How to do Energy Calibration for XRS-FP2

Note: This guide is for XRS-FP2 calibration using Amptek DPPs with Firmware v.6

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DOCUMENT CHANGE LOG

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1 XRF ANALYSIS OVERVIEW

Before the XRF system can be used for routine quantitative analysis it must be calibrated. There are two main parts to this calibration.

1. The first is the spectrometer energy calibration, which is described here. The spectrometer calibration ensures that the x-ray peaks are found in the correct locations with respect to the energy scales used by XRS-FP2. The standard energy scales used by XRS-FP2 are 5, 10, 20, 40, 80 and 160 eV per Channel.
2. The second is the FP calibration of the individual elemental sensitivities, also known as calibration coefficients, which is described a separate document (see XRS-FP2 User Manual). Note that this step may be ignored if you use standardless analysis or an empirical calibration method.

Prior to these steps, of course, the analyst should choose the optimum experimental conditions, and this involves detailed knowledge of the XRF analytical technique. This particular discussion is beyond the scope of this guide.

2 SPECTROMETER ENERGY CALIBRATION

The spectrometer calibration refers primarily to the adjustment of the electronics (Analog Amplifier, ADC, MCA, or DPP) so that all peaks across a spectrum are all located at their appropriate energies. If the peak energy scale is not set correctly, the spectrum processing cannot properly convert the x-ray peaks to elemental intensities. Therefore, incorrect values will be generated, resulting in incorrect assay analyses.

The objective of this document is to show how to first calibrate the hardware to give energy scales close to 10, 20, 40, etc., and then perform a secondary software-only calibration with the XRS-FP2 software. The XRS-FP2 software can *only* process spectra that have a zero offset and an integer gain value (i.e., 5, 10, 20, 40, 80 or 160 eV/channel). The XRS-FP2 software will automatically perform an adjustment of the real spectrum to this format using calibrated values of the actual gain and offset, which must both accurately known. This last step is performed on a routine basis, having calibrated the energy scale correctly, using the so-called "Adjust Spectrum" procedure, which redistributes the channel counts to a spectrum with the required zero offset and integer gain (eV/channel).

For the software calibration it is recommended that you use a known sample having **2** well-separated and well-defined Kα peaks to calibrate these factors (i.e., gain and offset). The Kα peaks are preferred to the Lα peaks because the peaks are more symmetric and it is easier to define a centroid position. The same gain setting must be used for both the standards and the unknown sample assay analyses. Note that this method is only meant to make relatively minor changes to the spectrum, of the order of a maximum of 20% change in the gain and offset values.

*Please refer to the appropriate manual from Amptek for installation of the hardware and driver software required for the USB connection. The initial setup parameters should be selected using Amptek's software, which should then be used to save these parameters in a configuration file. For the Amptek DP5 and PX5 with FW6 the file types are *.TXT. The default file names are DP5.TXT and PX5.TXT, respectively. This configuration file is automatically loaded by the XRS-FP2 software when a DPP connection is made, and it must be located in the same folder as the XRS-FP2 software, as it will be accessed by the latter for setup and control of the DPP module.

You should use Amptek's setup software (DppMCA for the FW6 electronics) for the appropriate detector type (Si pin diodes and drift detectors are very different). You can also setup the configuration with the XRS-FP2 software itself, but you need to be very familiar with the settings in your specific DPP type.

3 THE QUICK GUIDE TO CALIBRATION USING XRS-FP2

The procedure described here assumes that you are going to use a typical setup of 2048 channels, with 20 eV per channel and a Range of 0 to 40 keV. Before starting to calibrate the energy scale, you must setup the detector control parameters. This document does not discuss this procedure. Please refer to the Amptek manual for more information. Also you must choose the number of channels and the gain appropriate to your target spectral range and channel resolution. This guide assumes you have selected an MCA size of 2048 channels, and chosen a coarse gain that provides approximately 20 eV per channel before starting with the actual detailed fine calibration in the hardware setup described here.

Before acquiring any spectra you must have also tuned the fast and slow thresholds. This is done in the "Tune Slow/Fast Threshold" dialog in DppMCA (see Figure 1 below). The purpose of this step is to ensure that the DPP can acquire spectra satisfactorily before starting the calibration procedure. This is shown in Figure 1 below:

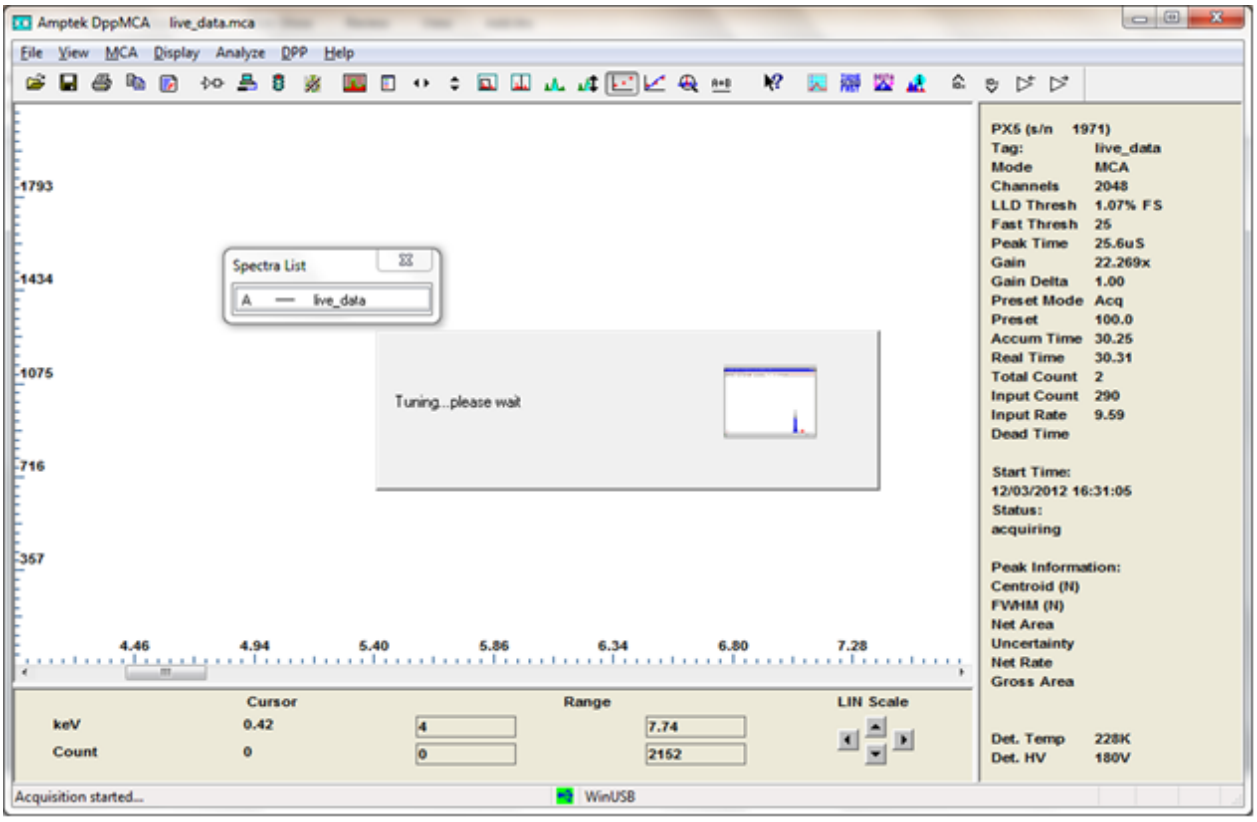

Figure 1. Amptek's DppMCA Software – Slow/Fast Threshold. Select "DPP -> Tune Slow/Fast Threshold".

The following steps describe the calibration process for XRS-FP2 using Amptek hardware:

a) In Amptek's DppMCA software select the *range* and *scale* that you would like to use for analysis (note: this is done **before** running XRS-FP2 as these programs should not be running

simultaneously). To do this in DppMCA, go to “Analyze->Calibrate”. This will display the dialog shown in Figure 2, part A. Change the units from “Channel” to “keV” to allow the spectrum to be displayed in keV. Then set the scale by “Adding” the appropriate channel and energy values (see Figure 2 below). A common scale would be set to 20 eV/ch, as assumed in this document. In DppMCA this is done by labeling channel 0 as 0 keV, and channel 1000 as 20 keV or whatever target is appropriate (see Figure 2 below). For instance, with 2048 channels this means setting a range of 0 to about 40 keV (at 20 eV/ch), which is the most common setup unless you are analyzing lines above 40 keV. Note that this is a fictitious “calibration” at this point. After selecting the appropriate range and scale for your application, click “OK” to move onto step (B) below.

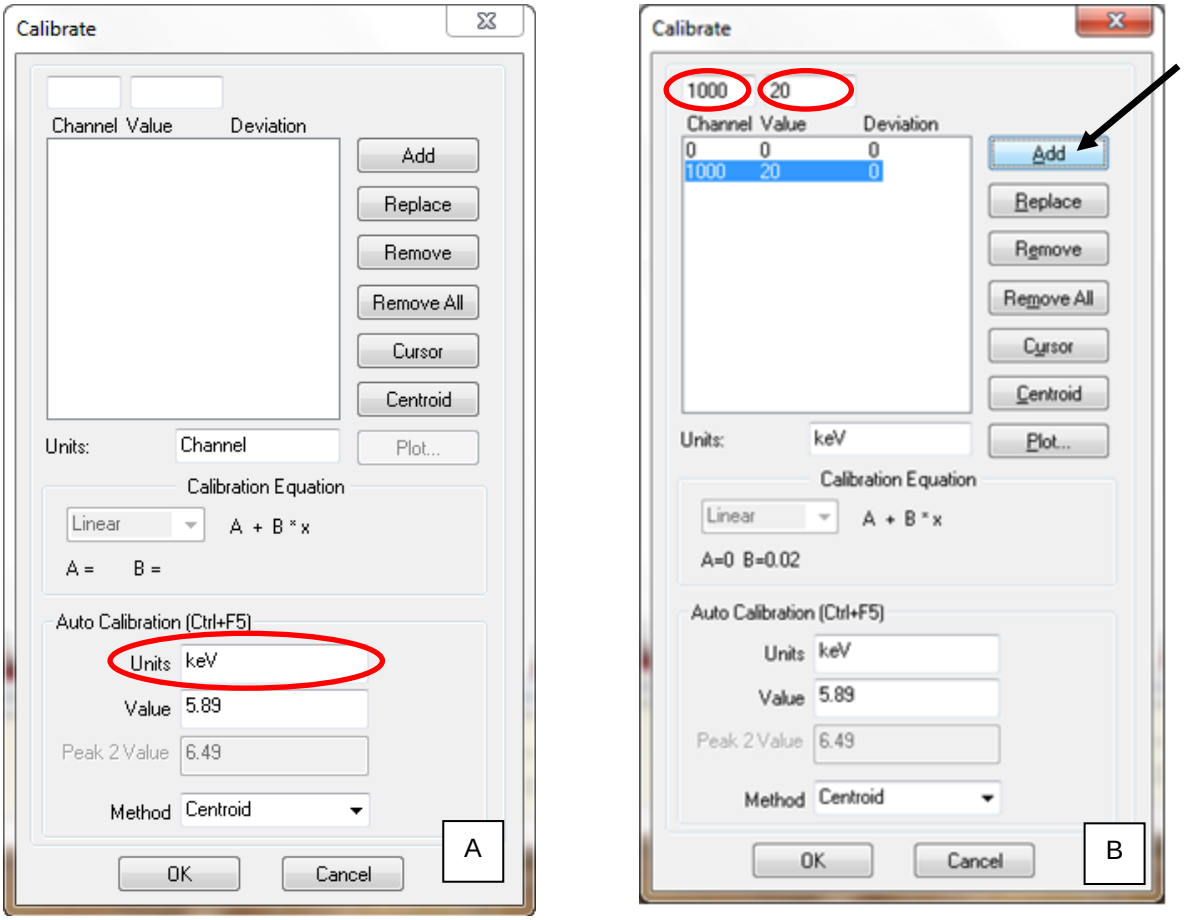

Figure 2. Amptek’s DppMCA Software – Calibrate Dialog. Select the “Units” edit box and change from “Channel” (A) to “keV” (B). Then set appropriate “Channel” and “Value” parameters by clicking in the boxes in the upper left corner of the dialog. For example, enter “0” in the channel box and “0” in the energy box, and then click “Add”. Next add a second point, such as channel “1000” and “20” keV, followed by clicking the “Add” button. This will set the energy range and scale (as shown in the list box in (B)).

b) Select “Acquire” in DppMCA and acquire a spectrum.

c) Still in the Amptek software (DppMCA) go to the *Gain* settings in "MCA -> Acquisition Setup -> Gain" (see Figure 3 below). Use the fine and course Gain to set the peak locations such that the peak centroid for each of the 2 well-separated and well-defined Kα peaks used for calibration are as closely aligned to the known line energies as possible. The Amptek Gain value (mostly the Coarse but also the Fine control) must be set so that the peaks fall at the right energies. Note that this subtly different from the way that the Amptek software normally calibrates in that they only change the X-scale to match the peaks – for correct calibration with XRS-FP2 we need the peaks to match the predefined scale (i.e., when the scale is set to 20 eV/ch, and the offset set to zero, as described in part (A) above). As you change the gain, you may need to return to step (a) above and re-tune the fast and slow thresholds.

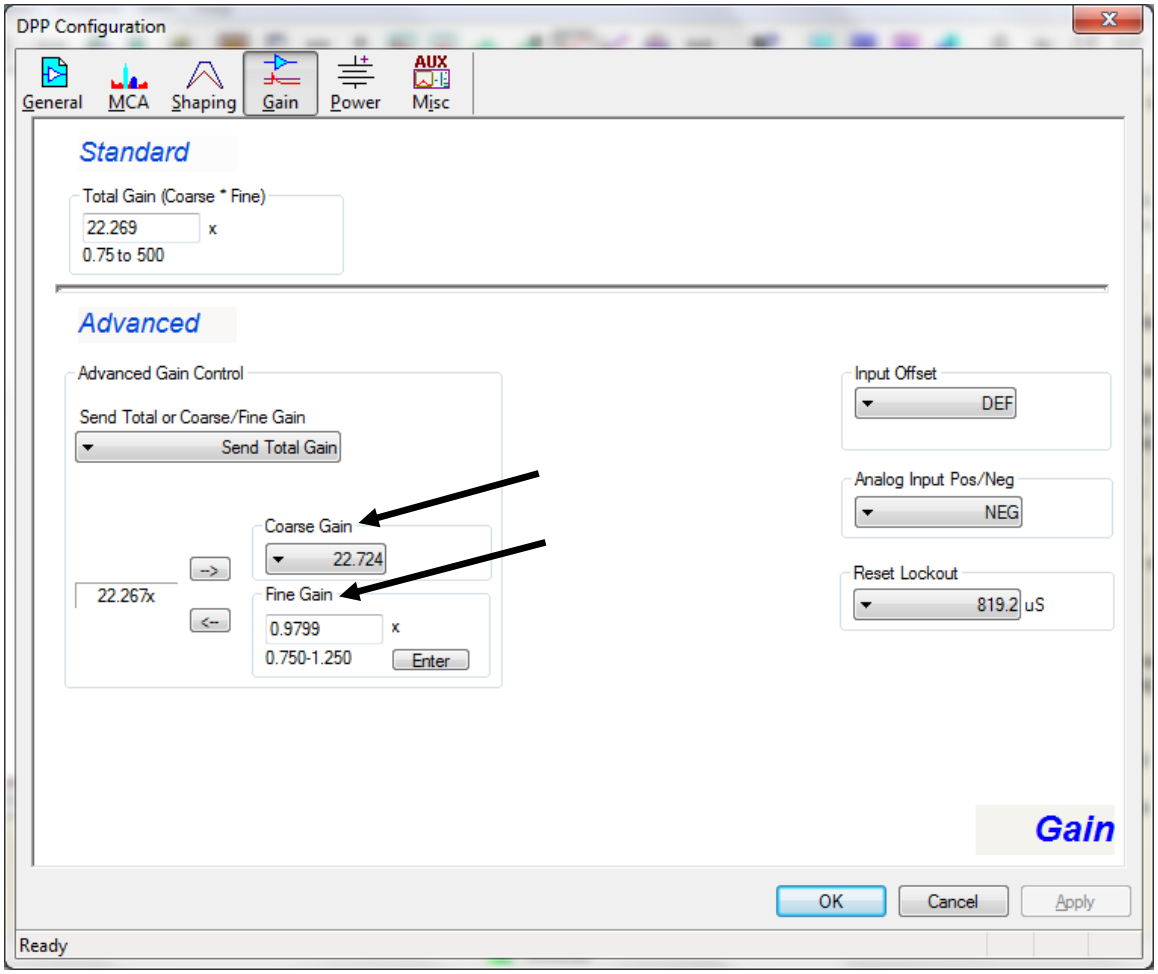

Figure 3. Amptek's DppMCA Software – Gain Panel. DPP Configuration-Gain panel, showing the course and fine gain settings. Note that the threshold settings are located in the DPP Configuration-Shaping panel, should they need adjusting.

d) After suitable adjustment of the gain, the peak centroids should be close to the known energies for those peaks, again using the predefined fixed scale. For example, in Figure 4 below, Mn Kα was used as one of the calibration peaks. Having followed the steps described above, the Mn

peak centroid was located at 5.90 keV, very close to the known Mn Kα value of 5.895 keV (see Figure 4).

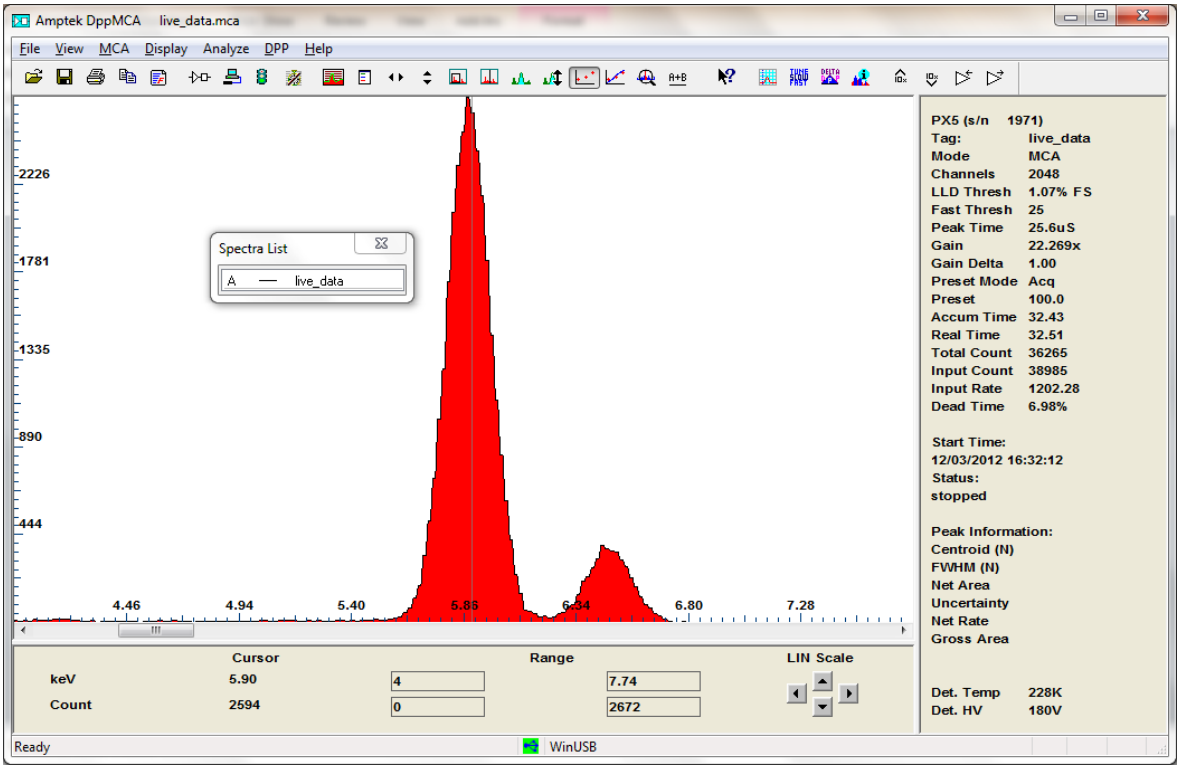

Figure 4. Amptek's DppMCA Software – Spectrum. Well calibrated Mn Kα peak after adjusting the course and fine gains and tuning the slow/fast thresholds. The obtained Mn peak centroid is 5.90 keV, very close to the known Mn Kα value of 5.895 keV.

e) Next save the configuration file in the Amptek software. Using DppMCA and Firmware V.6, you must then select the "Save Configuration File" option. For FW6, name the file as either "DP5.TXT" or "PX5.TXT", depending on the hardware (see Figure 5 below).

It is very important that the filename match the DPP device (e.g., DP5.TXT for a DP5 or X-123 module), because there are different parameters for each of these devices. Also, the XRS-FP2 software will later automatically recognize the type of DPP and then load the appropriate *.TXT configuration file.

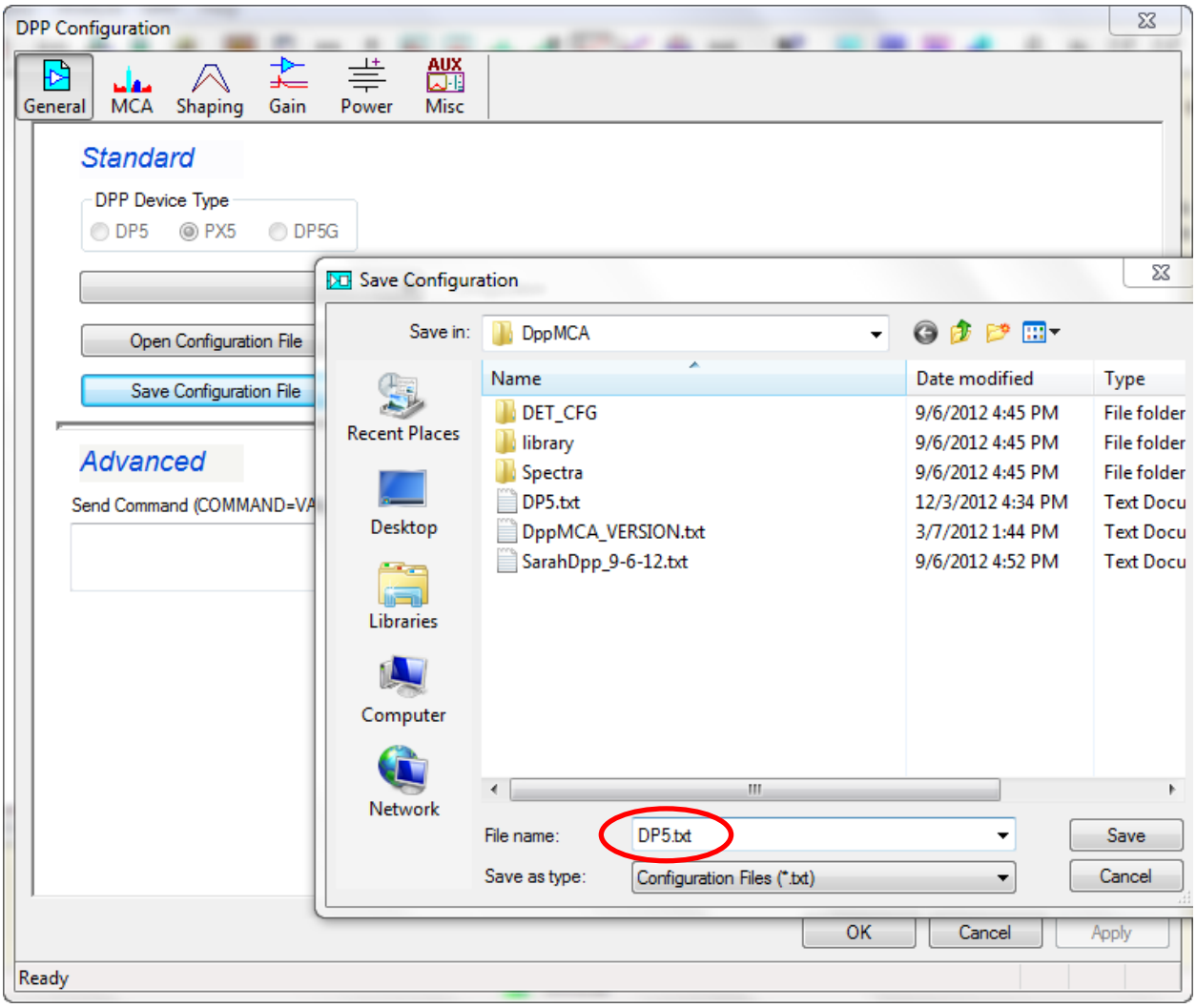

Figure 5. Amptek's DppMCA Software – Save Configuration Dialog. DPP Configuration-Save Configuration File dialog. Save configuration file (e.g. DP5.TXT, etc.) for use with XRS-FP in the steps below.

f) Close the Amptek software at this point. It is important that DppMCA is ***not*** running simultaneously with XRS-FP2, because the DPPs cannot be connected to more than one program.

g) Now copy the configuration file (i.e. DP5.TXT or PX5.TXT) saved above, in step (e), to the XRS-FP2 software folder location (e.g., C:\CrossRoads Scientific\XRS-FP2).

h) Load XRF-FP2, select the **Setup Hardware** button from the top ribbon and then select the **DPP-MCA** button from the L-hand panel. *Before* you connect to the DPP, check that the parameters

eV/ch and the number of channels are the same as the values previously set in the Amptek DppMCA software, by selecting the “MCA” tab in the R-hand panel. See Figure 6 below.

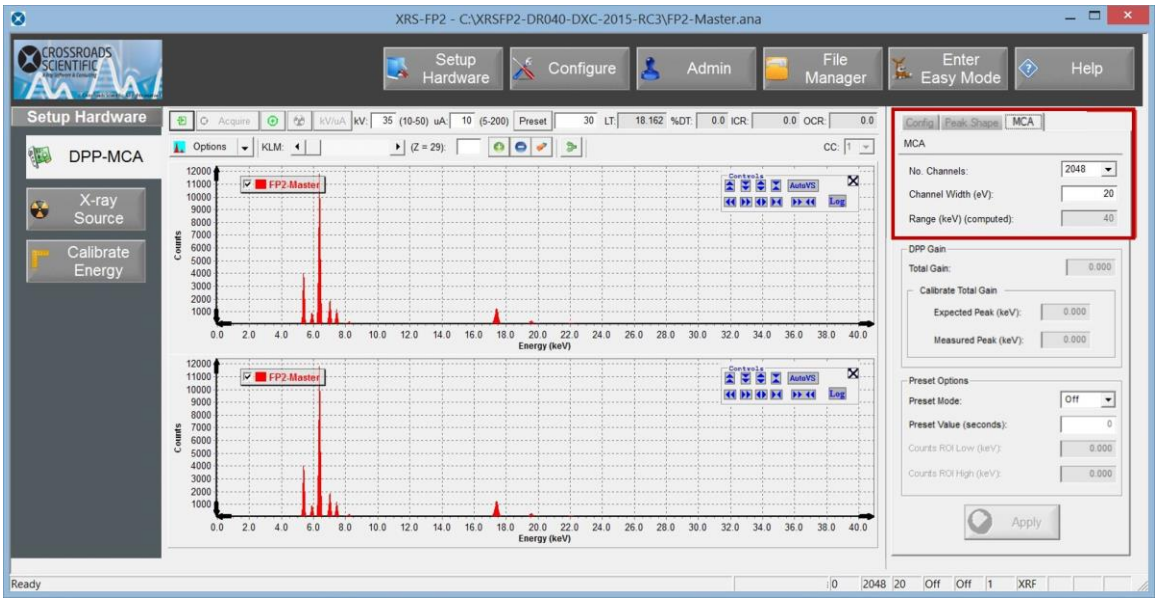

Figure 6. XRS-FP2 Software – Amptek Acquisition Setup Panel. This panel shows the setup values before connecting to the DPP.

i) Note that at this point, all the parameters displayed in this panel have come from the XRS-FP2.INI file, except the Preset Value, which may have come from an ANA file.

Next, go to the “Config” tab in the R-hand panel and click on the **Connect** button to connect to the DPP (Fig. 7). After the connection is made with the DPP (using the USB interface in the example above), the XRS-FP2 software will load, apply and display the configuration file (PX5.txt) that was saved from the Amptek software (as shown in the example in Figure 7 below) and copied to the XRS-FP2 software directory. You should check that the parameters under the “Config” tab are set to the same values (e.g. total gain, number of channels, thresholds, peaking times, etc.) as seen in DppMCA.

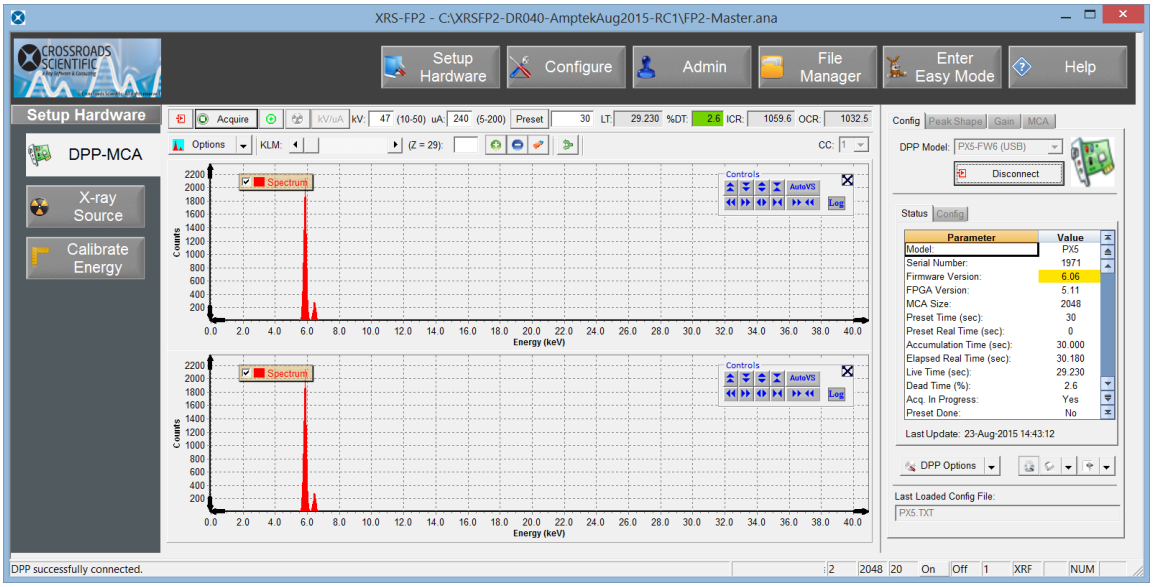

Figure 7. XRS-FP2 Software – Amptek Hardware Configuration Dialog. This panel allows the user to check that all the parameters are correctly set after saving a configuration file in the Amptek software and copying it to the XRS-FP2 folder location.

Also note, that the hardware parameters of the connected DPP are displayed in this panel. Figure 7 above shows that a PX5 type of DPP was connected with firmware version 6 installed. Also shown is the serial number of the DPP, the version of the FPGA firmware, and the path (truncated here) of the location of the loaded configuration file (PX5.txt in this case). Also note that the Preset Value (acquire time) has been changed from 20 to 100 seconds, as this value also came from the configuration files.

Figure 8 below shows the XRS-FP2 Amptek MCA Acquisition Setup panel *after* the connection has been completed. Note that the number of channels is set to 2048, along with the eV/Channel and range, which are set to 20 and 40, respectively. These parameters should match the range and channel width previously set in the Amptek software, and reflects the most common setup for typical XRF analysis (unless you are analyzing lines above 40 keV).

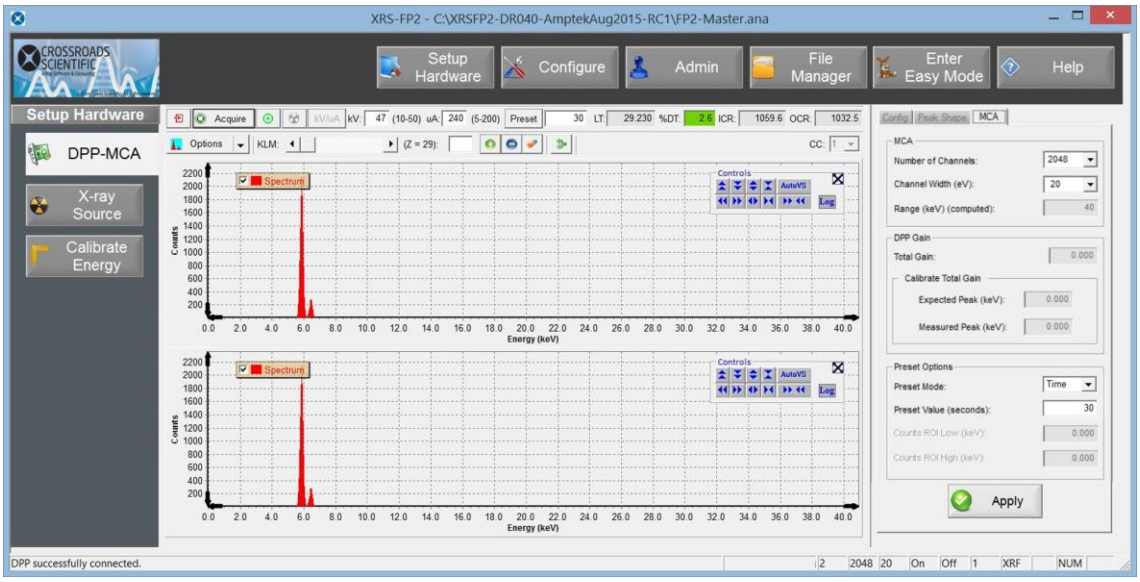

Figure 8. XRS-FP2 Software – DPP-MCA Hardware Configuration Dialog. In the R-hand panel under the "MCA" tab note the number of channels, eV/Channel (width) and the range after connecting to the DPP.

j) Use an Fe-55 radioisotope source or an x-ray tube to generate a spectrum from a known sample. For this step we just need one peak that is not overlapped, so Fe-55 is perfect for this. Click on the **Acquire** button above the spectrum display in XRS-FP2 to collect a spectrum for about 30 seconds. Compare the peak location to the expected location. In the example below (Figure 9) the Mn-Kα,β peaks are shown along with the Mn-K line markers.

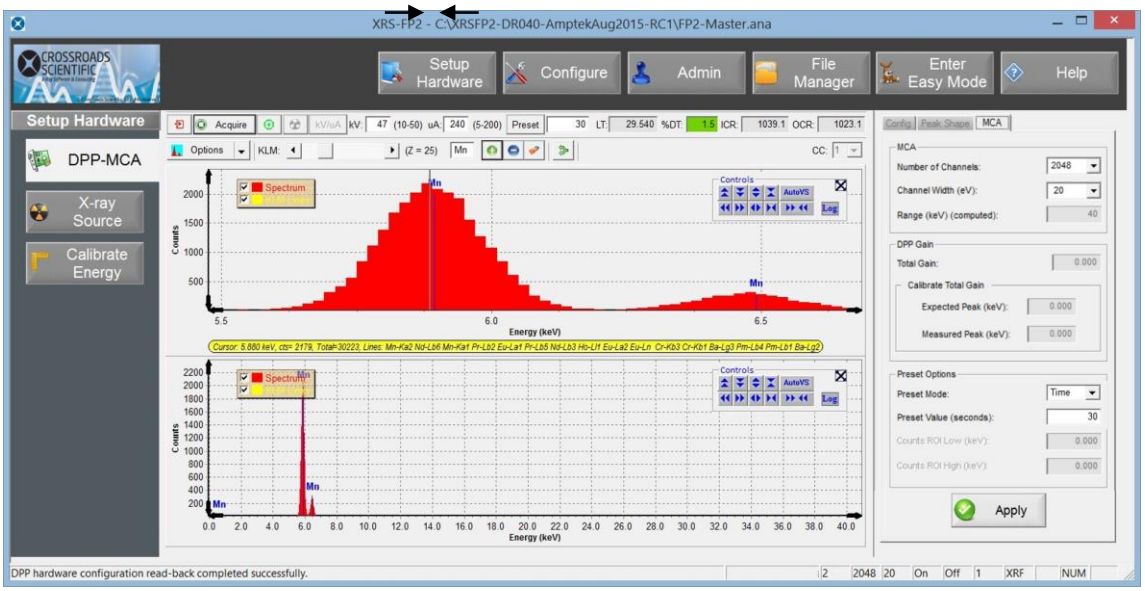

Figure 9. XRS-FP2 Software – Calibrate Energy Workflow. Compare the actual & expected peak locations.

k) Go to the "DPP Gain" section in the R-hand panel. Enter both the expected centroid energy of a known peak (e.g. Mn-Kα) in the spectrum, and the measured value as seen with the cursor in the Spectra-X panel (Figure 10). Then click the **Apply** button, which will update the "Total Gain" value (Figure 10). This will give you an approximate but close starting point for getting calibrated spectra. Note this step is optional in the calibration process but highly recommended.

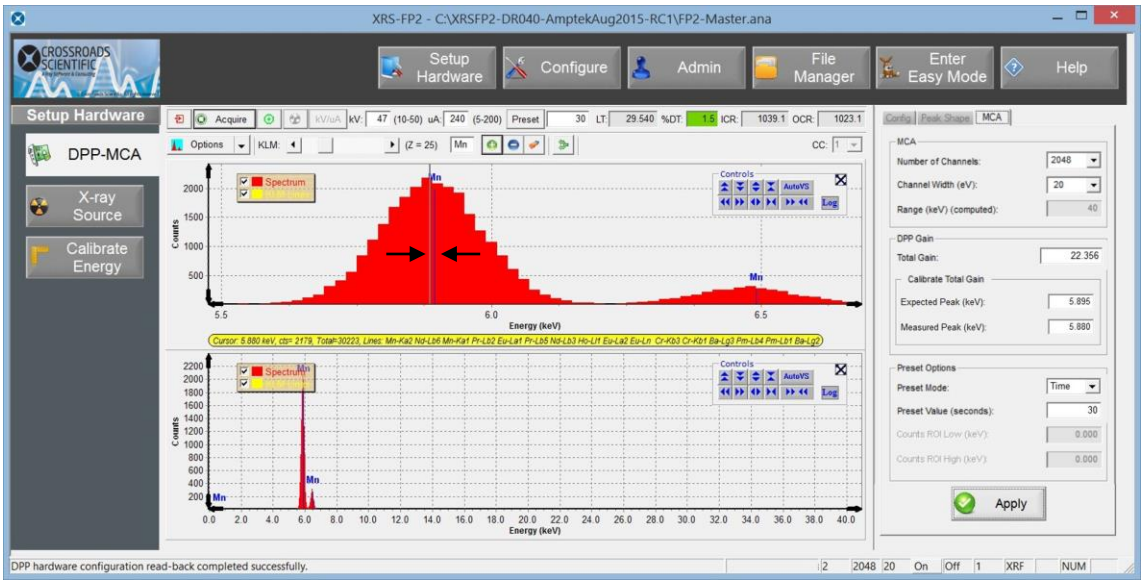

Figure 10. XRS-FP2 Software – DPP-MCA Hardware Configuration Dialog. In the R-hand panel under the "DPP Gain" section in the "MCA" tab enter the expected and measure peak values then click the Calculate button.

Note: This completes the hardware (and firmware) calibration. All necessary parameters are now set in the configuration file that controls the hardware. You are now ready to move onto the software calibration at this point (see steps below).

l) Now, we are ready to start the XRS-FP2 software gain and offset calibration. Select the **X-ray Source** button. Click the Connect button in the R-hand panel to **Connect** to the Mini-X x-ray tube. Then click the **HV On** button and adjust the kV and uA parameters as necessary. Select the **Apply** button to make these changes. See Figure 11 below.

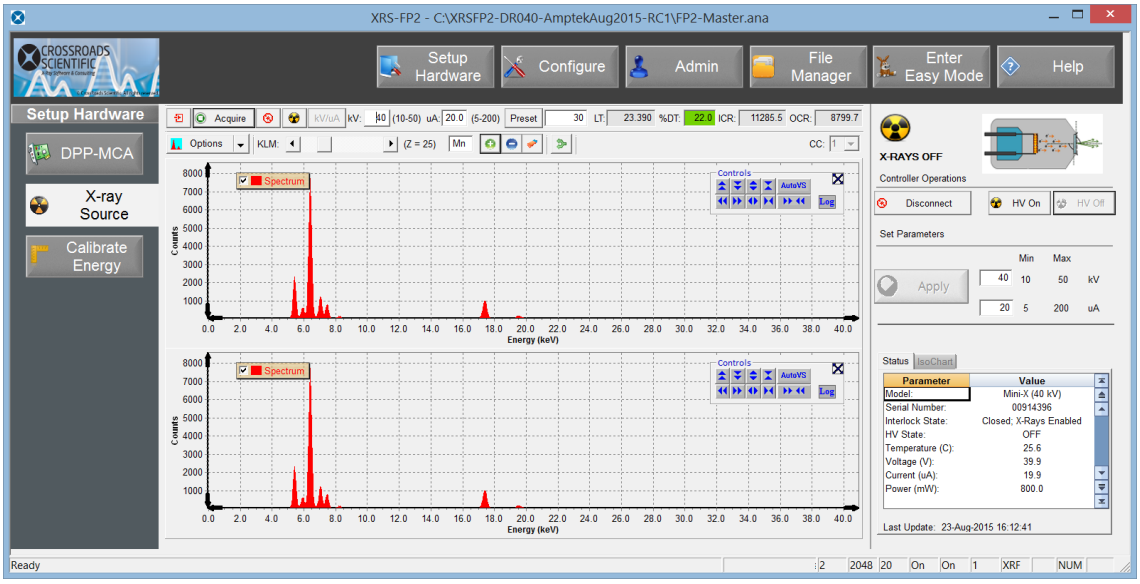

Figure 11. XRS-FP2 Software – X-ray Source Dialog. In the R-hand panel select the connect button to connect to the Mini-X x-ray source and adjust the kV and uA as needed.

m) Select the **Acquire** button to acquire a spectrum from a sample that has two well-separated Kα peaks. It is recommended that you acquire about 100,000 counts for each peak in this 2-point calibration. For example, collect spectra with 2 peaks as shown in Figure 12 below (stainless steel sample in this case). Look at the Spectrum and see if the KLM markers are close to their expected energies. In the example shown below, we see that both the Fe Kα and the Mo Kα peaks are reasonably close to their known centroid values of 6.400 and 17.443 keV, respectively, but not exactly aligned.

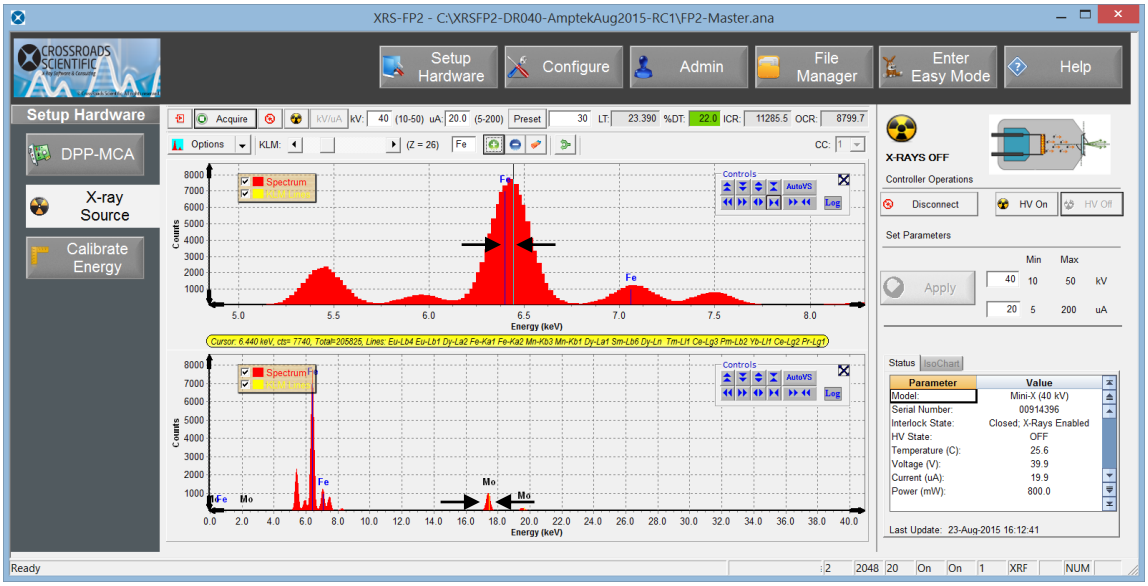

Figure 12. XRS-FP2 Software – X-ray Source Dialog. The measured peaks from the collected spectra are close to the known peak values for Fe and Mo, but not exactly aligned.

n) To improve the energy calibration, select the **Calibrate Energy** button from the L-hand panel and bring up the Calibrate Energy workflow shown below in Figure 13.

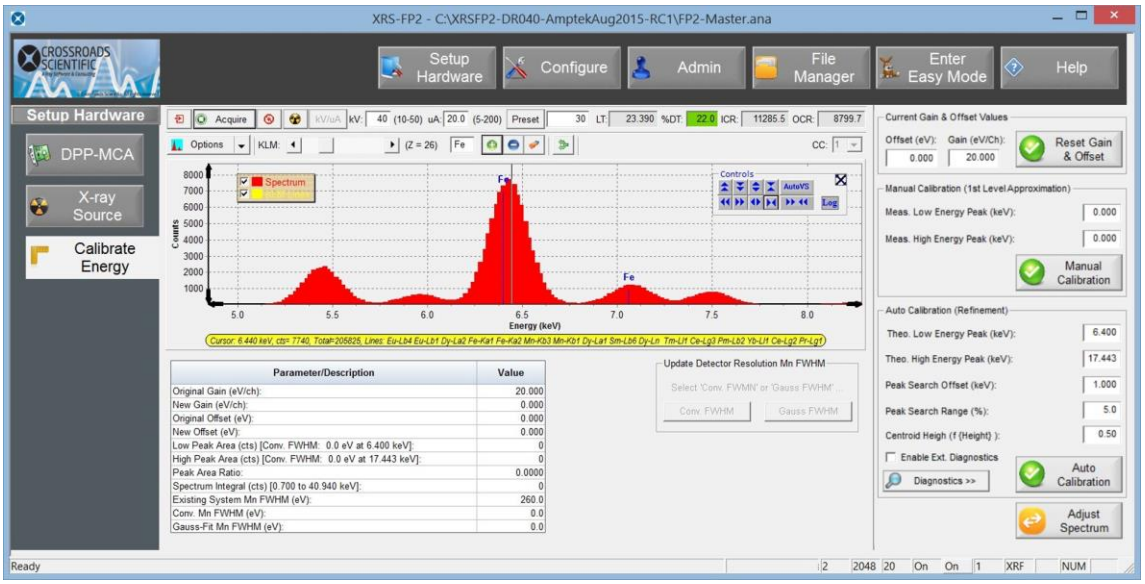

Figure 13. XRS-FP2 Software – Calibrate Energy Workflow. Compare the actual & expected peak locations.

o) Click the **Reset Gain & Offset** button from the R-hand panel. This ensures that any previous XRS-FP software calibration is reset (cleared) to the target values of a zero Offset energy (eV) and Gain (channel width in eV).

p) Next we will do a “Manual Calibration”. Use the cursor to find the centroid for each peak in the collected spectrum. Enter these values as the “Meas. Low Energy Peak” and “Meas. High Energy Peak” values in the “Manual Calibration (1st Level Approximation)” section (see Figure 14 below). In the example here, we typed in 6.420 keV for the Low energy peak (Fe Kα), and 17.420 keV for the High energy peak (Mo Kα). Now click on the **Manual Calibration** button. After this you should see changes in Offset and the Gain values (see Figure 15 below). The Offset has changed from 0 to -54.611 eV, and the Gain from 20.000 to 20.079 eV per channel.

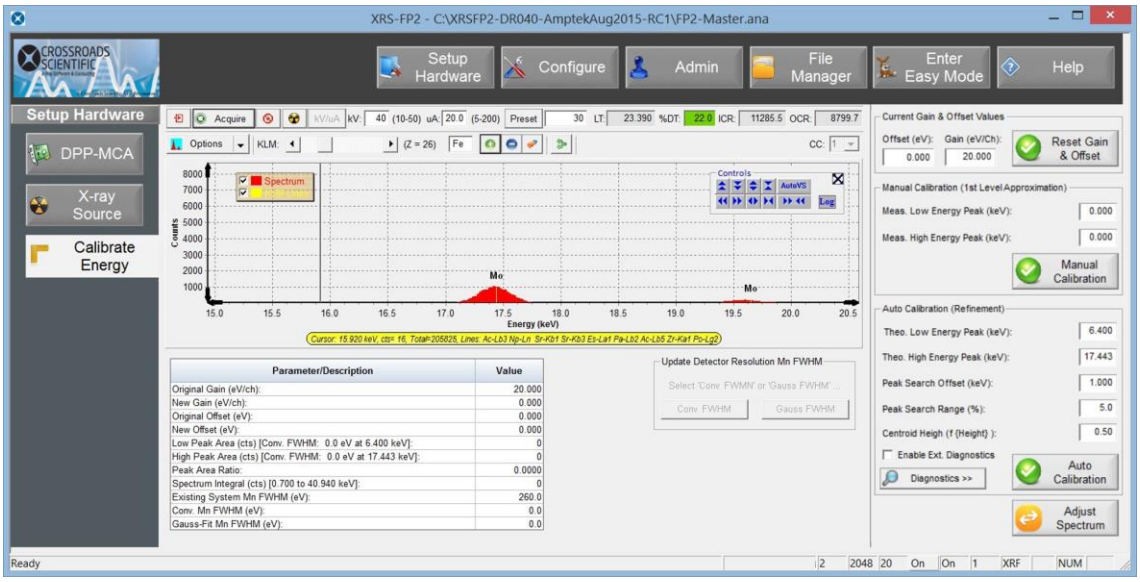

Figure 14. XRS-FP2 Software – Calibrate Energy Workflow. Enter the measured centroid (Kα) values of the Low and High energy peaks used for calibration, i.e. the centroid values for Fe-Kα and Mo-Kα were entered as the Low and High values, respectively.

q) The next step will be to do an automatic energy calibration. Strictly speaking, this step can be done independently of the two manual calibrations done previously (see above). However, it is often the case that the hardware is so far out of calibration that the automatic version cannot find the peaks successfully. It is still necessary to correctly setup the gain and number of channels in the Amptek DPP setup program (DppMCA), otherwise the hardware setup will never match the intended software setup.

r) Before starting the automatic calibration, check the values for the Peak search offset and range. More information on these parameters is contained in the XRS-FP2 Software User Manual. These two parameters control the search range for a "peak." Obviously if the true peak is too far away from the expected location for the search range to find, then an incorrect value will be used and the calibrations values will be of no use. In the displayed figures here the offset is 1.0 keV, which means the search program will search above and below the expected position by a maximum of 1.0 keV. Similarly there is a limit of 5% of the energy scale (peak search range) for each of the two peak energies.

s) Enter the centroid (Kα) values of the Low and High energy peaks used for calibration in the "Auto Calibration (Refinement)" section of the calibrate energy workflow. Now click on the **Auto Calibration** button to perform the automatic calibration step.

For example, in the calibration used here, the centroid values for Fe-Kα and Mo-Kα were entered as the Low and High values, respectively (see Figure 15 below). Note that we are always referring to centroids and not the actual individual line energies. The centroids are the weighted average of the dependent line energies. The auto calibration typically results in a non-zero Offset value; however, the Gain value should not be too different from the target eV/channel. An example is shown in Figure 15 below. Note: that you will see some change in offset and a slight change in the gain at this point

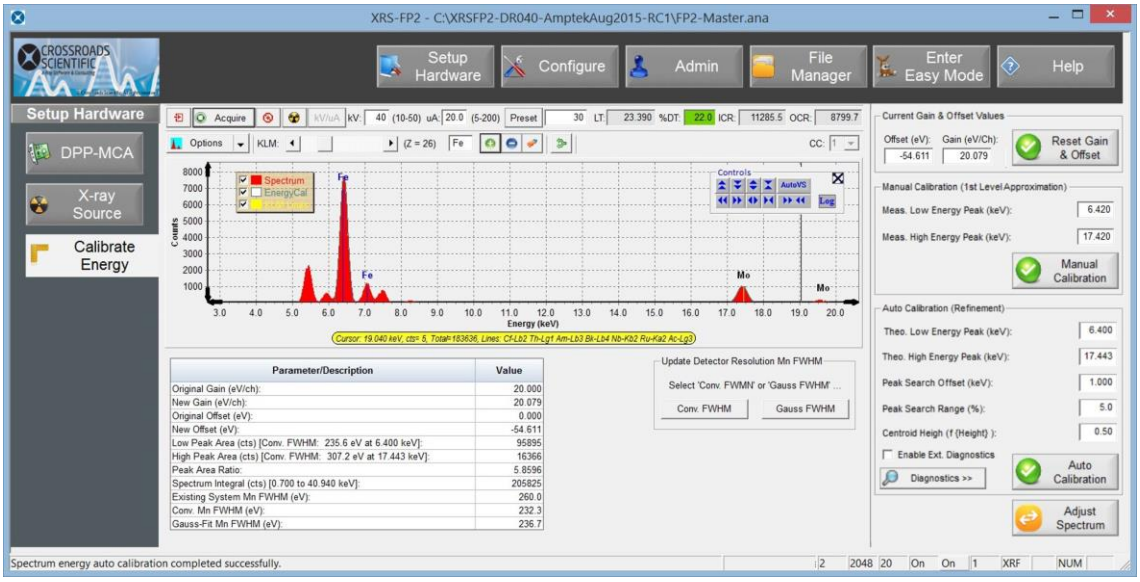

Figure 15. XRS-FP2 Software – Calibrate Energy Workflow. Enter the centroid (Kα) values of the Low and High energy peaks used for calibration in the "Auto Calibration (Refinement)" section, i.e. the centroid values for Fe-Kα and Mo-Kα peaks.

t) Next click on the **Adjust Spectrum** button (Figure 16). This will adjust the displayed spectrum based on the calibrated peak-energy locations. The Adjust function redistributes the channel counts into a spectrum with a zero offset and the required integer gain (eV/channel). At this point take a look at the spectrum to compare the "Adjusted" (calibrated) and "Original" spectra. See Figures 16 below, which show the adjusted and original spectra collected for the example used in this guide (i.e. the adjusted and original peaks for Fe and Mo).

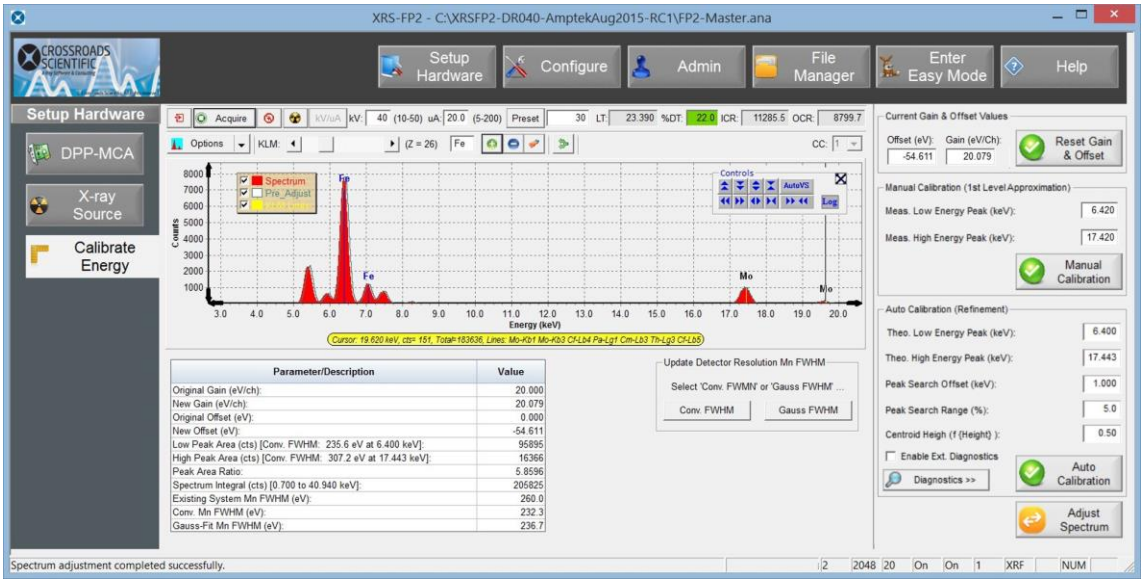

Figure 16. XRS-FP2 Software – Calibrate Energy Workflow. Select the Adjust Spectrum button to display the spectrum based on the calibrated peak-energy locations.

u) Finally go the **Acquisition** button in the Analyze workflow and select the "Auto-Adjust Spectrum" check box in under the "Acquire Options" section in the "Meas. Cons." tab. This will auto adjust spectra on acquire using the previously obtained energy calibration settings. See Figure 17 below.

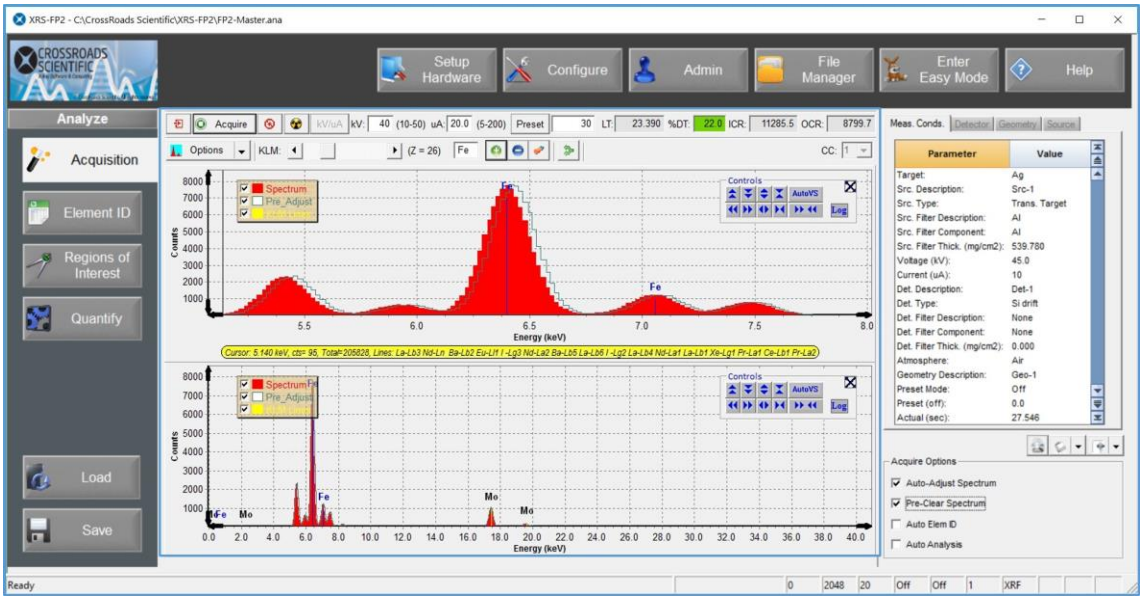

Figure 17. XRS-FP2 Software – Auto-Adjust Spectrum. Select the Auto-Adjust Spectrum option to adjust spectra on acquire using the previously obtained energy calibration settings.

v) Now, "Acquire" a spectrum. You should see that the acquired spectrum is now well calibrated (see Figure 18 below).

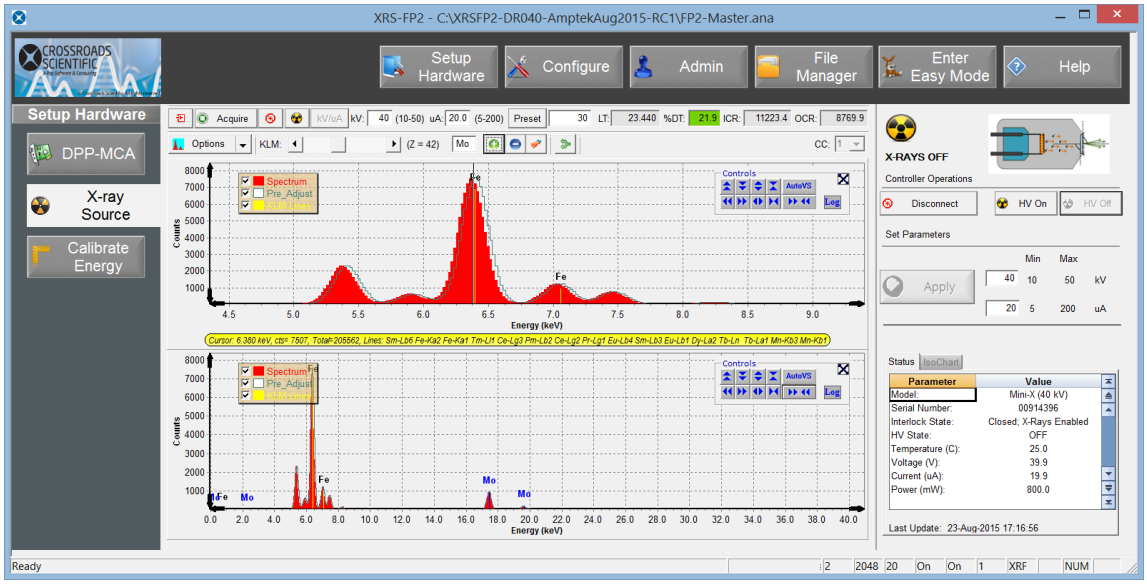

Figure 18. XRS-FP2 Software – X-ray Source, Acquire Workflow. Acquisition of a well-calibrated spectrum having followed all the calibration steps above.

w) This completes the energy calibration procedure. You are now ready to perform XRF analysis using calibrated spectra.