



High Voltage Calibration on MOXI™ Small, Compact X-ray Sources

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Moxtek is a manufacturer of x-ray sources for both handheld and bench-top XRF instruments which have many desired functional characteristics. One desired characteristic for the x-ray sources is to be spectroscopically identical between units. Spectroscopically identical x-ray sources allow instrument manufacturers to use a standard calibration and also allows direct switching of an x-ray source in an instrument with no additional effort. Each x-ray source will give you the same XRF spectra with minimal or no calibration of the instrument. Handheld XRF manufacturers can now reduce the manufacturing complexity in the calibration and in-field service time.

One major obstacle in making sources spectroscopically identical is having tight control over the x-ray source voltage. Moxtek has a proven solution with the digitally controlled MOXI™ x-ray source. The MOXI™ x-ray sources are spectroscopically identical in several XRF applications. Each digital x-ray source is calibrated to within ± 250 V over all settings between 5 to 50 kV, which allows for XRF equivalence.

First we will outline how the new digital sources are high voltage calibrated and second we will show how this high voltage calibration leads to a spectroscopically identical x-ray source.

High Voltage Calibration of the MOXI™ X-ray Source

In order to calibrate an x-ray source two basic requirements are needed:

- 1) a meaningful and accurate way to measure the high voltage
- 2) a way for each x-ray source to store the high voltage calibration information, thereby offsetting source-to-source differences caused by variance in electrical components which make up the source

The x-ray source's high voltage is measured by collecting a spectrum directly from the tube's anode. The source is pointed towards an energy dispersive detector with a pinhole in-between to limit the intensity from the source to a level that that detector can handle. The output spectrum's bremsstrahlung edge is fit with a proprietary algorithm to determine the value of the



bremsstrahlung edge in keV. This measurement provides the spectroscopic voltage in keV, while the electronic monitors on the source give an electronically measured voltage in kV (Figure 1). The difference between these two values is used to calculate a correction factor to improve the voltage accuracy.

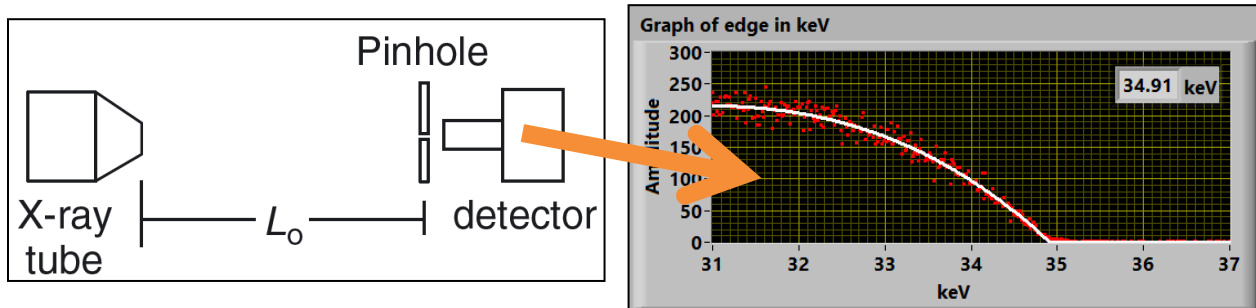


Figure 1. The simple schematic on the left shows the set-up used to measure the bremsstrahlung edge from an X-ray source. The graph on the right shows a spectrum measurement zoomed in on the bremsstrahlung edge. In this case, the high voltage calibrated source was set to 35 kV and the bremsstrahlung edge was measured to be at 34.9 keV; giving a small 0.1 kV offset between the two values.

The x-ray sources are meant to be operated over a range from 5 to 50 kV, therefore spectra are taken at several tube voltages. The bremsstrahlung edge is recorded over several tube voltage settings, generating a keV to kV offset for each voltage setting as shown in Figure 2. In Figure 2 the red line shows the offset between the spectroscopically measured bremsstrahlung edge in keV and the pre-calibrated tube's set voltage in kV. One key observation from Figure 2 is the pre-calibrated offset is non-linear, meaning a simple calibration is impossible. This non-linearity is due to variance in the high voltage power supply board components which do not have a linear response.

Because simple analog x-ray sources do not have any onboard memory, the aggregate non-linear variance in the electronic components sets the high voltage offset of any particular x-ray source. This represents the lower limit in high voltage accuracy for an analog source. The MOXI™ digital source with onboard memory has digital correction factors stored for each kV setting. Most of the high voltage variance due to analog board component variations is removed by the combination of measuring several bremsstrahlung edge spectrum and storing a correction factor in the source's memory.

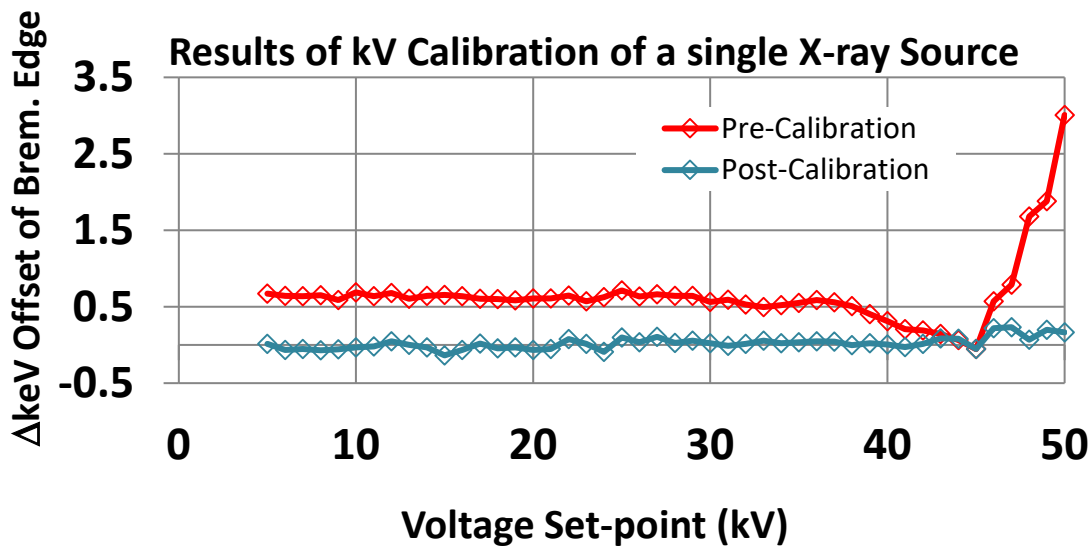


Figure 2. This shows the offset between the tube’s set voltage and bremsstrahlung edge measurements in 1 kV increments between 5 and 50 kV. The red line shows the offset before the x-ray source is high voltage calibrated, and the blue line shows the offset after the x-ray source is high voltage calibrated. Before calibration the offset was close to 600 V, and gets as high as 3 kV at 50 kV. After calibration the source is within 250 V, compared to the bremsstrahlung edge measurements.

In order to gain the benefits manufacturers need, every x-ray source must output the same tube spectra. Figures 3 & 4 show the high voltage offset between the bremsstrahlung edge measurement and the electronic high voltage setting for >100 x-ray sources. It is easily seen that the calibration done on the digital tube greatly reduces the voltage offset variation between multiple x-ray sources, improving both accuracy and precision.

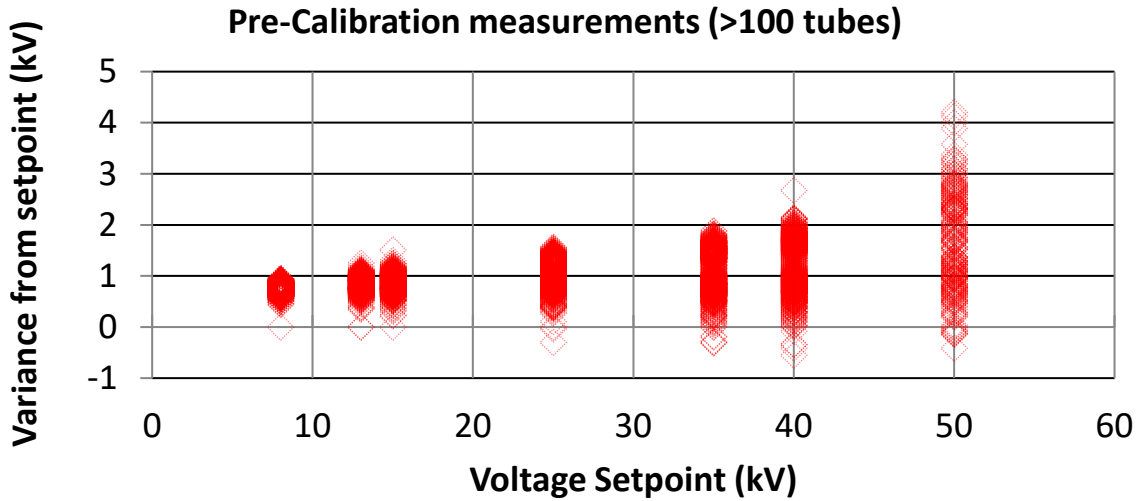


Figure 3. This graphic shows the pre-calibration offset between a tube’s set voltage and bremsstrahlung edge over seven settings, for over 100 separate x-ray sources. This represents the offset to be expected on a strictly analog x-ray source. Before the high voltage calibration, the voltage is typically offset by 0.80 kV, and can vary by up to ± 1.0 kV (@35 kV).

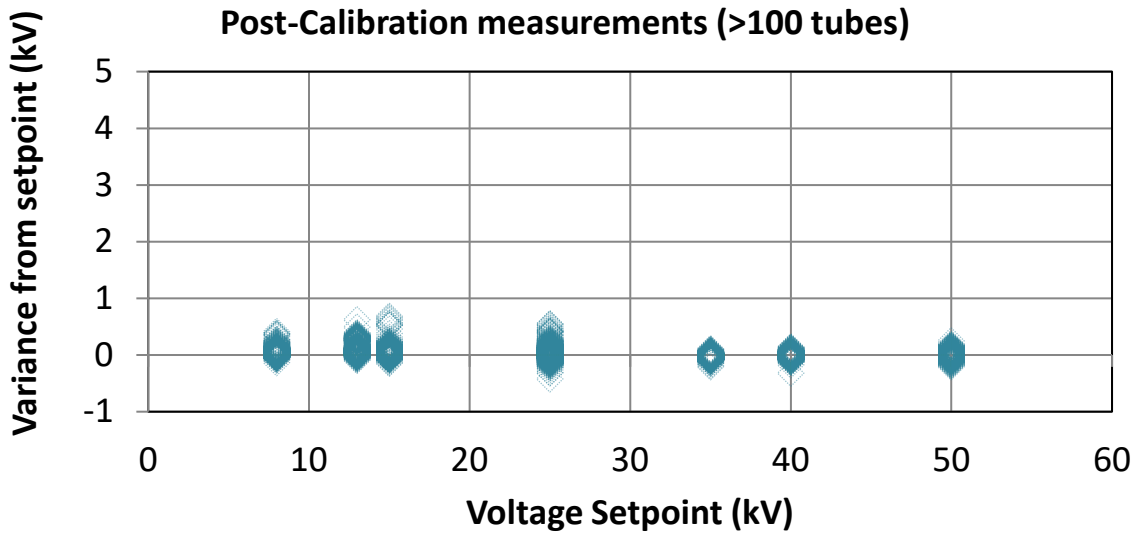


Figure 4. This graphic shows the post-calibration offset between a tube’s set voltage and bremsstrahlung edge over seven settings, for over 100 separate x-ray sources. This represents the offset to be expected on a digitally calibrated x-ray source. After the high voltage calibration, the voltage is typically offset by 0.10 kV, and can vary by up to ± 0.25 kV (@35 kV).

The voltage output of the MOXI™ digital x-ray source is more accurate and precise. The benefit of this improvement has resulted in reduced manufacturing complexity for OEM instrument calibration and service for XRF instruments. Some OEM instrument manufacturers have already adopted this technology and are realizing the benefits.

In the next section we will quantify the benefits of improved voltage accuracy, in terms of the stoichiometry from an XRF instrument.

X-ray Source High Voltage Dependence on XRF Stoichiometry

We performed several experiments exploring how voltage variation on the x-ray source impacts calculated sample stoichiometry. The basic outline of the experiment follows:

- Five spectra were collected from a sample with over 800k counts at a single high voltage setting.
- The spectra were converted into elemental stoichiometry percentages with SinerX fundamental parameters software.
- The kV of the tube was adjusted by a step size ranging from 200 to 500 Volts, depending on the sample, and another five spectra were collected.
- This was done over a range of voltages from 500 to 2000 Volts, depending on the sample.

Two samples were investigated:

- Hastelloy, with the tube nominally set to 35 kV to represent a more difficult scenario for non-aluminum metal alloy sorting. Hastelloy was chosen due to the Ni and Mo peaks being far apart in energy. This large difference in the characteristic x-ray energies (7.4 K α for Ni and 17.4 K α for Mo) maximizes the change in the calculated stoichiometry when the tube's voltage is changed.
- Al 7075, with the tube nominally set to 12 kV, to represent a more difficult scenario for aluminum metal alloy sorting. Al 7075 was chosen due to the Al and Zn peaks being far apart in energy. This large difference in the characteristic x-ray energies (1.5 K α for Al and 8.6 K α for Zn) maximizes the change in the calculated stoichiometry when the tube's voltage is changed.

From the data collected, the effects of changing the high voltage on the x-ray source on the stoichiometry was quantified. As expected, when the voltage was changed the number of counts in the peaks nearest to the bremsstrahlung edge changed the most (Mo for the Hastelloy and Zn for the Al 7075). The calculated Mo stoichiometry percentage was considerably more sensitive



to tube voltage changes than the Ni percentage in the Hastelloy, and likewise with the Zn compared to the Al in the Al 7075 sample.

When the Mo & Ni (or Al & Zn) calculated stoichiometry from an XRF spectra stays constant from an XRF measurement, then we expect spectral XRF-equivalence from the x-ray source. The question becomes what variance in x-ray source high voltage keeps the Mo & Ni (or Al & Zn) calculated stoichiometry small enough for XRF-equivalence? For our purposes we have defined XRF-equivalence as keeping major constituents within $\pm 1/4\%$. This $\pm 1/4\%$ internal standard was generated from our experience working with OEM manufacturers. Figure 5 shows the results for the stoichiometry of the Mo changing as a function of the tube's high voltage for the Hastelloy sample at 35 kV, and likewise Figure 6 for the Zn lines for the Al 7075 sample at 12 kV. From these graphs it is fairly easy to see the maximum voltage range the x-ray source needs to hold in order for the calculated stoichiometry to stay within $\pm 1/4\%$.

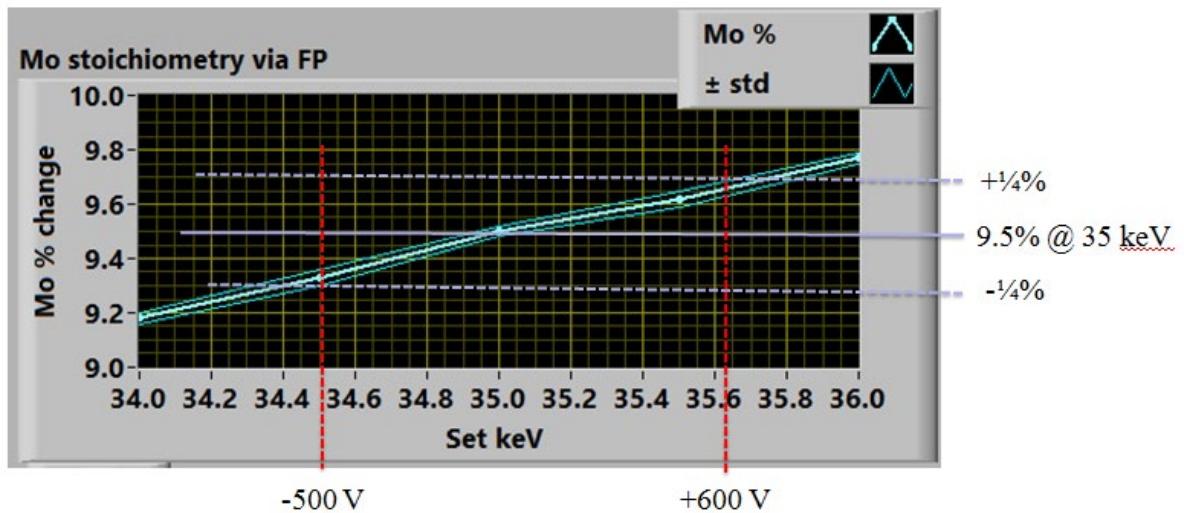


Figure 5. This graphic shows the stoichiometry change of the Mo within the Hastelloy sample as the high voltage of the tube is changed. The standard deviation of the measurements is represented by the thin lines just above and below the main center line. Holding the Mo peak stoichiometry to under $\pm 1/4\%$ deviation requires a high voltage accuracy on the order of ± 500 V. The Moxi™ tube high voltage calibration is under ± 250 V at 35 kV.

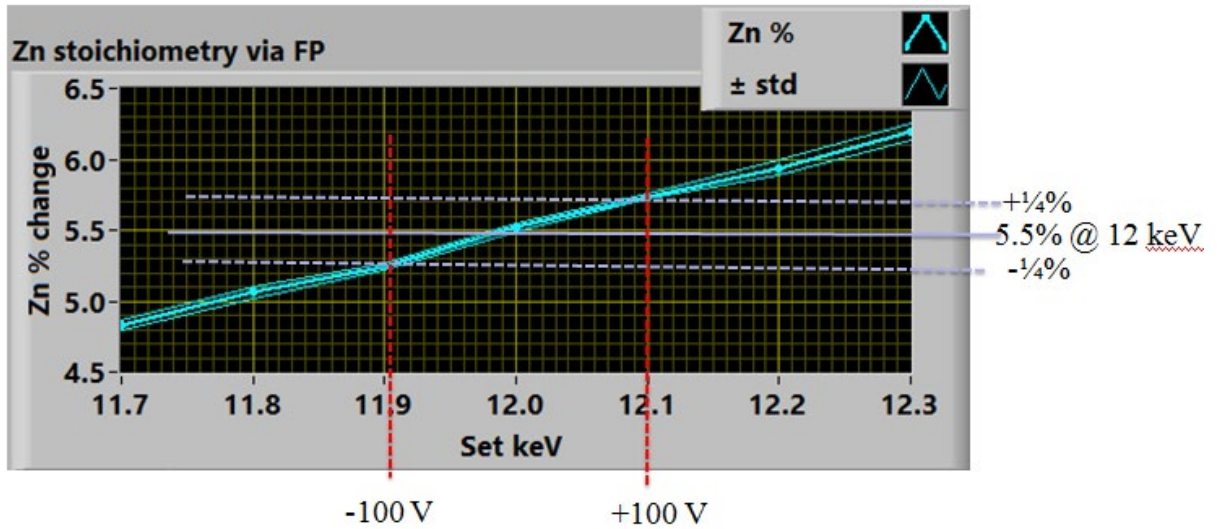


Figure 6. This graphic shows the stoichiometry change of the Zn within the Al 7075 sample as the high voltage of the tube is changed. Holding the Zn peak stoichiometry to under $\pm 1/4\%$ deviation requires a high voltage accuracy on the order of ± 100 V. The Moxi™ tube high voltage calibration is under ± 250 V at 12 kV.

Non-aluminum metal sorting sources could have as much as a ± 500 V variance in high voltage and still produce XRF-equivalent stoichiometry. The test case with the Hastelloy sample is a more demanding sample for this application, therefore with most other typical samples the results will tend to be even better. This test case can be extended to other XRF methods, such as ppm detection for ROHS/WEEE, in which the x-ray source is at or above 35 kV. For these XRF techniques, keeping the voltage of the source to ± 250 V should keep the measurements very close to equivalent.

Aluminum metal sorting sources could have as much as a ± 100 V variance in high voltage and still produce XRF-equivalent stoichiometry. The test case with the Al 7075 sample is a more demanding sample for this application, therefore with most other typical samples the results will tend to be even better. For low voltage XRF techniques, keeping the voltage of the source to ± 250 V is not enough to keep the stoichiometry to within $1/4\%$, but it is almost enough to keep it within $1/2\%$. Although not yet meeting our definition of XRF-equivalency, this level of improvement is a significant improvement when compared to an analog x-ray source counterpart. Work will continue on this front as we strive for a tighter tolerance at lower kV settings.

In Figure 7 we have re-presented the variance of over 100 digitally calibrated sources from Figure 4 with added red bars illustrating the range of voltages required for XRF-equivalency, keeping the stoichiometry under $\frac{1}{4}$ %. From this we can see that for higher voltage XRF applications the MOXI™ sources are well within the voltage tolerance needed to keep the voltage variance low enough to be XRF-equivalent. For the more demanding light element XRF applications at low kV, we are not yet controlled to a level we would consider XRF-equivalent, but we are much closer to this ideal with the MOXI™ digitally calibrated x-ray sources than without it.

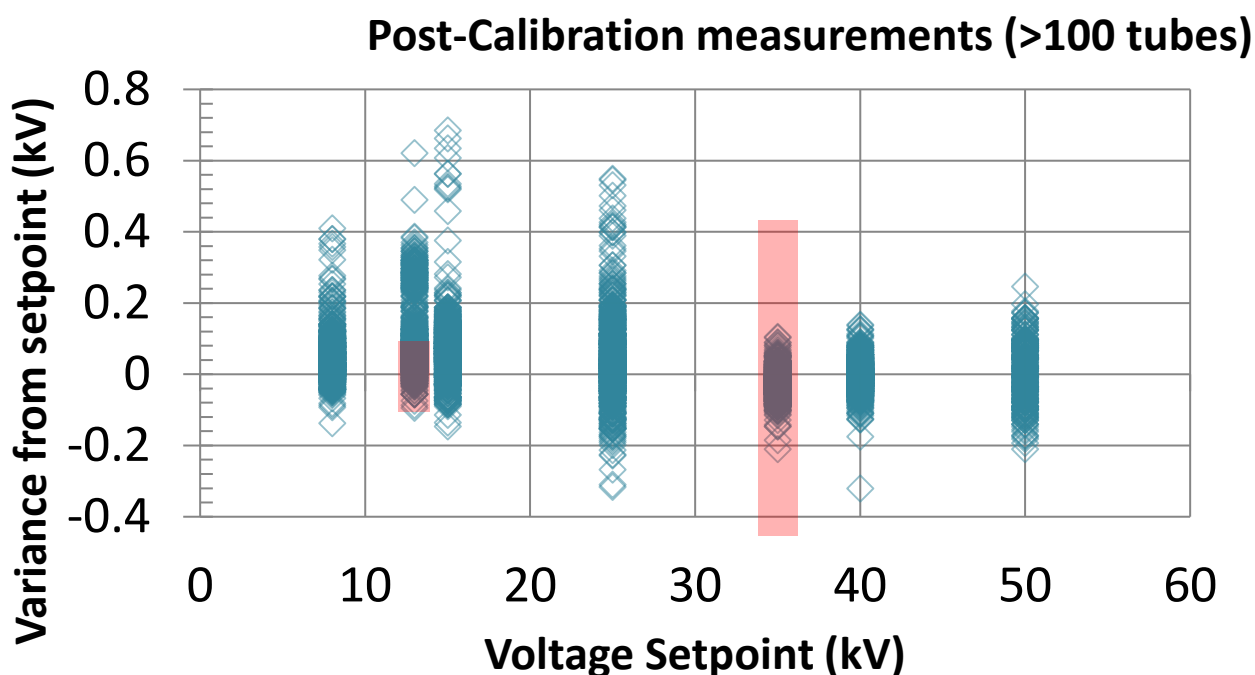


Figure 7. This graphic shows the post-calibration offset between a tube’s set voltage and bremsstrahlung edge over seven settings, for over 100 separate x-ray sources. The red bars show the high voltage accuracy needed to keep calculated stoichiometry from XRF spectra under $\pm\frac{1}{4}$ % for two different experimental set-ups – aluminum metal testing at 12 keV and non-aluminum metal sorting at 35 keV.

Conclusion

The MOXI™ x-ray source is an XRF equivalent x-ray source, available to the market. The key aspect for achieving spectral equivalence is high voltage calibration. The high voltage calibration is achieved through bremsstrahlung edge measurements and storing high voltage offset information on each source. The offset correction improves the high voltage accuracy and precision. This benefit is needed for XRF instrument manufacturers in order to reduce complexity in calibration and service. We have presented a case from two different XRF samples which show how much the stoichiometry changes as a function of the x-ray source's high voltage output. As additional validation of the high voltage calibration benefits, some OEM instrument manufacturers have already adopted this technology and are realizing the benefits.

