

BS ISO 17973:2016



BSI Standards Publication

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

**National foreword**

This British Standard is the UK implementation of ISO 17973:2016. It supersedes BS ISO 17973:2002 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CII/60, Surface chemical analysis.

A list of organizations represented on this committee can be obtained on request to its secretary.

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© The British Standards Institution 2016.  
Published by BSI Standards Limited 2016

ISBN 978 0 580 94110 8

ICS 71.040.40

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 September 2016.

**Amendments/corrigenda issued since publication**

Date	Text affected
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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 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](http://www.iso.org/directives)).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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](http://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 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.





# 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

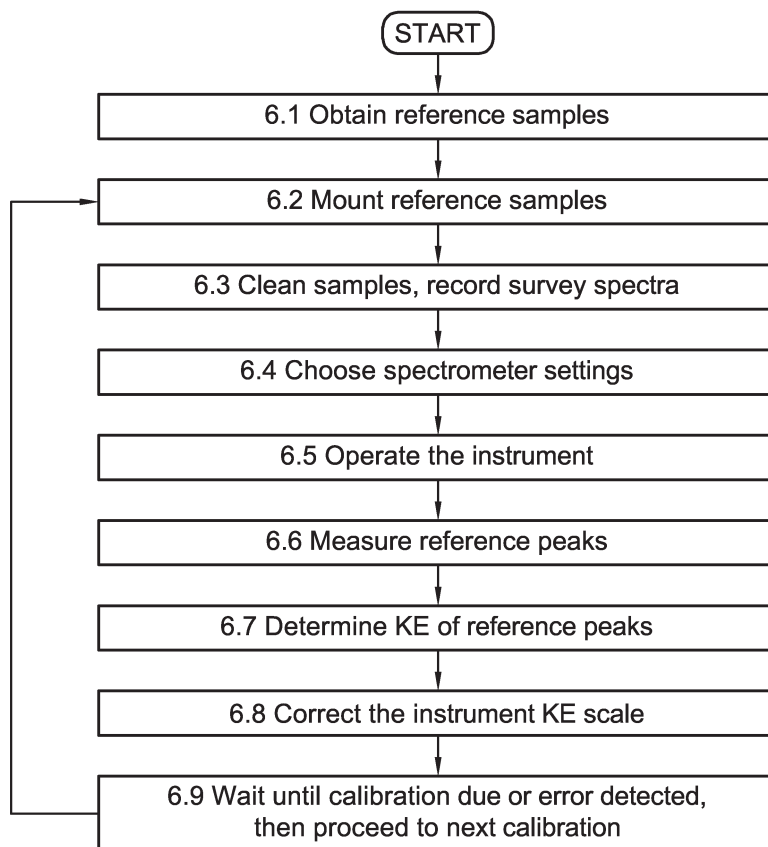
AES	Auger electron spectroscopy
$a$	measured energy scaling error
$b$	measured zero offset error, in eV
$E_{\text{corr}}$	corrected result for kinetic energy corresponding to given $E_{\text{meas}}$ , in eV
$E_{\text{meas}}$	measured kinetic energy, in eV
$E_{\text{meas},n}$	measured kinetic energy for peak $n$ (see <a href="#">Table 1</a> ), in eV
$E_{\text{ref},n}$	reference values for kinetic energy position of peak $n$ (see <a href="#">Table 1</a> ), in eV
FWHM	full width at half maximum peak intensity above background, in eV
$W$	FWHM of peak
$\Delta_n$	offset energy, given by average measured kinetic energy for calibration peak minus reference kinetic energy, in eV, for $n = 1, 2, 3, 4$ (see <a href="#">Table 1</a> )

- $\Delta E_{\text{corr}}$  correction added to  $E_{\text{meas}}$  after calibration to provide corrected kinetic energy result
- $\beta$  energy scale scan rate for analogue systems, in eV/s
- $\tau$  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 L<sub>3</sub>VV 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 L<sub>3</sub>VV 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 L<sub>3</sub>VV 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.<sup>[2]</sup>

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.

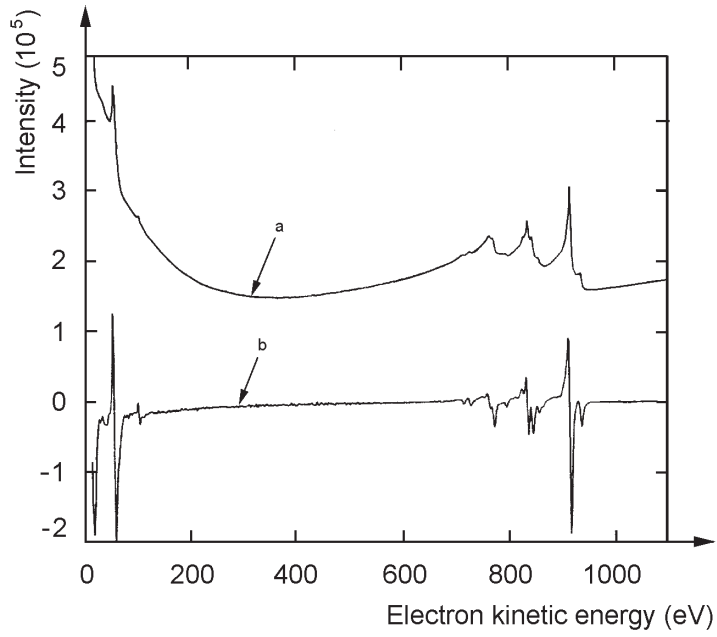
### 6.3 Cleaning samples

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.

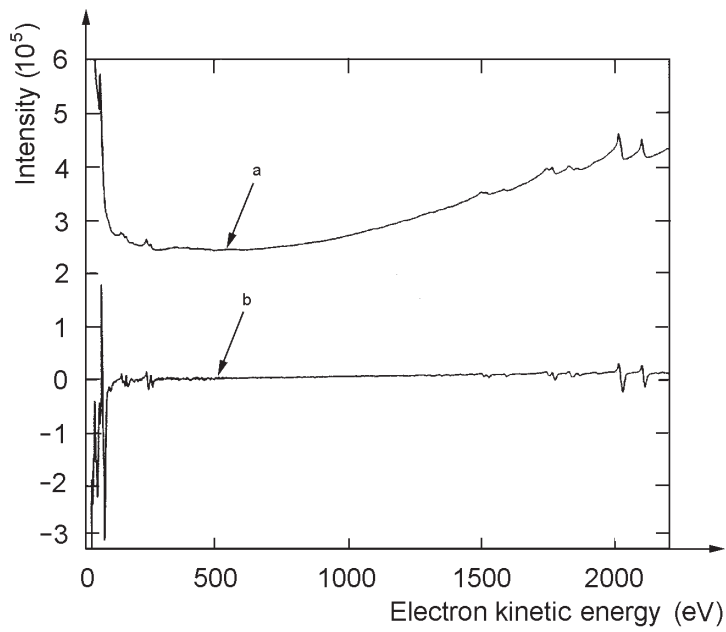
All relevant procedures of this International Standard should be completed in one working day. If more than one day is required, the cleanness of the samples shall be confirmed at the start of each day's work.

NOTE Inert gas ion sputtering conditions that have been found suitable for cleaning are 1 min of a 30  $\mu$ A beam of 5 keV argon ions covering 1 cm<sup>2</sup> of the sample.

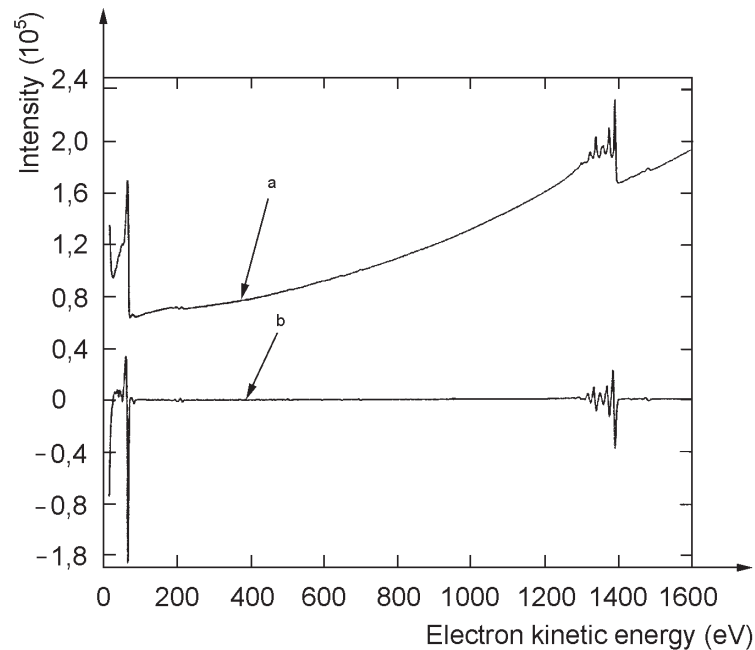
For examples of direct and differential spectra, see [Figure 2](#).



**a) Cu**



**b) Au**



c) Al

- a Direct spectra.
- b Differential spectra.

Figure 2 — Survey spectra of clean copper, gold and aluminium

#### 6.4 Choosing spectrometer settings for energy calibration

Set the spectrometer to acquire spectra in the direct mode, if possible. For spectrometers operating only in differential mode, set the amplitude of the differentiating function to 2 eV peak-to-peak. Choose the remaining spectrometer operating settings for which the energy calibration is required. Repeat the calibration procedure in accordance with 6.4 to 6.6 for each combination of analyser settings of pass energy, retardation ratio, slits, lens settings, etc., for which a calibration is required. Record the values of these settings in the spectrometer calibration log.

**NOTE** The designs of spectrometers and their circuits vary and a spectrometer calibration made for one combination of lens settings, slits and pass energy will not necessarily be valid for any other combination of lens settings, slits and pass energy. Many spectroscopists make measurements under one optimum combination of analyser settings so that only this combination of settings needs calibration. Any calibration made is only valid for the combination of settings used.

#### 6.5 Operating the instrument

**IMPORTANT** — High counting rates<sup>[3]</sup> or incorrect detector voltages<sup>[3][4]</sup> can cause peak distortions leading to erroneous peak energy assignments.

Operate the instrument in accordance with the manufacturer's documented instructions or local documented procedure. The instrument shall have fully cooled following any bakeout. Ensure that the operation is within the manufacturer's recommended ranges for counting rates, spectrometer scan rate and any other parameter specified by the manufacturer. Check that the detector multiplier settings are correctly adjusted. For multi-detector systems, ensure that any necessary optimizations or checks described by the manufacturer are conducted prior to this calibration.

## 6.6 Measurement of reference peaks

**6.6.1** Set the copper sample at the analytical position with the same angle of emission as normally used. The sample positioning procedure shall follow a documented protocol, taking account of the manufacturer's recommendations. Record the Cu  $M_{2,3}VV$  and  $L_3VV$  peaks [see [Figure 3, a](#)) and b)] for the direct or differential modes, using the settings chosen in accordance with [6.4](#).

- a) If using a digital scan for the energy scale, set the increment at, or near, 0,1 eV.
- b) If using an analogue system, set the scan rate,  $\beta$ , at less than  $0,07W\tau^{-1}$  eV/s, where  $W$  is the peak full width at half maximum (FWHM) and  $\tau$  is the detecting amplifier time constant.

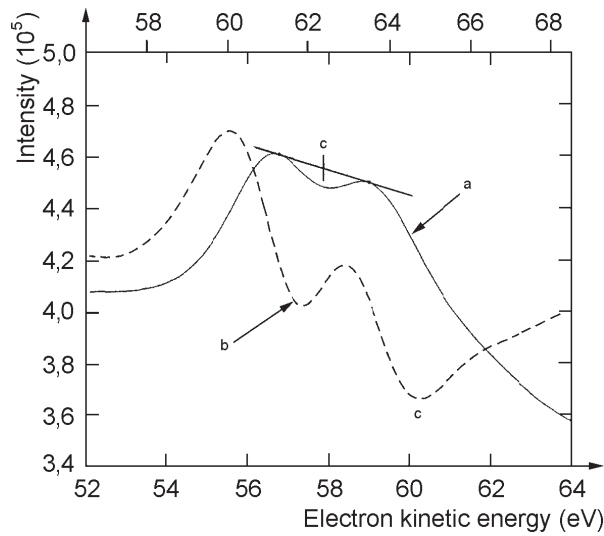
Record at least the signal levels in accordance with [Table 1](#) by a suitable choice of total channel dwell time or  $\tau/\beta$ . If Savitzky and Golay smoothing with nine or more points in the smooth is available, note that the minimum signal levels are lower (see [Table 1](#)).

**6.6.2** Recording the Au  $M_5N_{6,7}N_{6,7}$  peak takes approximately 10 times as long as the Cu  $L_3VV$  peak. If this time is unacceptable, or if the spectrometer only scans to 2 000 eV or if the electron beam energy is less than 5 keV, Cu and Al samples should be used instead of Cu and Au. Remove the Cu sample from the analytical position and replace it with the Au or Al sample. Using the same combination of analyser settings as for [6.6.1](#), except for the total channel dwell time or  $\tau/\beta$ , which may be set as required, record the Au  $M_5N_{6,7}N_{6,7}$  or Al  $KL_{2,3}L_{2,3}$  peaks shown in [Figure 3, c](#)) or d).

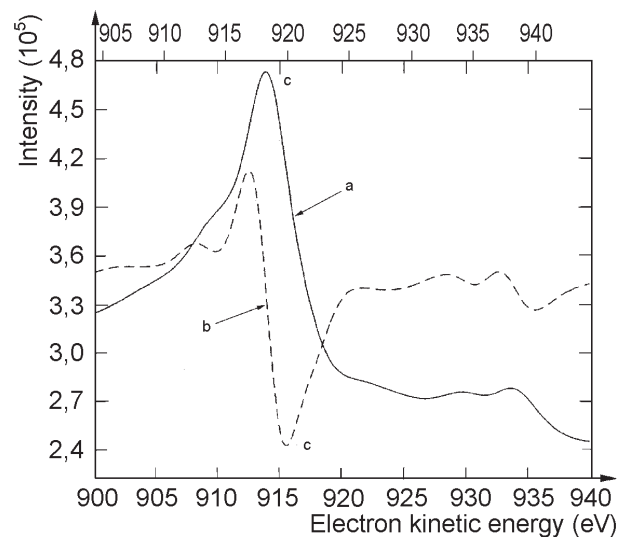
**Table 1 — Signal intensities to be recorded**

Peak number <i>n</i>	Assignment	Minimum counts per channel at the peak for direct mode <sup>a</sup>		Maximum acceptable root mean square noise as a percentage of peak-to-peak signal for differential mode	
		No smoothing	Savitzky and Golay $\geq 9$ point	No smoothing	Savitzky and Golay $\geq 9$ point
1	Cu $M_{2,3}VV$	400 000	100 000	1 %	2 %
2	Cu $L_3VV$	400 000	100 000	1 %	2 %
3	Al $KL_{2,3}L_{2,3}$	400 000	100 000	1 %	2 %
4	Au $M_5N_{6,7}N_{6,7}$	4 000 000	1 000 000	1 %	2 %

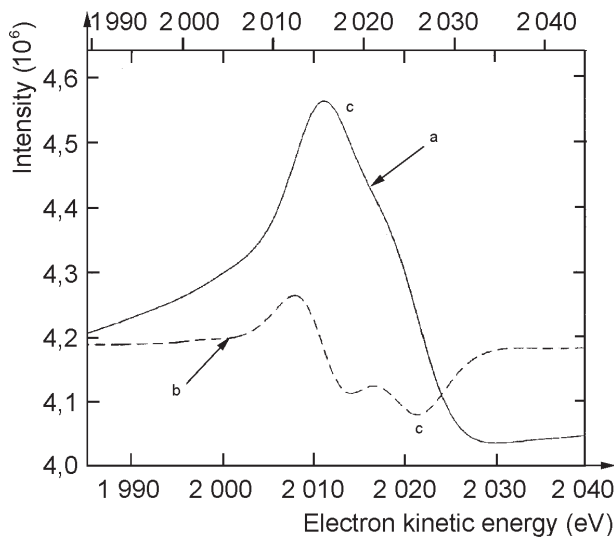
<sup>a</sup> No backgrounds are to be subtracted.



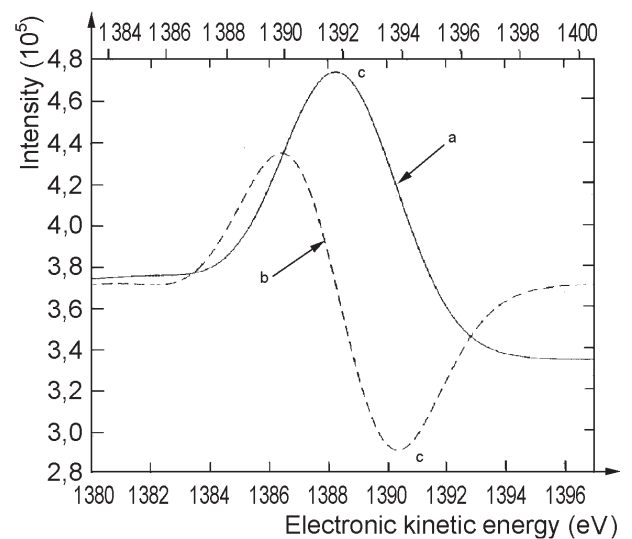
a) Cu M<sub>2,3</sub>VV



b) Cu L<sub>3</sub>VV



c) Au M<sub>5</sub>N<sub>6,7</sub>N<sub>6,7</sub>



d) Al KL<sub>2,3</sub>L<sub>2,3</sub>

- a Direct mode.
- b Differential mode.
- c Calibration peak.

NOTE The differential spectra have been displaced vertically for clarity. The energy scale at the bottom of the figures is vacuum level referenced and that at the top is Fermi level referenced. These spectra have had the noise level reduced for clarity.

**Figure 3 — Example narrow scan spectra of peaks at 0,3 % relative resolution**

### 6.7 Determining measured kinetic energies of reference peaks

By observation of the peaks on a suitably expanded scale, determine, with one exception, the energies of the absolute peak maxima in the direct spectra, or the negative peak minima in the differential spectra, identified in [Figure 3](#). The exception is the Cu M<sub>2,3</sub>VV direct spectral peak in [Figure 3, a](#)), where a tangent is drawn to the two peaks in the doublet to enable the kinetic energy mid-way between the

points of contact to be determined. This procedure may be conducted using the directly acquired data if the intensities specified for the “No smoothing” condition (see [Table 1](#)) have been recorded. If less than this, but if at least the levels shown in the Savitzky and Golay  $\geq 9$  point condition have been acquired, smooth the data with a single application of a Savitzky and Golay cubic/quadratic smooth of at least 9 points, but not exceeding the values given in [Table 2](#).

NOTE 1 Smoothing the data in accordance with [Table 2](#) will also help with intensities higher than those given in [Table 1](#).

NOTE 2 In the differential mode, the Au  $M_{5,6,7}N_{6,7}$  peak has a triplet structure that is not resolved at resolutions poorer than 0,15 %. This triplet reduces to a doublet and, at resolutions worse than about 0,3 %, only the minimum [see c in [Figure 3, c](#)] is visible.

**Table 2 — Maximum number of points for Savitzky and Golay smoothing procedure for channel interval of 0,1 eV and indicated values for maximum spectrometer relative resolution**

Peak number <i>n</i>	Assignment	Maximum spectrometer relative resolution				
		0,1 %	0,2 %	0,3 %	0,4 %	0,5 %
1	Cu $M_{2,3}VV$	9	9	9	9	9
2	Cu $L_3VV$	9	15	19	25	29
3	Al $KL_{2,3}L_{2,3}$	9	19	29	39	49
4	Au $M_{5,6,7}N_{6,7}$	19	29	39	55	69

## 6.8 Determining correction of instrument kinetic energy scale

6.8.1 Calculate the measured instrument offset error,  $\Delta_n$ , for the peak  $n$  given in [Table 1](#) using [Formula \(1\)](#):

$$\Delta_n = E_{\text{meas},n} - E_{\text{ref},n} \quad (1)$$

where  $E_{\text{meas},n}$  is the measured peak energy and  $E_{\text{ref},n}$  is the reference peak energy given in [Table 3](#).

Ensure that the reference energy is appropriate for the choice of mode, direct or differential, being used.

NOTE Reference values are provided for calibration of the spectrometer with energies referred to either the vacuum level or Fermi level. Traditionally, the vacuum level is used and examples of spectra referenced in this way are given in References [\[5\]](#) to [\[8\]](#). If work is also done involving X-ray photoelectron spectroscopy, it may be more convenient to refer the data to the Fermi level as in References [\[9\]](#) and [\[10\]](#).

6.8.2 Review the three values,  $\Delta_1$ ,  $\Delta_2$ , and  $\Delta_3$  or  $\Delta_4$ . If the moduli (i.e. the magnitude, ignoring the sign) of these values are all  $\leq 2$  eV, the instrument is in calibration. If the moduli of some values are  $> 2$  eV, the instrument shall either be recalibrated according to the manufacturer’s instructions or the energy correction defined in [6.8.3](#) shall be applied. After the instrument has been recalibrated for the first time, the measurements defined in [6.6](#) shall be repeated and the values of  $\Delta_1$ ,  $\Delta_2$ , and  $\Delta_3$  or  $\Delta_4$  shall be redetermined to ensure that their moduli after recalibration are  $< 2$  eV.

NOTE If the values of  $\Delta_n$  are all in the range 3 eV to 6 eV, the instrument may currently be calibrated with reference to the Fermi level and not the vacuum level. This is usual for instruments of high energy resolution more commonly calibrated in accordance with ISO 17974.



**Table 3 — Kinetic energy,  $E_{\text{ref},n}$ , referenced to vacuum and Fermi levels**

Peak number $n$	Assignment	Kinetic energy, $E_{\text{ref},n}$ [eV]			
		Direct spectra		Differential spectra	
		Vacuum level	Fermi level	Vacuum level	Fermi level
1	Cu M <sub>2,3</sub> VV	58	62	60	64
2	Cu L <sub>3</sub> VV	914	919	915 <sup>b</sup>	920 <sup>b</sup>
3	Al KL <sub>2,3</sub> L <sub>2,3</sub>	1 388	1 393	1 390 <sup>b</sup>	1 395 <sup>b</sup>
4	Au M <sub>5</sub> N <sub>6,7</sub> N <sub>6,7</sub>	2 011 <sup>a</sup>	2 016 <sup>a</sup>	2 021	2 026

NOTE This table is derived from work in References [11] to [13].  
<sup>a</sup> For beam energies below 6 keV and for 0,3 % <  $R$  ≤ 0,5 %, add 1 eV.  
<sup>b</sup> For 0,27 % <  $R$  ≤ 0,5 %, add 1 eV.

**6.8.3** Where no adjustments are made to the instrument, the corrected kinetic energy,  $E_{\text{corr}}$ , is assumed to be linearly related to the measured kinetic energy,  $E_{\text{meas}}$ , by

$$E_{\text{corr}} = E_{\text{meas}} + \Delta E_{\text{corr}} \quad (2)$$

where  $\Delta E_{\text{corr}}$  is given by

$$\Delta E_{\text{corr}} = aE_{\text{meas}} + b \quad (3)$$

The energy scaling error,  $a$ , and the zero offset error,  $b$ , may be determined by a least squares fit to the negative of the three measured  $\Delta_n$  values,  $-\Delta_1$ ,  $-\Delta_2$  and  $-\Delta_3$  or  $-\Delta_4$ , treated as estimates for  $\Delta E_{\text{corr}}$  at the respective values of  $E_{\text{ref},n}$ . This may be conducted using a computer or with [Formulae \(4\)](#) and [\(5\)](#):

$$a = \frac{-\sum_n E_{\text{meas},n} \Delta_n + \frac{1}{3} \sum_n E_{\text{meas},n} \sum_n \Delta_n}{\sum_n E_{\text{meas},n}^2 - \frac{1}{3} \left( \sum_n E_{\text{meas},n} \right)^2} \quad (4)$$

and

$$b = -\frac{1}{3} \sum_n \Delta_n - \frac{1}{3} a \sum_n E_{\text{meas},n} \quad (5)$$

**6.8.4** Record the values of  $\Delta_1$ ,  $\Delta_2$  and  $\Delta_3$  or  $\Delta_4$ , the values of  $a$  and  $b$ , and the corrective action taken, as appropriate, in the equipment log.

## 6.9 Next calibration

**6.9.1** The next calibration shall be made either directly after any significant modification or service of the equipment, or when peak energies appear to change by 2 eV or more, in accordance with [6.9.2](#), or before the elapse of three calendar months. If at the first recalibration the modulus of any of  $\Delta_1$ ,  $\Delta_2$  and  $\Delta_3$  or  $\Delta_4$  is 2 eV or more, this interval shall be halved. If at the second recalibration the modulus of any of  $\Delta_1$ ,  $\Delta_2$  and  $\Delta_3$  or  $\Delta_4$  is still 2 eV or more, this interval shall be halved again. The reduction in calibration interval shall continue until the moduli of  $\Delta_1$ ,  $\Delta_2$  and  $\Delta_3$  or  $\Delta_4$  are all less than 2 eV at the repeat calibration.

NOTE In principle, a short interval at the start, increased to ensure a safe level of drift, is prudent.<sup>[1]</sup> For the purposes of this International Standard, experience has shown that, very few instruments will need a calibration interval of less than three months; thus, to reduce work for the analyst, this reverse procedure is adopted.

**6.9.2** Electronic components of the spectrometer could fail or change in an unpredictable manner. To give warning of this during the three-month interval, it is useful to review the energies of selected peaks in regularly analysed samples and to compare them with previous values or, if of pure elements, with any literature reference values available. Investigate differences in excess of 2 eV and, if necessary, recalibrate the instrument.

NOTE Handbooks of AES spectra are available with peak energies listed in both the differential mode[5][6][7][8][9] and the direct mode.[8] The energies of the differential minima are typically 1 eV to 5 eV higher in value than the energies of the direct peaks. Further differences between the handbooks arise as follows.

- References [5] to [8] all have data referenced to the vacuum level, whereas in Reference [9], the Fermi level is used. The Fermi level reference value is typically 4,5 eV greater than that referenced to the vacuum level and so some values appear greater by 4 eV and some by 5 eV. The values given in these handbooks may differ from each other and from the values obtained by the user by up to 5 eV, since the peak energy, particularly in the differential mode, depends on both the analyser resolution[14] and the amplitude of the differentiating function.[14]
- The analyser resolution in References [6] to [8] is nominally 0,5 %, whereas in References [5] and [9], it is 0,6 %.
- The amplitude of the differential function in Reference [6] is 2 eV, whereas it is 2 eV for Auger electron energies below 1 000 eV, and 5 eV or 6 eV for higher energies, for References [5] and [7], respectively. In References [8] and [9], the amplitudes of the differentiating functions are 5 eV and 4 eV, respectively. The effect of the higher differential amplitude is to shift peaks to higher energy by up to 2 eV.
- Data listed in compilations associated with X-ray photoelectron spectroscopy[10] include compounds, but are only for high-resolution spectrometers in the direct mode, referenced to the Fermi level.

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