**ASTM INTERNATIONAL**

**Designation: E 2108 – 00**

## Standard Practice for
## Calibration of the Electron Binding-Energy Scale of an
## X-Ray Photoelectron Spectrometer[1]

### 1. Scope

1.1 This practice describes a procedure for calibrating the electron binding-energy (BE) scale of an X-ray photoelectron spectrometer that is to be used for surface analysis with unmonochromated aluminum or magnesium Kα X-rays or monochromated aluminum Kα X rays.

1.2 It is recommended that the BE scale be calibrated after the instrument is installed or modified in any substantive way. Also, it is recommended that the instrumental BE scale be checked, and if necessary, recalibrated at intervals chosen to ensure that BE measurements are statistically unlikely to be made with greater uncertainty than a tolerance limit, specified by the analyst, based on the instrumental stability and the analyst's needs. Information is provided by which an analyst can select an appropriate tolerance limit for the BE measurements and the frequency of calibration checks.

1.3 This practice is based on the assumption that the BE scale of the spectrometer is sufficiently close to linear that the BE scale can be calibrated by measurements of reference photoelectron lines made near the extremes of the working BE scale. In most commercial instruments, X-ray sources with aluminum or magnesium anodes are employed and BEs are typically measured over the 0–1000 eV range. This practice can be used for the BE range from 0 eV to 1040 eV.

1.4 The assumption that the BE scale is linear is checked by a measurement made with a reference photoelectron line or Auger-electron line that appears at an intermediate position. A single check is a necessary but not sufficient condition for establishing linearity of the BE scale. Additional checks can be made with specified reference lines on instruments equipped with magnesium or unmonochromated aluminum X-ray sources, with secondary BE standards, or by following the procedures of the instrument manufacturer. Deviations from BE-scale linearity can occur because of mechanical misalignments, excessive magnetic fields in the region of the analyzer, or imperfections or malfunctions in the power supplies. This practice does not check for, nor identify, problems of this type.

1.5 After an initial check of the BE-scale linearity and measurements of the repeatability standard deviation for the main calibration lines for a particular instrument, a simplified procedure is given for routine checks of the calibration at subsequent times.

1.6 This practice is recommended for use with X-ray photoelectron spectrometers operated in the constant-pass-energy or fixed-analyzer-transmission mode and for which the pass energy is less than 200 eV; otherwise, depending on the configuration of the instrument, a relativistic equation could be needed for the calibration equation. The practice should not be used for instruments operated in the constant-retardation-ratio mode at retardation ratios less than 10, for instruments with an energy resolution worse than 1.5 eV, or in applications for which BE measurements are desired with tolerance limits of ± 0.03 eV or less.

1.7 On instruments equipped with a monochromated aluminum Kα X-ray source, a measurement of the position of a specified Auger-electron line can be used, if desired, to determine the average energy of the X rays incident on the specimen. This information is needed for the determination of modified Auger parameters.

1.8 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

2.1 *ASTM Standards:*
E 456 Terminology Relating to Quality and Statistics[2]
E 673 Terminology Relating to Surface Analysis[3]
E 902 Practice for Checking the Operating Characteristics of X-Ray Photoelectron Spectrometers[3]
E 1016 Guide for Literature Describing Properties of Electrostatic Electron Spectrometers[3]
E 1078 Guide for Specimen Preparation and Mounting in Surface Analysis[3]
E 1523 Guide to Charge Control and Charge Referencing

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Techniques in X-Ray Photoelectron Spectroscopy[3]

2.2 *ISO Standards:*[4]

ISO 9001:2000, Quality Management Systems—Requirements

ISO 15472:2001, Surface Chemical Analysis—X-Ray photoelectron Spectrometers—Calibration of Energy Scales

ISO 18115:2001, Surface Chemical Analysis—Vocabulary

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in X-ray photoelectron spectroscopy and surface analysis, see Terminol-

ogy E 673 and ISO 18115. For definitions of terms used in statistics, see Terminology E 456.

3.2 *Symbols and Abbreviations*—Table 1 shows definitions of the symbols and abbreviations used in this practice.

## 4. Summary of Practice

4.1 A procedure is given for calibrating the BE scale of an X-ray photoelectron spectrometer equipped with one or more of the following sources of characteristic Kα X rays: magnesium (Mg) source; unmonochromated aluminum (Al) source; or monochromated Al source. This procedure is based on ISO 15472. In a first calibration for particular operating conditions of the instrument, or after the instrument has been modified, measurements are made of the binding energies of specified core levels of copper and gold, and these values are then

**TABLE 1 Definitions of Symbols and Abbreviations**

| | |
|---|---|
| $a$ | measured energy scaling error |
| $BE$ | binding energy, in eV |
| $b$ | measured zero offset error, in eV |
| $c_i$ | number of counts in the $i$-th channel |
| eV | electron volts |
| $E_{corr}$ | corrected result for the binding energy corresponding to a given $E_{meas}$, in eV |
| $E_{elem}$ | binding energy of a frequently measured element at which the indicated binding energy scale is set, after calibration, to read correctly, in eV |
| $E_K$ | kinetic energy of a peak, with reference to the Fermi level, in eV |
| $E_{meas}$ | a measured binding energy, in eV |
| $E_{meas\ n}$ | average of the measured binding energies for the peak, $n$, in Table 3, in eV |
| $E_{meas\ ni}$ | one of a set of measurements of binding energy for the peak, $n$, in Table 3, in eV |
| $E_{ref\ n}$ | reference values for the position of peak, $n$, in Table 3, on the binding energy scale, in eV |
| $E_p$ | peak binding energy, in eV |
| $E_0$ | binding energy for first data channel at lower binding energy than the channel with the maximum number of counts, for a peak, in eV |
| FWHM | full width at half maximum peak intensity above the background, in eV |
| $g$ | channel energy separation, in eV |
| $h\nu_{Al}$ | effective X-ray energy from an unmonochromated Al X-ray source, in eV |
| $h\nu_{Al}^{mon}$ | effective X-ray energy from a monochromated Al X-ray source, in eV |
| $h\nu_{Mg}$ | effective X-ray energy from an unmonochromated Mg X-ray source, in eV |
| $i$ | index to represent channel number, where $i = 0$ represents the first channel at lower binding energy than the channel with the maximum number of counts, for a peak, in eV |
| $j$ | number of repeat measurements for a new peak |
| $k$ | number of repeat measurements for the Au $4f_{7/2}$, Cu $2p_{3/2}$ and Ag $3d_{5/2}$ or Cu $L_3VV$ peaks in the repeatability standard deviation and linearity determinations |
| $m$ | number of repeat measurements for the Au $4f_{7/2}$ and Cu $2p_{3/2}$ peaks in the regular calibrations |
| $n$ | designation of the peak identifier in Table 3 |
| $p$ | parameter in Eq A1.1 defined in Eq A1.2 |
| $q$ | parameter in Eq A1.1 defined in Eq A1.3 |
| $r$ | parameter in Eq A1.1 defined in Eq A1.4 |
| $t_x$ | Student's $t$ value for $x$ degrees of freedom of a two-sided distribution for a confidence level of 95 % |
| $U_{95}$ | total uncertainty of the calibrated energy scale at a confidence level of 95 %, in eV |
| $U_{95}^l(E)$ | uncertainty at a confidence level of 95 % arising from the calibration using the Au $4f_{7/2}$ and Cu $2p_{3/2}$ peaks at binding energy $E$, assuming perfect scale linearity, in eV |
| $U_{95}^1$ | uncertainty of $\epsilon_2$ or $\epsilon_3$ at a confidence level of 95 % from Eq 7, in eV |
| $U_{95}^{cl}$ | uncertainty of the calibration at a confidence level of 95 % in the absence of a linearity error, from Eq 12 and Eq 13, in eV |
| XPS | X-ray photoelectron spectroscopy |
| $\alpha$ | Auger parameter, in eV |
| $\alpha'$ | modified Auger parameter, in eV |
| $\Delta_n$ | offset energy, given by the average measured binding energy for a calibration peak minus the reference energy, in eV, for $n = 1, 2, 3, 4$ in Table 3, for a given X-ray source |
| $\Delta E_{corr}$ | correction to be added to $E_{meas}$, after calibration, to provide the corrected result for the binding energy, in eV |
| $\Delta E_{corr\ n}$ | value of $\Delta E_{corr}$ for peaks 1 and 4 in Table 3, in eV |
| $\Delta E_n$ | drift of the binding-energy scale following a calibration for peaks 1 and 4 in Table 3, in eV |
| $\Delta\phi$ | the average of $\Delta_1$ and $\Delta_4$ from Eq 16, in eV |
| $\Delta h\nu$ | difference between $h\nu_{Al}^{mon}$ and $h\nu_{Al}$, in eV |
| $\delta$ | value for the tolerance limit of energy calibration at a confidence level of 95 % (set by the analyst), in eV |
| $\epsilon_2$ | measured scale linearity error at the Ag $3d_{5/2}$ peak from Eq 4, in eV |
| $\epsilon_3$ | measured scale linearity error at the Cu $L_3VV$ peak from Eq 5 or Eq 6, in eV |
| $\sigma_R$ | maximum of $\sigma_{R1}$, $\sigma_{R2}$ or $\sigma_{R3}$, and $\sigma_{R4}$, in eV |
| $\sigma_{Rn}$ | repeatability standard deviation for the seven measurements of the binding energy of peak, $n$, in Table 3, in eV |
| $\sigma_{Rnew}$ | repeatability standard deviation for a new peak, in eV |

compared with corresponding reference energies (**1**).[5] The linearity of the BE scale is checked at a single point on the scale using a measurement of the position of either a specified core level of silver (monochromated Al source) or a specified Auger-electron transition of copper (Mg source or unmonochromated Al source) (**1,2**); additional checks can be made, if desired, with secondary standards. Procedures are given for determining the components of an uncertainty budget in BE measurements and for determining the uncertainties of BE measurements (at the 95 % confidence level) at various times following a calibration. The analyst can thus establish tolerance limits, for example, at the same level of confidence, based on the instrument stability and the analyst's needs so that BE measurements statistically are likely to be made within these limits during specified time intervals following a calibration. The instrument is then adjusted or subsequent BE measurements are corrected. For a routine check of the instrumental calibration, either one or two measurements are made each of the same core levels of copper and gold. Fig. 1 is a flow chart that summarizes the steps of the calibration procedure; references are given to relevant sections of this standard. An optional procedure is provided for determining the average energy of the X rays from a monochromated Al X-ray source, using a measured position of a copper Auger peak in order to determine modified Auger parameters.

## 5. Significance and Use

5.1 X-ray photoelectron spectroscopy is used extensively for the surface analysis of materials. Elements (with the exception of hydrogen and helium) are identified from comparisons of the binding energies determined from photoelectron spectra with tabulated values. Information on chemical state can be derived from the chemical shifts of measured photoelectron and Auger-electron features with respect to those measured for elemental solids.

5.2 Calibrations of the BE scales of XPS instruments are required for four principal reasons. First, meaningful comparison of BE measurements from two or more XPS instruments requires that the BE scales be calibrated, often with an uncertainty of about 0.1 to 0.2 eV. Second, identification of chemical state is based on measurement of chemical shifts of photoelectron and Auger-electron features, again with an uncertainty of typically about 0.1 to 0.2 eV; individual measurements, therefore, should be made and literature sources need to be available with comparable or better accuracies. Third, the availability of databases (**3**) of measured BEs for reliable identification of elements and determination of chemical states by computer software requires that published data and local measurements be made with uncertainties of about 0.1 to 0.2 eV. Finally, the growing adoption of quality management systems, such as, ISO 9001, in many analytical laboratories has led to requirements that the measuring and test equipment be calibrated and that the relevant measurement uncertainties be known.
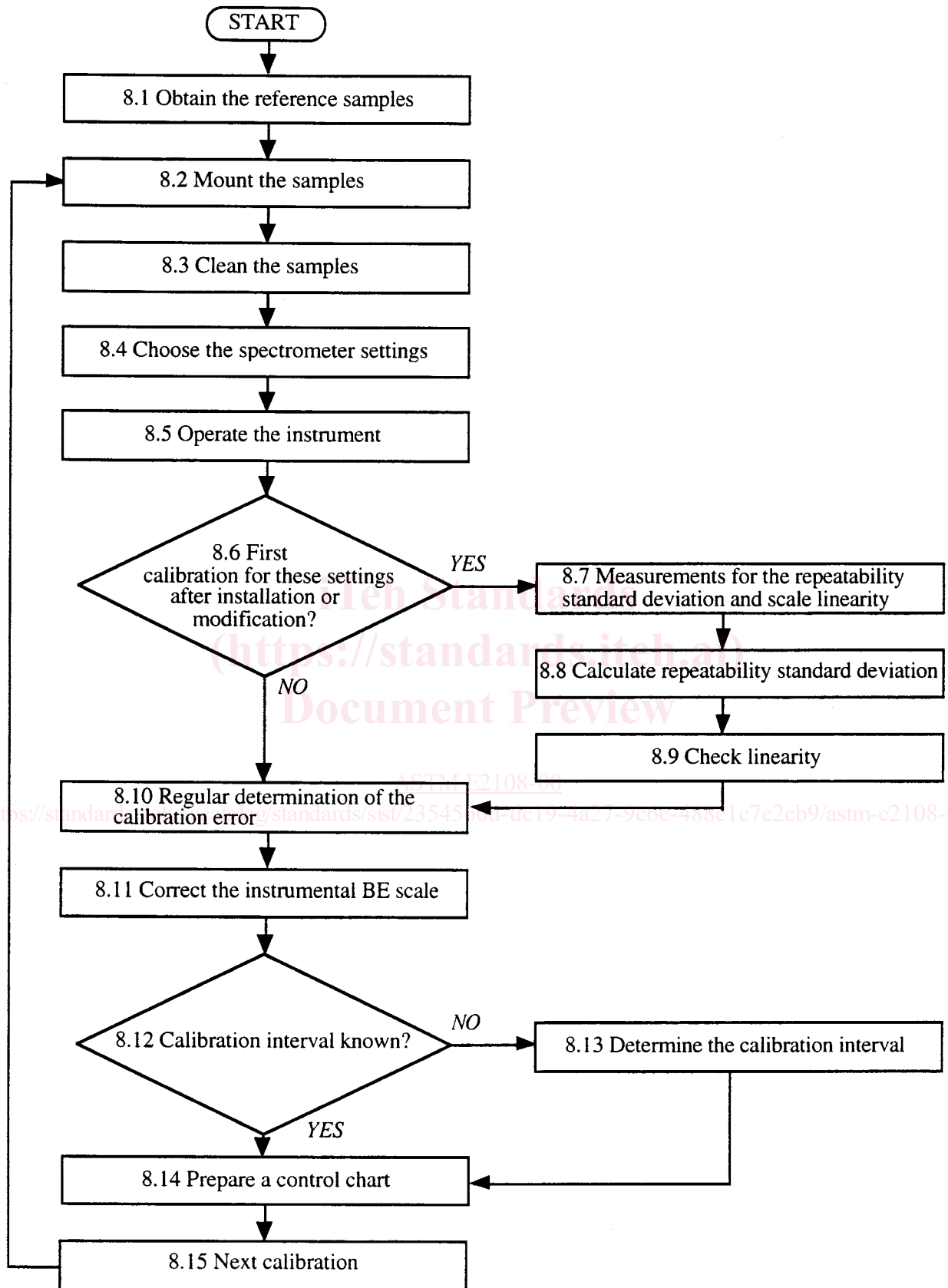
5.3 The actual uncertainty of a BE measurement depends on instrument properties and stability, measurement conditions, and the method of data analysis. This practice makes use of tolerance limits $\pm\delta$ (chosen, for example, at the 95 % confidence level) that represent the maximum likely uncertainty of a BE measurement, associated with the instrument in a specified time interval following a calibration (ISO 15472). A user should select a value of $\delta$ based on the needs of the analytical work to be undertaken, the likely measurement and data-analysis conditions, the stability of the instrument, and the cost of calibrations. This practice gives information on the various sources of uncertainty in BE measurements and on measurements of instrumental stability. The analyst should initially choose some desired value for $\delta$ and then make tests, as described in 8.14 to determine from subsequent checks of the calibration whether BE measurements are made within the limits $\pm\delta$. Information is given in Appendix X1 on how to evaluate the uncertainty of a binding-energy measurement for a material of interest that is associated with the uncertainty of the calibration procedure. This information is provided for four common analytical situations. It is important to note that some BE measurements may have uncertainties larger than $\delta$ as a result of poor counting statistics, large peak widths, uncertainties associated with peak synthesis, and effects of surface charging.

5.4 Instrument settings typically selected for analysis should be used with this practice. Separate calibrations should be made if key operating conditions, such as choices of analyzer pass energy, aperture sizes, or X-ray source, are varied. Settings not specified in this practice are at the discretion of the user, but these same settings should be recorded and consistently used whenever this practice is repeated in order that the current results will be directly comparable to the previous results.

5.5 All of the operations described in Section 8 should be performed the first time that the BE scale is calibrated or after any substantial modification of the instrument. For later checks of the calibration, to be performed on a regular schedule, only the operations in 8.2-8.5. 8.10, 8.11, and 8.14 need to be performed. While the measurements described in 8.7-8.9 for the first calibration require moderate time and effort, they are essential for ensuring that realistic tolerance limits $\pm\delta$ have been chosen. The control chart, described in 8.14, is a simple and effective means of demonstrating and documenting that the BE scale of the instrument is in calibration, that is, within the tolerance limits, for a certain period of time.

5.6 The average energy of the X rays incident on the specimen for instruments equipped with a monochromated Al X-ray source will generally be slightly higher, by up to about 0.2 eV, than the average X-ray energy for instruments equipped with an unmonochromated Al X-ray source (**4**). The actual energy difference depends on the alignment and thermal stability of the X-ray monochromator. An optional procedure to determine this energy difference is given in Appendix X2 from measurements of the Cu $L_3VV$ Auger-electron peak. This information is needed for the determination of modified Auger parameters and Auger-electron kinetic energies on instruments with the monochromated Al X-ray source.

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[5] The boldface numbers in parentheses refer to the list of references at the end of this standard.

START

8.1 Obtain the reference samples

8.2 Mount the samples

8.3 Clean the samples

8.4 Choose the spectrometer settings

8.5 Operate the instrument

8.6 First calibration for these settings after installation or modification? — *YES* → 8.7 Measurements for the repeatability standard deviation and scale linearity

8.8 Calculate repeatability standard deviation

8.9 Check linearity

*NO*

8.10 Regular determination of the calibration error

8.11 Correct the instrumental BE scale

8.12 Calibration interval known? — *NO* → 8.13 Determine the calibration interval

*YES*

8.14 Prepare a control chart

8.15 Next calibration

NOTE 1—The number associated with each step indicates the section in which the operation is described.

**FIG. 1 Flowchart Indicating Sequence of Operations for the Calibration.**

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## 6. Apparatus

6.1 *X-ray Photoelectron Spectrometer*—The X-ray photoelectron spectrometer is the instrument to be calibrated. The essential components of the spectrometer are an X-ray source (preferably with an X-ray tube containing one or more anodes of aluminum, or magnesium, or both, a specimen holder that can hold three or more specimens, and an electron energy analyzer with which photoelectron intensities are measured as a function of photoelectron binding energy. Some instruments with an Al X-ray source are equipped with an X-ray monochromator. XPS instruments also measure the intensities of Auger electrons that are emitted at characteristic energies. See Practice E 902 for procedures to check the operating characteristics of an X-ray photoelectron spectrometer and Practice E 1016 for information on the properties of electrostatic electron spectrometers.

6.2 *Reference Specimens*—The reference specimens, with which the spectrometer is calibrated, are polycrystalline foils of high-purity (at least 99.8 % pure) copper and gold. For instruments with a monochromated Al X-ray source, a polycrystalline silver film of similar purity is also required as a reference specimen. If desired, other high-purity metals can be used to check the linearity of the BE scale throughout its range. See Guide E 1078 for guidance on specimen preparation and mounting. The foil dimensions should be chosen to be compatible with the specimen holder of the X-ray photoelectron spectrometer, and typically are 10 mm by 10 mm with a thickness of 0.2 mm.

6.3 *Ion Gun*—This practice requires use of an ion gun on the X-ray photoelectron spectrometer for sputter cleaning of the reference specimens used for the calibration measurements.

6.4 *Electron Emission Angle*—The electron emission angle should be between 0° and 56° with respect to the surface normal **(5)**. The reference energies used in this practice are only valid for this range of emission angles.

## 7. Principle of the Calibration Method

7.1 For calibration of the XPS instrument with chosen instrumental settings, for example, X-ray source, analyzer pass energy, and apertures, measurements are made of the binding energies of the Au $4f_{7/2}$ and Cu $2p_{3/2}$ photoelectron peaks. These peaks are chosen because they are near the low and high BE limits for many practical analyses, Au and Cu foils of sufficient purity are readily available, the foil surfaces can be cleaned readily by ion sputtering, and the reference energies needed for the calibration have been validated for a range of operating conditions **(5)**. In an initial calibration of the instrument (or a calibration made after any substantive instrumental modifications), a check of the BE-scale linearity is made with either the Ag $3d_{5/2}$ photoelectron peak (with a monochromated Al X-ray source) or with the Cu $L_3$VV Auger-electron peak (with an unmonochromated Al or Mg X-ray source). In this first calibration, a series of seven independent measurements are made for each of the Au $4f_{7/2}$, Cu $2p_{3/2}$ and either Ag $3d_{5/2}$ or Cu $L_3$VV peaks (see 8.7). These measurements are made to determine the repeatability standard deviations for measurements of the three peaks. These standard deviations have contributions from changes in outputs of the spectrometer

power supplies, from the sensitivity of the measured BE to sample position, and from counting statistics in the recorded spectra (although the latter uncertainty is relatively small for the conditions specified in this practice). The other magnitudes of the other two contributions may vary with BE, and so an overall repeatability standard deviation $\sigma_r$ is defined as the largest of the repeatability standard deviations for the three peaks. The calibration will be valid only if the same sample-positioning procedure used in the calibration also is used for regular analyses.

7.2 It has been found in interlaboratory comparisons of XPS measurements that any measured error in measured BEs varies approximately linearly with BE. This practice is based on the assumption that the differences between measured BEs and reference BEs for the designated peaks are both small and linearly, or close to linearly, dependent on BE. A test of the validity of this assumption is provided (see 8.7 and 8.9) by a measurement at an intermediate point on the BE scale using the Ag $3d_{5/2}$ photoelectron peak (monochromated Al X-ray source) or the Cu $L_3$VV Auger peak (unmonochromated X-ray sources). The Ag $3d_{5/2}$ measurement is needed for monochromated Al X-ray sources because the average X-ray energy from this source may be larger, by up to 0.2 eV, than for an unmonochromated Al X-ray source and, as a result, the relative energies of photoelectron and Auger-electron peaks may vary by up to 0.2 eV **(4)**. The Ag $3d_{5/2}$ peak could be used for the linearity check with unmonochromated X-ray sources, if desired, but it will generally be simpler to make this check with the Cu $L_3$VV Auger peak since the copper foil is needed for measurement of the Cu $2p_{3/2}$ peak. Additional checks of BE-scale linearity can be made, if desired, using appropriate elemental metals and the corresponding recommended BEs as secondary standards **(6)**.

7.3 If the BE scale is judged to be sufficiently linear (see 8.9), subsequent checks of the calibration can be performed with the number $m$ of measurements each of the Au $4f_{7/2}$ and Cu $2p_{3/2}$ photoelectron peaks chosen to be 1 or 2 as described in 8.10. An expression is given in this section for calculating the uncertainty $U_{95}$ of the BE-scale calibration at the 95 % confidence level (at the time of the calibration).

7.4 The means by which the BE scale is corrected following a calibration or a calibration check depends on practical details of the instrument. Some methods for making this correction are described in 8.11.

7.5 An important parameter, to be chosen by the analyst, is the tolerance limit $\delta$ for the calibration at the 95 % confidence limit. This parameter also is the tolerance limit, that is, the maximum allowed inaccuracy, again at the 95 % confidence limit, for measured BEs for some defined time following a calibration. The chosen value of $\delta$ depends in part on the analytical requirements and in part on the stability of the instrumental BE scale. Table 2 shows illustrative values for the various uncertainties that contribute to $U_{95}$ for values of $\delta$ of 0.1 eV and 0.2 eV and for values of $m$ of 1 or 2. The difference between $\delta$ and $U_{95}$ is the maximum drift of the instrumental BE scale that should be allowed between calibration checks. Information on how to determine the maximum interval between calibration checks is given in 8.13. It is strongly

**TABLE 2 Contributions to an Error Budget for Calibration of the BE scale (from ISO 15472)**

NOTE 1—The uncertainties are for a 95 % confidence level. The numerical values shown are illustrations to indicate the effects of different choices (in the values of $\delta$ and $m$) on the uncertainty of the calibration $U_{95}$ and the required interval between calibrations for assumed values of $\sigma_r$, $\epsilon_2$, or $\epsilon_3$, and the average drift rate of the BE scale.

| Item | Symbol | Calculated from | Examples | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- |
| | | | If you require a high accuracy | | If you require a lower accuracy | | |
| Tolerance limits, eV | ± $\delta$ | You choose | ± 0.1 | | ± 0.2 | | (Your choice is dictated by the accuracy you require and the number of spectra you have time to acquire in regular calibrations.) |
| Repeatability standard deviation, eV | $\sigma_R$ | Eq 1 | 0.020 | | 0.020 | | (Characteristic of your spectrometer measured at first calibration (see 8.7).) |
| Number of times each pair of spectra is acquired | $m$ | You choose $m$= 1 or 2 | $m$ = 1 | $m$ = 2 | $m$ = 1 | $m$ = 2 | |
| Uncertainty of calibration measurements, eV | $U_{95}^{c1}$ | Eq 12 or Eq 13 | 0.074 | 0.052 | 0.074 | 0.052 | |
| Measure of scale non-linearity, eV | $\epsilon_2$ or $\epsilon_3$ | Eq 4, Eq 5 or Eq 6 | 0.020 | 0.020 | 0.020 | 0.020 | (Characteristic of your spectrometer measured at first calibration (see 8.7).) |
| Uncertainty of energy scale after calibration, eV | $U_{95}$ | Eq 11 | 0.078 | 0.057 | 0.078 | 0.057 | |
| Maximum allowable drift between calibrations, eV | ± ($\delta$-$U_{95}$) | $\delta$ and $U_{95}$ | ± 0.022 | ± 0.043 | ± 0.122 | ± 0.143 | (Define the drift allowable before you are in danger of exceeding your chosen limits± $\delta$ eV.) |
| Maximum calibration interval (for a steady drift rate of 0.025 eV per month), months | - | Section 8.13 | 0.9 | 1.7 | 4.9 | 5.7 | (Choose a convenient interval below this maximum, and less than four months, with safety margin for any erratic behavior.) |
| Your choice of calibration interval, months | - | You choose based on observed drift behavior | Option not practical | 1 | 3 | 4 | (No more than 70 % of the maximum interval in the preceding row to allow a safety margin.) |

recommended that analysts prepare their own version of Table 2 using numerical values from their measurements and the indicated equations and steps.

7.6 An analyst unfamiliar with this practice will have to make an initial choice for $\delta$, and it is suggested that a value of 0.1 eV be chosen. Follow this practice and complete a version of Table 2 to decide whether the selected value of $\delta$ is realistic. In general, the value of $\delta$ should be based on the accuracy needs of the planned analytical work, the expected measurement and data-analysis conditions, the linearity of the BE scale, the stability of the instrument, and a judgment based on the needs and benefits of calibration with the chosen value of $\delta$ versus the cost in instrument and staff time in performing calibrations. For example, if $\delta$ is chosen to be too small, calibration checks may have to be made too frequently to account for possible drift of the BE scale in the interval between calibration checks. Other options in such a situation are to review the operating procedures, for example, the

sample-alignment procedure, or the calibration procedure, for example, the choice of $m$), to determine if one or more of the terms contributing to $U_{95}$ can be reduced. Otherwise, $\delta$ may need to be increased to an acceptable value.

## 8. Calibration Procedure

8.1 *Obtain the Reference Samples*—Use gold and copper reference samples for XPS instruments with unmonochromated X-ray sources. For instruments with a monochromated Al X-ray source, also use a silver sample on the first occasion that the instrument is calibrated for the settings chosen in 8.4; for later determinations of the calibration error, as described in 8.10, it is not necessary to use the silver sample. The samples shall be polycrystalline and with a purity of at least 99.8 %. The samples can conveniently be foils of typical dimensions 10 mm by 10 mm and with thicknesses between 0.1 mm and 0.2 mm.