Standard Practice for
Checking the Operating Characteristics of X-Ray Photoelectron Spectrometers[1]

This standard is issued under the fixed designation E 902; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers a procedure for checking some of the operating characteristics of an X-ray photoelectron spectrometer. Tests herein provide checks of the repeatability of intensity measurements and the drift of the intensities with time. This practice may be conducted at the same time as the spectrometer energy calibration using Practice E 2108.

1.2 *Limitations*—This practice is meant to augment, and not to replace, the calibration procedures recommended by the manufacturer of the spectrometer. This practice is also not meant to be used as a means of comparison between X-ray photoelectron spectrometers, but only as a self-consistent check of the operating characteristics of an individual spectrometer.

1.3 *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:* [2]

E 673 Terminology Relating to Surface Analysis

E 996 Practice for Reporting Data in Auger Electron Spectroscopy and X-ray Photoelectron Spectroscopy

E 1078 Guide for Specimen Preparation and Mounting in Surface Analysis

E 2108 Practice for Calibration of the Electron Binding-Energy Scale of an X-Ray Photoelectron Spectrometer

2.2 *ISO Standards*

ISO 21270 Surface Chemical Analysis — X-ray Photoelectron and Auger Electron Spectrometers – Linearity of Intensity Scale

ISO 24237 Surface Chemical Analysis— X-ray Photoelectron Spectroscopy – Repeatability and Constancy of Intensity Scale

3. Terminology

3.1 *Definitions*—Terms used in X-ray photoelectron spectroscopy are defined in Terminology E 673.

3.2 Additionally, the following terms and abbreviations are used throughout this practice:

c	=	counts
ch	=	channel
cps	=	counts per second
eV	=	electron volts
i	=	number of data channels acquired for the peak of interest
n	=	number of channels
no.	=	number of
S/B	=	signal-to-background ratio
sc	=	span
Δx	=	step size, eV, between successive data channels
A	=	peak area above background, mm^2
B	=	background height, mm
H	=	maximum peak height above background, mm
I_A	=	peak area intensity above background, c-eV/s
I_H	=	maximum signal intensity above background, cps
P	=	peak position on the binding energy scale, eV
FWHM	=	full width at half maximum

4. Significance and Use

4.1 This practice should first be used to establish the operating characteristics of a particular X-ray photoelectron spectrometer at a time when the spectrometer performance is known to be optimum. Hence, the spectrometer settings in Section 5 and the expected performance figures given in

Section 7 are to be taken only as guides, to be supplanted by the behavior of the user's actual spectrometer.

4.2 Subsequently, this practice should be used as a routine check, performed at frequent intervals with the same instrument settings, and the results compared with those obtained in 4.1. Significant deviation from optimum performance may indicate that the spectrometer requires recalibration or other maintenance.

4.3 Typical analysis settings should be used with this practice. The use of settings not specified by this practice is left to the discretion of the user, however, the settings should be recorded in accordance with Practice E 996 and the same settings should be used consistently whenever this practice is repeated, so that the results obtained will be directly comparable to previous results.

5. Procedure

5.1 *Obtain the Reference Sample*—Use a copper reference sample; the sample shall be polycrystalline and with a purity of at least 99.8 %. The sample can conveniently be a foil of typical dimensions 10 by 10 mm. The specimen should be larger than the analysis area of the spectrometer; the analysis area being defined as either the area viewed by the analyzer or the area of illumination of the X-ray beam, whichever is smaller. The thickness should be between 0.1 mm and 0.2 mm. A new copper gasket segment can also be used. If the Cu sample appears discolored, a short dip in 1 % nitric acid may be used to clean the surface with subsequent rinsing in distilled water. If the Cu sample has been stored in air for more than a few days, the cleaning with nitric acid will reduce the time needed for later sputter cleaning. Guide E 1078 recommends additional specimen handling precautions that may be required.

5.2 Mount the copper specimen at the usual specimen position in the spectrometer, and in electrical contact with the specimen holder.

5.3 Use an ion sputter gun to clean the specimen until the C1s and O1s peak heights above background are each less than or equal to 2 % of the Cu 3p peak height above background. If sputtering is not available, the intensities of the C1s and O1s peaks may exceed 10 % of the Cu 3p peak; in this case, record the C1s and O1s peak heights above background.

5.4 Set and record the anode material, excitation potential, emission current, any leakage current, anode height (if adjustable), specimen tilt, and the pass energy or slit widths. Use typical analysis settings. For all spectra, choose at least ten data channels per electron volt and adjust the time per point and number of sweeps to meet the counting criteria noted in 5.5-5.7. Record these settings in a manner consistent with Practice E 996 and use them exactly the same way each time the spectrometer is checked.

5.5 Acquire and store the photoelectron spectrum of the Cu 2p doublet between 963 and 923 eV, with enough scans to collect at least 10 000 counts at the peak maximum.

5.6 Acquire and store the photoelectron spectrum of the Cu 3p doublet between 86 and 66 eV binding energy, with enough scans to collect at least 5000 counts at the peak maximum.

5.7 If using Mg X-rays, acquire and store the spectrum of the Cu $L_3M_{4,5}M_{4,5}$ Auger line between 340 and 330 eV (Note 1) on the binding energy scale. If using Al X-rays, acquire and store the spectrum of the Auger line between 573 and 563 eV on the binding energy scale (Note 1). In either case, acquire enough scans to collect at least 10 000 counts at the peak maximum.

NOTE 1—For instruments where the minimum scan width is larger than recommended, use the minimum allowable scan width. For instruments where the energy interval cannot be set up in integer steps, use the closest allowed energy.

6. Treatment of Data

6.1 Print or plot the spectra as obtained in 5.5-5.7. In addition:

6.1.1 Print or plot the Cu $2p_{3/2}$ line between 938 and 928 eV binding energy.

6.1.2 Print or plot the Cu $3p_{3/2}$ line between 86 and 66 binding energy.

6.2 *For Spectra 6.1.1 and 6.1.2*—If a computer can be used to measure the peak and background, fit the background with a linear baseline, as shown in Fig. 1, and record I_H and the endpoints. If a linear background fit is not available, an alternate background shape (for example, an integral background) may be used, however, this background may change the signal intensity. Whichever background shape is chosen, the same background shape and endpoints should be used whenever this practice is repeated. Also measure B (Note 2) for each spectrum, as shown in Fig. 1.

NOTE 2—Some data-processing software packages automatically suppress the background, either when the data is collected or when it is displayed. The user should ensure that the background has not been altered before making the background measurement. Information about the treatment of backgrounds for a particular instrument can often be obtained by contacting the manufacturer of the software package or indirectly by comparison of computer-acquired data with analog data acquired with the same instrument settings over the same energy range.

6.2.1 If a computer cannot be used to establish the background, draw a linear background, as shown in Fig. 1 and record the energies above and below the peak maximum where the baseline intersects the data spectrum. Measure H, in millimetres, for each spectrum and make note of the counts per millimetre. Also measure B, in millimetres, for each spectrum (Note 2).

6.2.2 If possible, use peak fitting algorithms to measure and record P, in electronvolts, for each spectrum and the FWHM, in electronvolts, for spectrum 6.1.1, as shown in Fig. 1. For an accurate comparison to the standard binding energies in Table 1, note that for the Cu 3p doublet there is fit to a quadratic to the top 10 % of the most intense peak. It must be further noted that a curve fit or peak synthesis routine will likely yield results that differ slightly from that method. Whichever method is chosen, the same method should be used whenever this practice is repeated. If a peak fitting routine is not available, then accurately measure, compute, and record these values from the data outputs of spectra 6.1.1 and 6.1.2 as shown in Fig. 1.

6.3 ISO standards 21270 and 24237 discuss parallel issues.

7. Analysis of Data

7.1 Refer to Section 3 for definitions and abbreviations. For all peaks, record the signal intensity. If this information is not