Calibrated Procedure for Setting Pulse-Height Parameters in Wavelength-Dispersive Spectrometry

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Electron-probe microanalysis (EPMA) routinely makes use of wavelength-dispersive spectrometry (WDS) to measure characteristic X-ray photons over a wide range of concentration, so the WDS pulse processing system should separate counts from electronic noise over a wide range of count rate. WDS pulse processing hardware is simple compared to the sophisticated pulse processing hardware used in energy-dispersive spectrometry (EDS), as there is no monitoring of deadtime or active referencing to a zero strobe. WDS uses X-ray diffraction to select the desired X-ray wavelength, and all measured pulses are presumed to be for the element of interest. The pulse height analysis (PHA) system is set to discriminate between baseline noise and the measured X-ray pulses.

Amplification of the detector signal is made using detector voltage referred to as *bias* (typically in the 1600-1800 volt range) and a gain setting in the electronic amplifier. On JEOL electron microprobes the gain setting is an integer multiplier (e.g., 16x, 32x, 64x, or 128x), and on Cameca microprobes the gain parameter is specified from a range of values. The bias and gain are set for a particular X-ray of interest, and a single-channel analyzer (SCA) scan is used to inspect the pulse distribution of the X-ray detector. For JEOL instruments this SCA scan is 0-10V with a pulse ideally located at 4V, and for Cameca instruments it is 0-5V with a pulse ideally located at 2V. Failure to set the PHA parameters to the correct values may result in a pulse not being registered at all, or some portion of baseline noise being registered as counts. Further, gain shift due to changing count rate requires attention to PHA settings to ensure processing over this range of count rate. It is therefore up to the analyst to understand and implement PHA settings.

JEOL electron microprobes use flow-proportional detectors streaming P-10 (90% Ar – 10% CH_4) for measurement of low energy X-rays, and sealed Xe detectors for the measurement of high energy X-rays. Cameca electron microprobes use either low or high-pressure P-10 detectors for low and high energy photons, respectively. Two different approaches are taken with setting of PHA parameters on these systems. JEOL PHA settings use an integral gain values coupled with a per-element bias, and Cameca PHA settings are made at constant bias and either fixed or per-element gain selected from a range of values. The JEOL SCA is set to use a constant baseline level (0.5 to 0.7 volt) with gain and bias set to place the pulse at a nominal 4V, and either an integral or differential window is used. The Cameca SCA ranges from 0-5 volts and baseline and window settings are defined by either the operating system or the user. Experience with measurement of WDS deadtime constants on JEOL microprobe detectors resulted in use of bias set on a per-element basis on each detector. This places the pulse at 4V and provides consistent clearance above the baseline with room for the escape peak, and headroom to permit inclusion of coincidence pulses at high count rates.

The PHA calibration method described here is specific to the JEOL instrument, and was initially developed for Sandia-TASK [1]. First, X-ray energies are selected that span the energy range accessible by each spectrometer. For example, an instrument with several spectrometers each with PET and LiF crystals could use the Kα lines of Si, Ca, Ti, Fe, Cu, and Ge for calibration. Pure element standards or compounds are ideal for this use. All spectrometers with PET crystals are then peaked on the same X-ray line (e.g. Si Kα on Si metal). With all spectrometers at this peak position, the probe current is adjusted to obtain no higher than 20 kcps count rate. All spectrometers are then set to the same gain value for simplicity. Next, a bias scan is acquired by setting the SCA to a 4 volt baseline with 0.2 volt window in differential mode so that only 4-4.2V is counted. The bias values are then varied between 1500 and 1800V and the resulting peak on the bias scan, if any, is the bias value at which an SCA scan will produce a 4V pulse (Fig. 1A). An SCA scan can confirm the bias value does indeed produce a 4V pulse (Fig. 1B), but in practice the bias scan is all that is necessary. This procedure is then repeated for each set of gain values and then repeated for all the elements that are used to span the energy range as listed above. The data are plotted using the natural log of the X-ray energy vs. the bias value measured for each element. For Ti Kα the

energy is 4.508 keV, ln(4.508)=1.506 and a bias scan at 32x gain measures a peak at 1685V (Fig 1D, green diamond). Note that the same bias will be observed using either PET or LiF since it is the energy not spectrometer position that matters. The relationship between ln(E) and bias is linear (of form y = mx + b, where x=ln(energy) and y=bias in volts), and a linear least squares fit is made to these data to determine the slope and intercept. This LS equation can then be used for any element on that spectrometer by inputting the energy value, computing the bias, and using that bias for measurement of that X-ray line. Several gain and bias combinations can be used on the JEOL (e.g., 32x, 64x, and 128x in Fig. 4), and experience shows these combinations produce identical intensities and P/B ratios. In our lab we use higher gain coupled with lower bias to prolong life of the detector and limit the bias to the range 1600-1750 V.

It is straightforward to determine the correct PHA settings for an analytical run using this method. This capability has been added to the Probe for EPMA software so that a "calibrated" bias value can be calculated for a given integral gain. Additionally, the use of PHA plots identifies problems with detector performance, internally inconsistent PHA measurements, and can be used to track changes in P-10 gas chemistry as the tank empties out and the Ar:CH$_4$ ratio changes. The Excel sheet used to set up this calibration will be discussed.

References
[1] Chambers, W.F., "Sandia-TASK 8 A Subroutined Electron Microprobe Automation Program", Sandia National Laboratories, Contract DE-ACO4-76DPO0789, 1978.
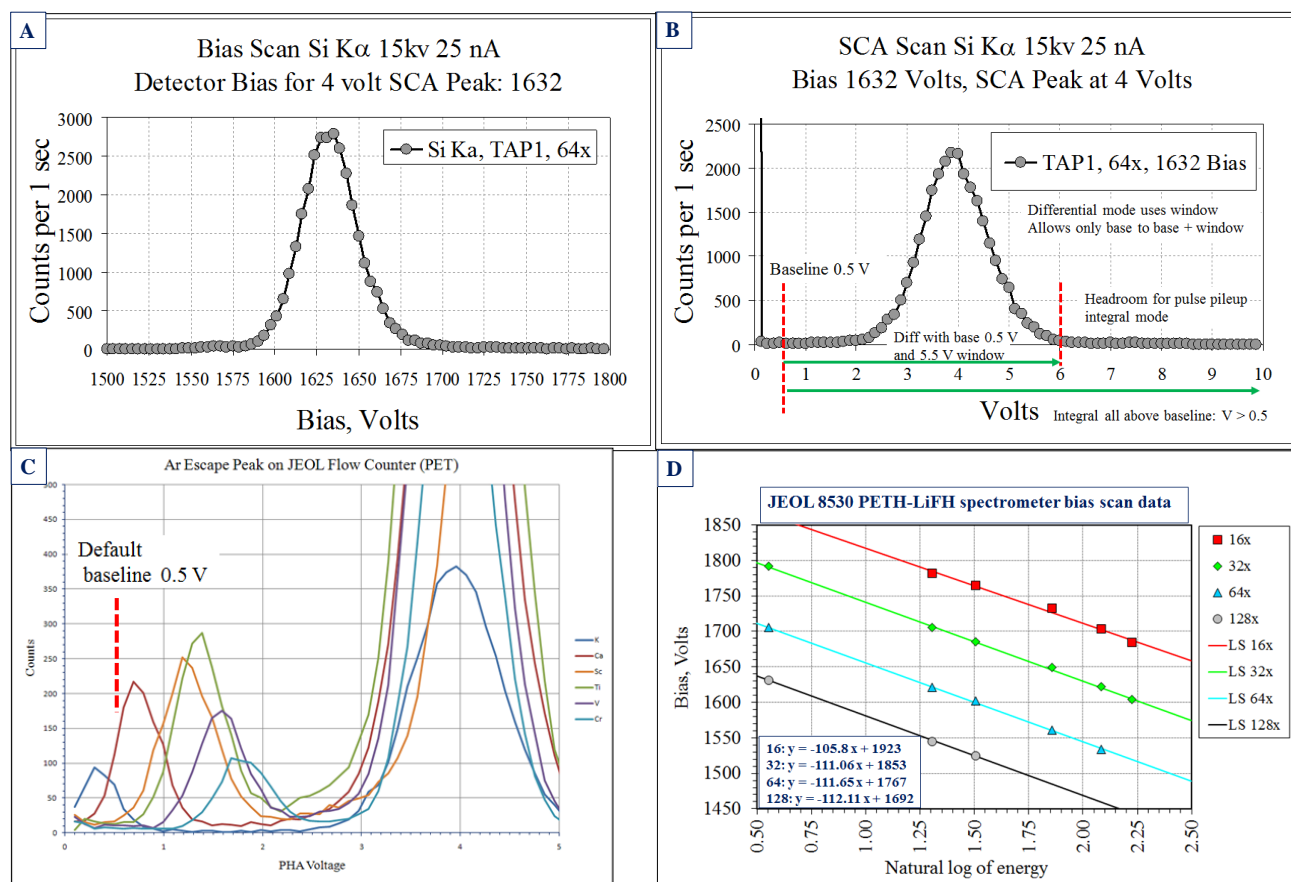
Figure 1 A. Bias scan for Si Kα using TAP crystal and PHA set to 4 V baseline with 0.2 V window, bias peak at 1632V. **B**. SCA scan at bias value of 1632 V. Baseline placement allows for pulse shift due to count rate and change in P-10 gas. Use of integral setting allows headroom for pulse pileup at high count rates. **C**. SCA scans for K-alpha lines of K, Ca, Ti, V, and Cr on PET with flow-proportional detector showing Ar escape peaks and necessity of baseline setting on per-element basis. **D**. Plot of ln(energy) vs. bias for data collected on JEOL spectrometer with sealed Xe detector and PETH-LiFH crystals, illustrating linear relationship and internal consistency of bias data. Least squares fit equations LS are used to calculate bias for any X-ray energy on this spectrometer.