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Surface chemical analysis — Medium-resolution Auger electron spectrometers — Calibration of energy scales for elemental analysis

Analyse chimique des surfaces — Spectromètres d'électrons Auger à résolution moyenne — Étalonnage des échelles d'énergie pour l'analyse élémentaire

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

The committee responsible for this document is Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 7, *Electron spectroscopies*.

This third edition cancels and replaces the second edition (ISO 17973:2016), which has been technically revised.

The main changes are as follows:

~~A note has been added to Clause 6.36.3 to provide an example of the sputtering conditions that are usually found satisfactory for cleaning the sample surface of a sample.~~ **NOTE** added to Clause 6.36.3 to provide an example of the sputtering conditions that are usually found satisfactory for cleaning the sample surface of a sample.

~~A paragraph has been added to Clause 6.5 to warn the analyst that it may be necessary to check that the detector is operating within its linear region.~~ **6.5** regarding **need** to check that the detector is operating within its linear region.

~~Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.~~ **Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.**

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 peak energies and peak shapes, with tabulations of peak energies and data in handbooks of spectra for the different elements. To identify the peaks, calibration of the energy scale with an uncertainty of 3 eV is generally adequate, and this document is only intended for work at that level of accuracy (for greater accuracy, see ISO 17974).

The method for calibrating kinetic energy scales specified in this document uses metallic samples of pure copper (Cu) and either aluminium (Al) or gold (Au). It does not include tests for defects in the instrument, since few defects are significant at the level of accuracy concerned.

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 can cause uncertainty in measurements at 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 document, the user is free to choose the reference appropriate to their work.

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Surface chemical analysis — Medium-resolution Auger electron spectrometers — Calibration of energy scales for elemental analysis

1 Scope

This document specifies a method for calibrating the kinetic energy scales of Auger electron spectrometers with an uncertainty of 3 eV, for general analytical use in identifying elements at surfaces. This document also specifies a method for establishing a calibration schedule.

It is applicable to instruments used in either direct or differential mode, where the resolution is less than or equal to 0,5 % and the modulation amplitude for the differential mode, if used, is 2 eV peak-to-peak. It is applicable to those spectrometers equipped with an inert gas ion gun or other method for sample cleaning and with an electron gun capable of operating at 4 keV or higher beam energy.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 18115 (all parts), *Surface chemical analysis — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in the ISO 18115 series apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

4 Symbols and abbreviated terms

AES	Auger electron spectroscopy
a	measured energy scaling error
b	measured zero offset error, in eV
E_{corr}	corrected result for kinetic energy corresponding to given E_{meas} , in eV
E_{meas}	measured kinetic energy, in eV
$E_{\text{meas},n}$	measured kinetic energy for peak n (see Table 1 , Table 1), in eV
$E_{\text{ref},n}$	reference values for kinetic energy position of peak n (see Table 1 , Table 1), in eV
FWHM	full width at half maximum peak intensity above background, in eV
W	FWHM of peak

Δ_n	offset energy, given by average measured kinetic energy for calibration peak minus reference kinetic energy, in eV, for $n = 1, 2, 3, 4$ (see Table 1) <u>Table 1</u>)
ΔE_{corr}	correction added to E_{meas} after calibration to provide corrected kinetic energy result
β	energy scale scan rate for analogue systems, in eV/s
τ	time constant for analogue detector electronics, in s

5 Outline of method

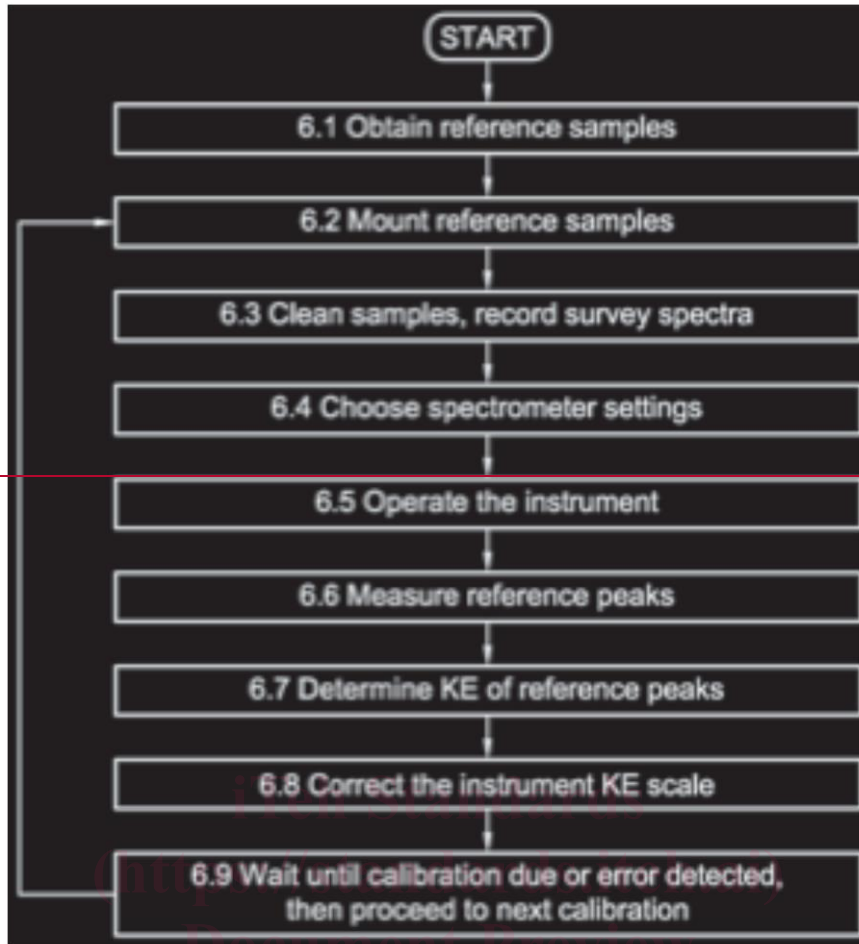
Calibration of an Auger electron spectrometer using this document is performed by obtaining and preparing copper and gold or aluminium reference foils in order to measure the kinetic energies of selected Auger electron peaks. These reference materials are chosen as they provide one Auger electron peak in the high energy range, one in the middle range and one at low energies. The samples are cleaned and spectra are recorded in the direct mode, if that is available, or in the differential mode if not. The energies of the peaks are compared with reference values to provide an energy scale correction. How this correction is implemented depends on the facilities available with the spectrometer. Because this calibration can alter with time, a procedure is defined to enable the calibration to be established at regular intervals.

See ~~Figure 1~~Figure 1 for a flowchart showing the general structure of the work and the sequence of procedures.

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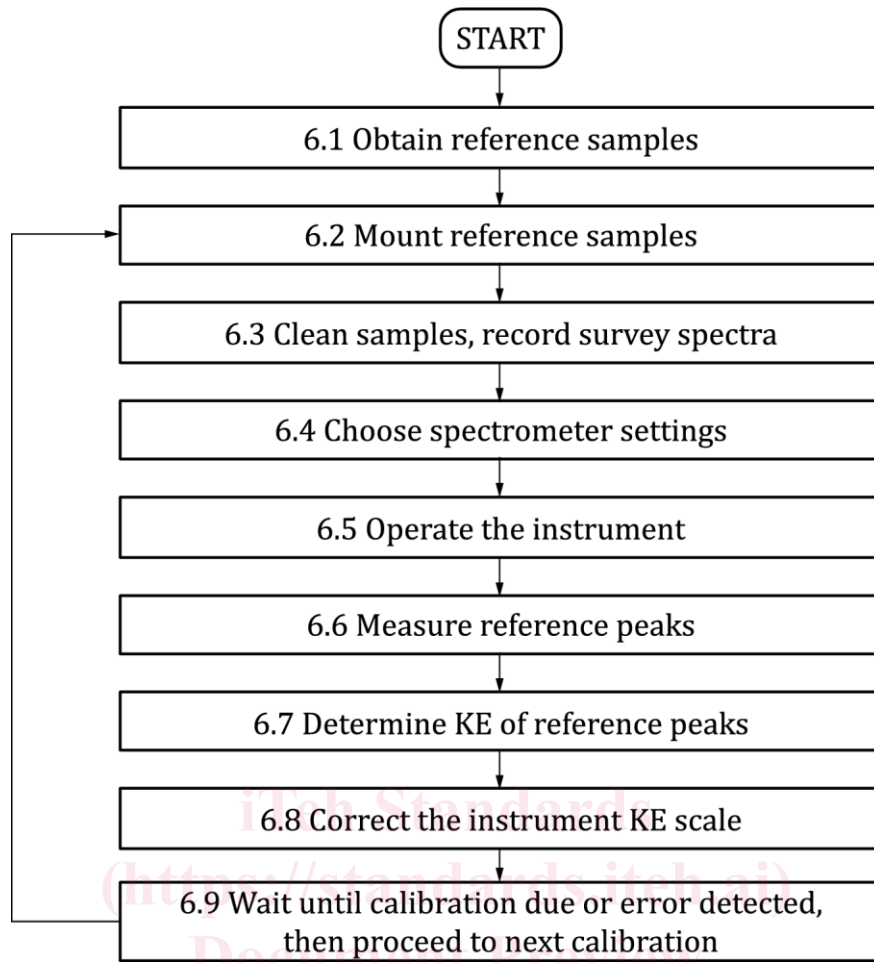
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NOTE The numbers refer to the corresponding subclauses in this document.

Figure 1 — Flowchart of method — Sequence of procedures

6 Energy scale calibration procedures

6.1 Obtaining reference samples

For the calibration of Auger electron spectrometers providing a high signal-to-noise ratio, as defined below, and able to scan the kinetic energy range up to 2 100 eV, use samples of Cu and Au. For spectrometers with a lower signal-to-noise ratio or those only able to scan to 2 000 eV, use samples of Cu and Al.

If, for the spectrometer used, the counts for the Cu L₃VV peak in the direct mode are less than 400 000 counts per channel, or the root mean square noise in the differential spectrum exceeds 0,3 % of the Cu L₃VV peak-to-peak signal, or if the maximum electron beam energy is less than 5 keV, Cu and Al may be used instead of Cu and Au, since the recording of suitable Au data can be time consuming.

For instruments with higher signal intensities and for spectrometers able to scan to 2 100 eV, the use of Au ~~could~~ can be found to be the more convenient and able to provide a calibration over a wider energy range. The requirement for 400 000 counts per channel for the Cu L₃VV peak may be relaxed to 100 000 counts per channel if Savitzky and Golay smoothing is available at nine or more points in the smooth^{[2]-[2]}.