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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

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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 documents 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).

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For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 17973:2002), of which it constitutes a minor revision, which includes changing "counts per second" to "counts per channel".

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 International Standard 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 International Standard 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 may 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 International Standard, the user is free to choose the reference appropriate to his or her work.

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

1 Scope

This International Standard 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. In addition, it 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, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 ISO 18115 apply.

4 Symbols and abbreviated terms

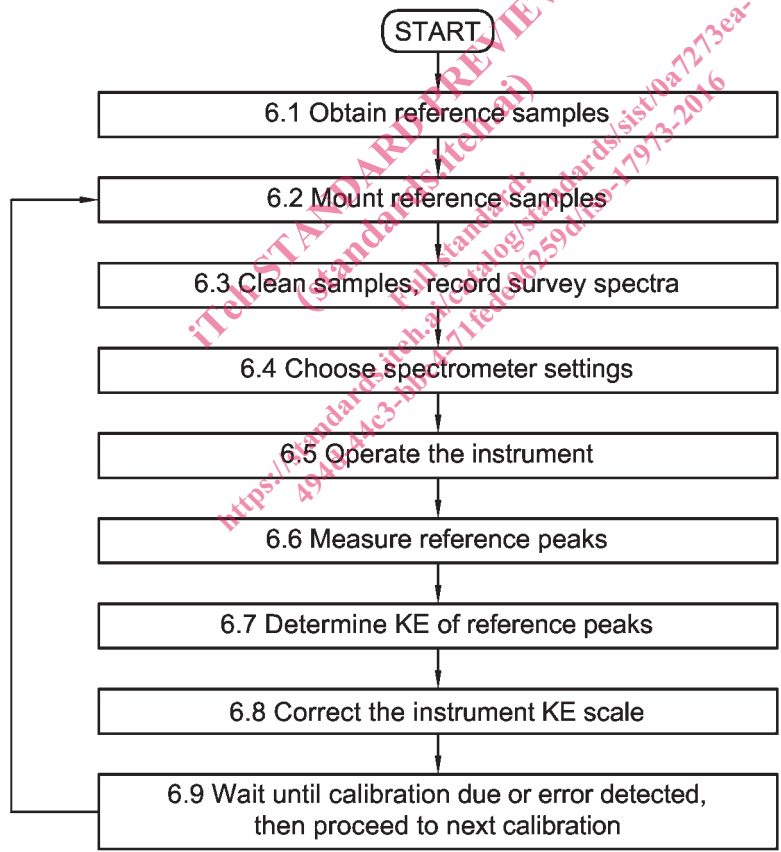
AES	Auger electron spectroscopy
a	measured energy scaling error
b	measured zero offset error, in eV
E_{corr}	corrected result for kinetic energy corresponding to given E_{meas} , in eV
E_{meas}	measured kinetic energy, in eV
$E_{\text{meas},n}$	measured kinetic energy for peak n (see Table 1), in eV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

- 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 International Standard 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 may alter with time, a procedure is defined to enable the calibration to be established at regular intervals.

See [Figure 1](#) for a flowchart showing the general structure of the work and the sequence of procedures.



NOTE The numbers refer to the corresponding subclauses in this International Standard.

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 may be time consuming.

For instruments with higher signal intensities and for spectrometers able to scan to 2 100 eV, the use of Au could 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]

All samples shall be polycrystalline and of at least 99,8 % purity metals which, for convenience, are usually in the form of foils typically of an area 10 mm by 10 mm, and from 0,1 mm to 0,2 mm thick.

If the samples appear to need cleaning, a short dip in 1 % nitric acid may be used for Cu with subsequent rinsing in distilled water. If the Cu sample has been stored in the air for more than a few days, the dip in nitric acid will make the sample cleaning (see 6.3) much easier.

NOTE Better signal-to-noise ratios are often obtained for 10 keV to 20 keV beam energies, rather than for lower beam energies.

6.2 Mounting samples

Mount the samples of Cu and Au or Al on the sample holder or on separate sample holders, as appropriate, using fixing screws or other metallic means to ensure electrical contact. Double-sided adhesive tape shall not be used.

6.3 Cleaning samples

Achieve ultra-high vacuum and clean the samples by ion sputtering to reduce the contamination until the heights of the oxygen and carbon Auger electron peaks are each less than 2 % of the height of the most intense metal peak in each survey spectrum. Record a survey (wide scan) spectrum for each of the samples to ensure that the only significant peaks are those of the required pure elements. The quality of vacuum necessary here is such that the oxygen and carbon peak heights shall not exceed 3 % of the heights of the most intense metal peaks by the completion of the procedure in accordance with 6.6 or at the end of the working day, whichever is the earlier.

All relevant procedures of this International Standard should be completed in one working day. If more than one day is required, the cleanness of the samples shall be confirmed at the start of each day's work.

NOTE Inert gas ion sputtering conditions that have been found suitable for cleaning are 1 min of a 30 μ A beam of 5 keV argon ions covering 1 cm² of the sample.

For examples of direct and differential spectra, see [Figure 2](#).