
**Surface chemical analysis — X-ray
photoelectron spectroscopy —
Repeatability and constancy of intensity
scale**

*Analyse chimique des surfaces — Spectroscopie de photoélectrons par
rayons X — Répétabilité et constance de l'échelle d'intensité*

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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.

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Introduction

X-ray photoelectron spectroscopy (XPS) is used extensively for the surface analysis of materials. Elements in the sample (with the exception of hydrogen and helium) are identified from comparisons of the measured binding energies of their core levels with tabulations of those energies for the different elements. Information on the quantities of such elements can be derived from the measured photoelectron intensities. Calculation of the quantities present may then be made using formulae and relative sensitivity factors provided by the spectrometer manufacturer. It is important that the sensitivity factors are appropriate for the instrument and this will generally be the case directly after installation of the equipment or calibration of the instrument intensity/energy response function by an appropriate organization. There are two important instrumental contributions to the uncertainty of XPS intensity measurements that are addressed in this International Standard: (i) the repeatability of intensity measurements and (ii) the drift of the intensities with time.

Repeatability is important for analysing the trends and differences between samples that are similar. The instrumental issues that limit the measurement repeatability include the stability of the X-ray source, the settings of the detector, the sensitivity of the instrument to the sample placement, the data acquisition parameters and the data-processing procedure. The drift of the instrument intensity scale will limit the overall accuracy of any quantitative interpretation and arises from such effects as the ageing of components of the structure of the spectrometer, of its electronic supplies and of the detector. In XPS instruments, it has been found that, in service, the instrument intensity/energy response function may change as the instrument ages.

This International Standard describes a simple method for determining the repeatability and constancy of the intensity scale of the instrument so that remedial action, such as improving the operating procedure, resetting of the instrument parameters or recalibration of the intensity/energy response function, may be made. This method should, therefore, be conducted at regular intervals and is most useful if the data include a period in which the instrument has been checked to be working correctly by the manufacturer or other appropriate body. This method uses a sample of pure copper (Cu) and is applicable to X-ray photoelectron spectrometers with unmonochromated aluminium (Al) or magnesium (Mg) X-rays or monochromated Al X-rays.

This method does not address all of the possible defects of instruments since the required tests would be very time-consuming and need both specialist knowledge and equipment. This method is, however, designed to address the basic common problem of repeatability and of drift of the intensity scales of XPS instruments. This method may be conducted at the same time as the spectrometer energy calibration using ISO 15472 [1].

Surface chemical analysis — X-ray photoelectron spectroscopy — Repeatability and constancy of intensity scale

1 Scope

This International Standard specifies a method for evaluating the repeatability and constancy of the intensity scale of X-ray photoelectron spectrometers, for general analytical purposes, using unmonochromated Al or Mg X-rays or monochromated Al X-rays. It is only applicable to instruments that incorporate an ion gun for sputter cleaning. It is not intended to be a calibration of the intensity/energy response function. That calibration may be made by the instrument manufacturer or other organization. The present procedure provides data to evaluate and confirm the accuracy with which the intensity/energy response function remains constant with instrument usage. Guidance is given on some of the instrument settings that may affect this constancy.

2 Symbols and abbreviations

A_2	average peak area for the Cu 2p _{3/2} peak after removing the Shirley background
A_{2j}	a value contributing to A_2 for the j th measurement in a set of measurements
A_3	average peak area for the Cu 3p peak after removing the Shirley background
A_{3j}	a value contributing to A_3 for the j th measurement in a set of measurements
i	identifier for one of the three parameters P_i
j	index for one of the individual measures of the parameter P_{ij}
P_i	parameter representing the mean value of any of A_2 , A_3 and A_3/A_2
P_{ij}	the j th measure of parameter with average value P_i
$U_{95}(P_i)$	uncertainty in the mean value of P_i , at 95 % confidence level
XPS	X-ray photoelectron spectroscopy
δ	value of the tolerance limit for A_3/A_2 for compliance at 95 % confidence level (set by the analyst)
Δ	energy offset for the instrumental binding energy scale, equal to the measured Cu 2p _{3/2} binding energy value for the maximum intensity at the peak minus 932,7 eV
$\sigma(P_i)$	repeatability standard deviation for the parameter P_i