AS ISO 24237—2006

Australian Standard®

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

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- Australian Institute of Physics
- CSIRO Energy Technology
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- Department of Natural Resources and Mines Qld
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- Curtin University of Technology
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Surface chemical analysis—X-ray photoelectron spectroscopy—Repeatability and constancy of intensity scale

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PREFACE

This Standard was prepared by the Standards Australia Committee CH-016, Spectroscopy. This Standard is identical with, and has been reproduced from, ISO 24237:2005, *Surface chemical analysis—X-ray photoelectron spectroscopy—Repeatability and constancy of intensity scale*.

The objective of this Standard is to ensure that the requirements for evaluating repeatability and constancy of the intensity scale if X-ray photoelectron spectrometers for general analytical purposes are achieved.

As this Standard is reproduced from an international standard, the following applies:

(a) Its number appears on the cover and title page while the International Standard number appears only on the cover.

(b) In the source text 'this International Standard' should read 'this Australian Standard'.

(c) A full point substitutes for a comma when referring to a decimal marker.

The term 'informative' has been used in this Standard to define the application of the annex to which it applies. An informative annex is only for information and guidance.

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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].

AUSTRALIAN STANDARD

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 jth 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 jth 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 jth 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
$s(P_i)$	repeatability standard deviation for the parameter P_i