AS ISO 15472—2006
ISO 15472:2001

AS ISO 15472—2006

Australian Standard®

Surface chemical analysis—X-ray photoelectron spectrometers—Calibration of energy scales

This Australian Standard® was prepared by Committee CH-016, Spectroscopy. It was approved on behalf of the Council of Standards Australia on 20 September 2006.
This Standard was published on 20 October 2006.

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- Australian Chamber of Commerce
- Australian Institute of Physics
- CSIRO Energy Technology
- Department of Defence (Australia)
- Department of Natural Resources and Mines Qld
- National Association of Testing Authorities Australia
- Queensland Health Scientific Services

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- Curtin University of Technology
- La Trobe University
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- The University of New South Wales
- The University of Queensland
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This Standard was issued in draft form for comment as DR 06405.

Standards Australia wishes to acknowledge the participation of the expert individuals that contributed to the development of this Standard through their representation on the Committee and through public comment period.

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Surface chemical analysis—X-ray photoelectron spectrometers—Calibration of energy scales

First published as AS ISO 15472—2006.

Published by Standards Australia, GPO Box 476, Sydney, NSW 2001, Australia

ISBN 0 7337 7791 0

PREFACE

This Standard was prepared by the Standards Australia Committee CH-016, Spectroscopy. This Standard is identical with, and has been reproduced from, ISO 14975:2001, *Surface chemical analysis—X-ray photoelectron spectrometer—Calibration of energy scales*.

The objective of this Standard is to ensure that the requirements for calibrating the binding-energy scales of X-ray photoelectron spectrometers 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.

References to International Standards should be replaced by references to Australian or Australian/New Zealand Standards, as follows:

Reference to International Standard ISO		*Australian Standard* AS ISO	
18115	Surface chemical analysis—Vocabulary	18115	Surface chemical analysis—Vocabulary

The terms 'normative' and 'informative' have been used in this Standard to define the application of the annex to which they apply. A 'normative' annex is an integral part of a Standard, whereas 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 binding energies of their core levels, determined from the measured photoelectron spectra, with tabulations of those energies for the different elements. Information on the chemical state of such elements can be derived from the chemical shifts of measured photoelectron and Auger electron features with respect to those for reference states. Identification of chemical states is based on measurements of chemical shifts with accuracies in the range down to 0,1 eV; individual measurements should therefore be made and reference sources need to be available with appropriate accuracies. Calibrations of the binding-energy scales of XPS instruments are therefore required often with an uncertainty of 0,2 eV or less.

This method for calibrating instrumental binding-energy scales uses metallic samples of pure copper (Cu), silver (Ag) and gold (Au) and is applicable to X-ray photoelectron spectrometers with unmonochromated aluminium (Al) or magnesium (Mg) X-rays or monochromated Al X-rays. It is valid for the binding-energy range 0 eV to 1 040 eV.

XPS instruments calibrated for providing analyses within the scope of ISO 17025 [4] and for other purposes may need a statement of the estimated calibration uncertainty. These instruments are in calibration for binding-energy measurements within certain defined tolerance limits, $\pm\delta$. The value of δ is not defined in this International Standard since it will depend on the application and design of the XPS instrument. The value of δ is selected by the user of this International Standard, based on experience in the use of the standard, the calibration stability of the instrument, the uncertainty required for binding-energy measurements in the intended applications of the instrument and the effort incurred in conducting the calibration. This International Standard provides information by which a suitable value of δ may be chosen. Typically, δ is equal to or greater than 0,1 eV and greater than about 4 times the repeatability standard deviation, σ_R. To be in calibration, the divergence from the reference binding-energy values plus the expanded calibration uncertainty for a 95 % confidence level, when added to the instrumental drift with time, must not exceed the chosen tolerance limits. Before the instrument is likely to be out of calibration, it will have to be re-calibrated to remain in calibration. An instrument is re-calibrated when a calibration measurement is made and action is taken to reduce the difference between the measured and reference values. This difference may not necessarily be reduced to zero but will normally be reduced to a small fraction of the tolerance limits required for the analytical work.

This International Standard 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 International Standard is, however, designed to address the basic common problems in the calibration of the binding-energy scales of XPS instruments.

AUSTRALIAN STANDARD

Surface chemical analysis — X-ray photoelectron spectrometers — Calibration of energy scales

1 Scope

This International Standard specifies a method for calibrating the binding-energy scales 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 which incorporate an ion gun for sputter cleaning. This International Standard further specifies a method to establish a calibration schedule, to test for the binding-energy scale linearity at one intermediate energy, to confirm the uncertainty of the scale calibration at one low and one high binding-energy value, to correct for small drifts of that scale and to define the expanded uncertainty of the calibration of the binding-energy scale for a confidence level of 95 %. This uncertainty includes contributions for behaviours observed in interlaboratory studies but does not cover all of the defects that could occur. This International Standard is not applicable to instruments with binding-energy scale errors that are significantly non-linear with energy, to instruments operated in the constant retardation ratio mode at retardation ratios less than 10, to instruments with a spectrometer resolution worse than 1,5 eV, or to instruments requiring tolerance limits of ±0,03 eV or less. This International Standard does not provide a full calibration check, which would confirm the energy measured at each addressable point on the energy scale and which would have to be performed in accordance with the manufacturer's recommended procedures.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 18115, *Surface chemical analysis – Vocabulary*.

3 Symbols and abbreviated terms

a measured energy-scaling error

b measured zero-offset error, in eV

E_{corr} corrected result for the binding energy corresponding to a given E_{meas}, in eV

E_{lem} binding energy of a frequently measured element at which the indicated binding-energy scale is set, after calibration, to read correctly, in eV

E_{meas} a measured binding energy, in eV

$E_{meas\ n}$ average of the measured binding energies for a peak, n, in Table 2, in eV

$E_{meas\ ni}$ one of a set of measurements of binding energy for peak n in Table 2, in eV