ISO 15472:2001 – Surface Chemical Analysis – X-ray Photoelectron Spectrometers – Calibration of Energy Scales

M P Seah, *Surf. Interface Anal.* **31** (2001) 721
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X-ray photoelectron spectroscopy (XPS) is used extensively for the surface analysis of materials. Information on the chemical states of elements can be derived from the measured chemical shifts of photoelectron and Auger electron features by comparison with tabulated data requiring accuracies in the range down to 0.1 eV. Calibrations of the BE scales of XPS instruments are therefore required, often with an uncertainty of 0.2 eV or less.

The International Standard (IS), which is summarised in the above *SIA* reference, provides a detailed procedure to make such calibrations. It specifies a method for calibrating the binding-energy (BE) 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 that have an ion gun for sputter cleaning.

The method involves the use of samples of Cu, Au and Ag and the data of Table 1 [1,2]. It allows analysts to establish a calibration schedule, to test for the BE scale linearity, to confirm the uncertainty of the scale calibration, to correct for small drifts of that scale and to define the expanded uncertainty of the calibration of the BE scale for a confidence level of 95%. This uncertainty includes contributions for behaviours observed in interlaboratory studies but does not cover all defects. The uncertainties are determined with seven repeat measurements. Details of the uncertainty calculations are provided in Annexes. The instrument ageing behaviour is tracked by the use of a control chart.

This IS is not applicable to instruments with BE 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 better than ± 0.03 eV. It does not provide a full calibration check, which would confirm the energy measured at each addressable point on the BE scale and which would have to be performed in accordance with the manufacturer's recommended procedures.

Table 1 — The reference values for the peak positions on the binding energy scale

Peak	Reference binding energies, eV		
	Al Kα	Mg Kα	Monochromatic Al Kα
Au $4f_{7/2}$	83,95	83,95	83,96
Ag $3d_{5/2}$	(368,22)	(368,22)	368,21
Cu L_3VV	567,93	334,90	-
Cu $2p_{3/2}$	932,63	932,62	932,62
NOTE The Ag data included in parentheses are not normally used for calibrations.			

[1] M P Seah, I S Gilmore and S J Spencer, *Surf. Interface Anal.* **26** (1998) 617.
[2] M P Seah, I S Gilmore and G Beamson, *Surf. Interface Anal.* **26** (1998) 642.