

Australian Standard[®]

Surface chemical analysis—High-resolution Auger electron spectrometers—Calibration of energy scales for elemental and chemical-state analysis

STANDARDS
Australia



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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 17974:2002, *Surface chemical analysis—High-resolution Auger electron spectrometers—Calibration of energy scales for elemental and chemical-state analysis*.

The objective of this Standard is to ensure that the requirements for calibrating the kinetic energy scales of Auger electron spectrometers used for elemental and chemical-state analysis at surfaces are achieved.

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<i>Reference to International Standard</i>	<i>Australian Standard</i>
ISO	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

Auger electron spectroscopy (AES) 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 kinetic energies of their Auger electron peaks (determined from the measured spectra) with tabulations of those energies for the different elements or with handbooks of spectra. Information on the chemical state of such elements can be derived from the chemical shifts of measured 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, making necessary individual measurements having and reference sources available with, the appropriate accuracies. Calibrations of the kinetic energy scales of AES instruments are therefore required, often with an uncertainty of $\leq 0,3$ eV.

The method for calibrating kinetic energy scales specified in this International Standard uses metallic samples of pure copper (Cu), and either aluminium (Al) or gold (Au), and is applicable to Auger electron spectrometers measuring direct spectra with relative resolutions of 0,2 % or better. It is valid for the kinetic energy range 0 eV to 2 250 eV if gold is used, and 0 eV to 1 550 eV if aluminium is used.

Traditionally, kinetic energies of Auger electrons have been referenced to the vacuum level, and this reference is still used by many analysts. However, the vacuum level is ill-defined and can vary from instrument to instrument over a range of 0,5 eV. Although use of the vacuum level reference procedure will generally not cause ambiguity in elemental identification, it may cause uncertainty in measurements of high resolution relating to chemical states. Because of this, instruments designed for both Auger electron spectroscopy and X-ray photoelectron spectroscopy reference the kinetic energies to the Fermi level, giving values typically 4,5 eV higher than those referenced to the vacuum level. For the purposes of this International Standard, the kinetic energies are referenced to the Fermi level.

AES instruments calibrated for providing analyses within the scope of ISO 17025 [1] as well as for other purposes may need a statement of the estimated calibration uncertainty. These instruments are in calibration for kinetic 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 AES instrument. The value of δ is selected by the user, based on experience in the use of this International Standard, the calibration stability of the instrument, the uncertainty required for kinetic 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 $> 0,2$ eV and greater than about four times the repeatability standard deviation, σ_R .

For an instrument to be in calibration, the divergence from the reference kinetic energy values plus the expanded calibration uncertainty for a confidence level of 95 %, when added to the instrumental drift with time, must not exceed the chosen tolerance limits. Before it becomes likely that the instrument is out of calibration, it needs to be recalibrated: a calibration measurement made and action taken to reduce the difference between the measured and reference values. This difference may not necessarily be reduced to zero, but will normally come down to a small fraction of the tolerance limits required for the analytical work.

This International Standard does not address all possible defects of instruments, since the required tests would be very time consuming and would need both specialist knowledge and equipment. However, it does address the basic and common problems in the calibration of the kinetic energy scales of AES instruments.

AUSTRALIAN STANDARD

Surface chemical analysis — High-resolution Auger electron spectrometers — Calibration of energy scales for elemental and chemical-state analysis

1 Scope

This International Standard specifies a method for calibrating the kinetic energy scale of Auger electron spectrometers used for elemental and chemical state analysis at surfaces. It also specifies a calibration schedule for testing the kinetic energy scale linearity at one intermediate energy, for confirming the uncertainty of the scale calibration at one low and one high kinetic energy value, for correcting for small drifts of that scale and for defining the expanded uncertainty of the calibration of the kinetic energy scale with a confidence level of 95 % (this uncertainty includes contributions for behaviours observed in interlaboratory studies but does not cover all possible defects).

It is applicable only to those instruments incorporating an ion gun for sputter cleaning. It is not applicable to instruments with kinetic energy scale errors significantly non-linear with energy. Neither it is applicable to those instruments operated at relative resolutions poorer than 0,2 % in the constant $\Delta E/E$ mode or 1,5 eV in the constant ΔE mode, those requiring tolerance limits of $\pm 0,05$ eV or less, nor to those with an electron gun that cannot be operated in the energy range 5 keV to 10 keV. It does not provide a full calibration check for confirming the energy measured at each addressable point on the energy scale, this being performed according to the manufacturer's recommendations.

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 Terms and definitions

For the purposes of this International Standard, the terms and definitions given in ISO 18115 apply.