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

Analyse chimique des surfaces — Spectromètres de photoélectrons X — Étalonnage en énergie

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 15472 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 7, *X-ray photoelectron spectroscopy*.

This second edition cancels and replaces the first edition (ISO 15472:2001), of which it constitutes a minor revision affecting only Subclause 5.8.1.2. As a result of use of ISO 15472:2001, it became clear that the constraint in 5.8.1.2 limiting users to start and finish at intensities in the range 87 % to 95 % of the peak intensity above zero intensity was over-cautious. For a narrow peak, such as that for gold, it is necessary to include more of the peak to include the required number of data points. This can be done as indicated in the new text of 5.8.1.2 without compromising the accuracy.

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/IEC 17025 [1] 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.