



Standard Practice for Calibration of the Electron Binding-Energy Scale of an X-Ray Photoelectron Spectrometer¹

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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 $K\alpha$ X-rays or monochromated aluminum $K\alpha$ 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 $K\alpha$ 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 Statistics²

E 673 Terminology Relating to Surface Analysis³

E 902 Practice for Checking the Operating Characteristics of X-Ray Photoelectron Spectrometers³

E 1016 Guide for Literature Describing Properties of Electrostatic Electron Spectrometers³

E 1078 Guide for Specimen Preparation and Mounting in Surface Analysis³

E 1523 Guide to Charge Control and Charge Referencing Techniques in X-Ray Photoelectron Spectroscopy³

2.2 ISO Standards:⁴

¹ This practice is under the jurisdiction of ASTM Committee E42 on Surface Analysis and is the direct responsibility of Subcommittee E42.03 on Auger Electron Spectroscopy and X-Ray Photoelectron Spectroscopy.

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² *Annual Book of ASTM Standards*, Vol 14.02.

³ *Annual Book of ASTM Standards*, Vol 03.06.

⁴ Available from American National Standards Institute, 25 W. 43rd St., 4th Floor, New York, NY 10036.