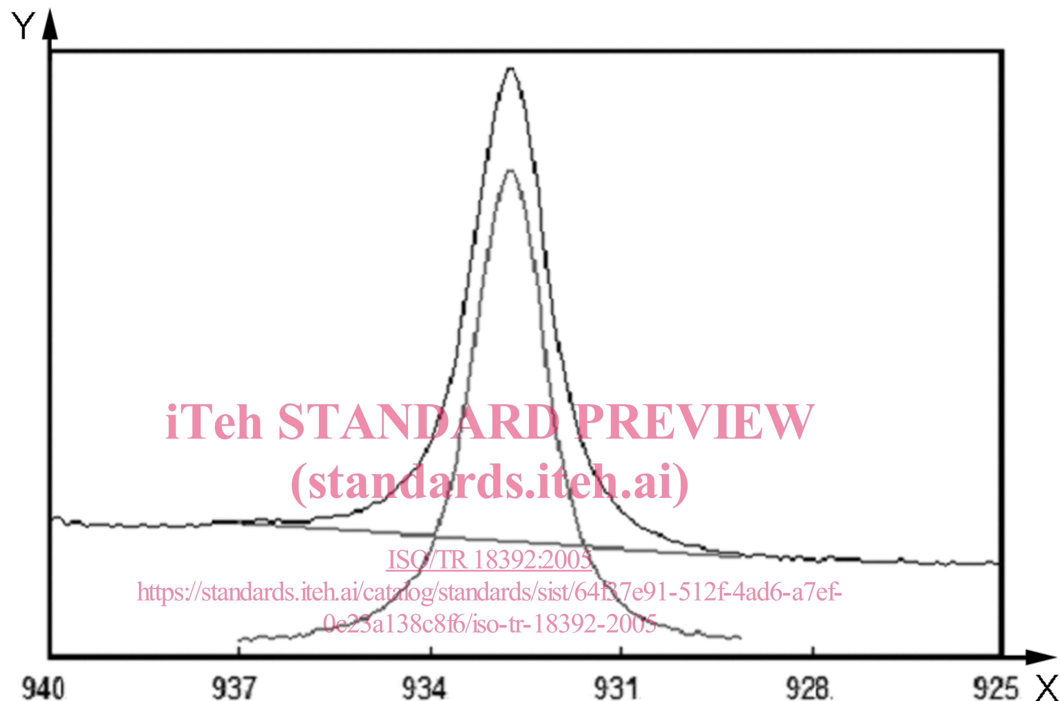
6.2.2 Estimation of the linear background and its removal

In this widely used method, two arbitrarily chosen points in the spectrum are selected and joined by a straight line [2] as an approximation of the true background. These points are generally chosen such that the peak is positioned between them. The intensity values at the chosen points may be the values at the corresponding energies or the average value over a small energy interval around the chosen points. Figure 1 illustrates a linear background for a Cu $2p_{3/2}$ XPS spectrum [11]. This is the most popular method for insulators, where the straight line is horizontal. This approach is used for polymers with great success for peak fitting. However, the use of the linear background in the case of peaks of transition metals (e.g., Fe 2p) leads to significant systematic errors in estimating the peak areas.



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Key

X binding energy (eV)

Y intensity (arbitrary units)

Figure 1 — Example of linear background and its subtraction

(The XPS spectrum used here is copper $2p_{3/2}$. The upper curve shows the measured spectrum and the linear background. The lower curve is the spectrum after subtraction of the background.)

6.2.3 Integral background removal

This (widely used) method, proposed by Shirley [12, 13], employs a mathematical algorithm to approximate the inelastic scattering of electrons as they escape from the solid. The algorithm is based on the assumption that the background is proportional to the area of the peak above the background at higher kinetic energies. This method has been modified to optimize the required iterations [14], to provide for a sloping inelastic background [15], to provide for a background based upon the shape of the loss spectrum from an elastically backscattered electron [16], and to include a band gap for insulators [2]. Figure 2 shows the Shirley or integral background for the Cu $2p_{3/2}$ XPS spectrum given in Figure 1 [11] and the spectrum after this background has been subtracted. It should be emphasized that the correct use of this method requires the application of the valid algorithm [12] and the proper iteration limit [14].

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