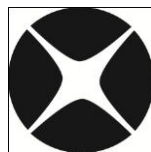


# How to do Energy Calibration for XRS-FP

Note: This guide is for XRS-FP calibration using Amptek DPPs with Firmware v.5 or Firmware v.6



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# TABLE OF CONTENTS

Table of Contents .....	2
Document Change Log .....	3
1 XRF Analysis Overview .....	4
2 Spectrometer Energy Calibration.....	5
3 The Quick Guide to Calibration Using XRS-FP.....	6

## DOCUMENT CHANGE LOG

Date	Person	Pages	Description
29-Nov-12	Sarah Cross	All	Created document for Amptek Hardware (DP5 & PX5, FW5 & FW6)
05-Dec-12	Sarah Cross	Most	Updated document
13-Dec-12	Brian Cross	Many	Revised document
14-Dec-13	Brian Cross	Many	Further revision and updates
16-Dec-13	Sarah Cross	Some	General revision
16-Dec-13	Brian Cross	Some	Adjusted placement of figures

# 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-FP. The standard energy scales used by XRS-FP 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-FP Software Guide v51X). 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-FP software. The XRS-FP 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-FP 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” 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 K $\alpha$  peaks to calibrate these factors (i.e., gain and offset). The K $\alpha$  peaks are preferred to the L $\alpha$  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. In all Amptek DPPs prior to the DP5/PX5 using firmware version 6 (i.e., version 5 or FW5) the files are saved as the \*.CFG file type (such as PX4.CFG). 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-FP software when a DPP connection is made, and it must be located in the same folder as the XRS-FP software, as it will be accessed by the latter for setup and control of the DPP module.

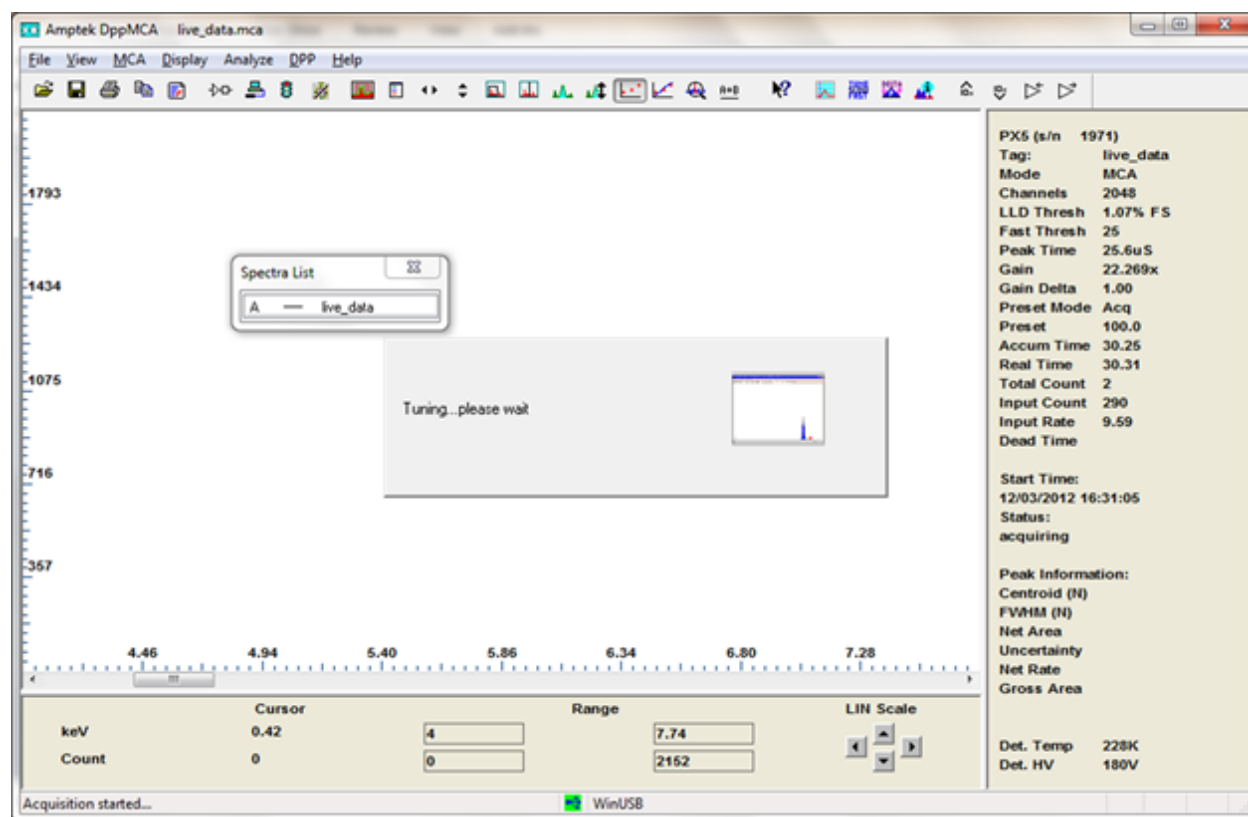
Note that older DP5 electronics using FW5 actually emulate an older DPP called the PX4, and therefore the configuration files should be named as PX4.CFG.

You should use Amptek’s setup software (DppMCA for the FW6 electronics, and ADMCA for FW5) for the appropriate detector type (Si pin diodes and drift detectors are very different). You can also setup the configuration with the XRS-FP 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-FP

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 (or ADMCA) (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:

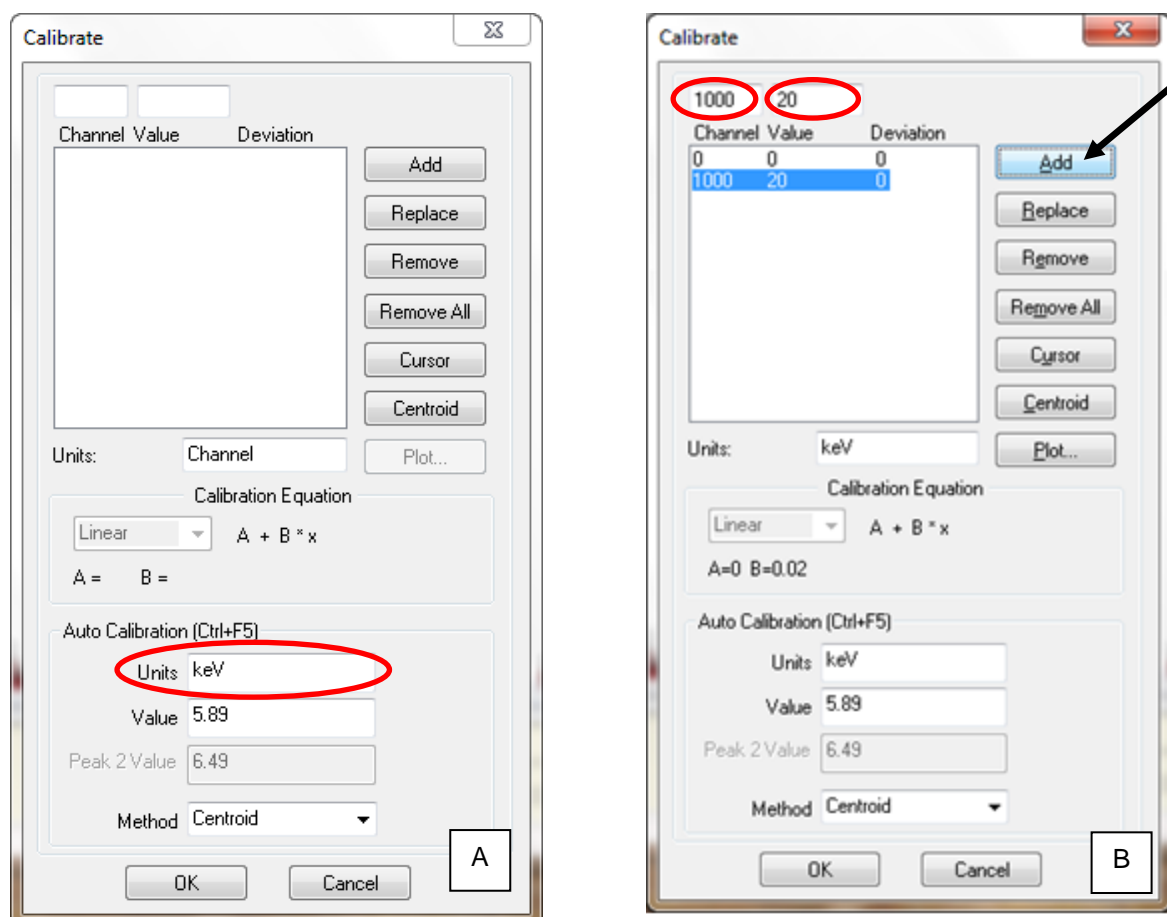


**Figure 1. Amptek’s DppMCA Software – Slow/Fast Threshold.** Select “DPP -> Tune Slow/Fast Threshold”.

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

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

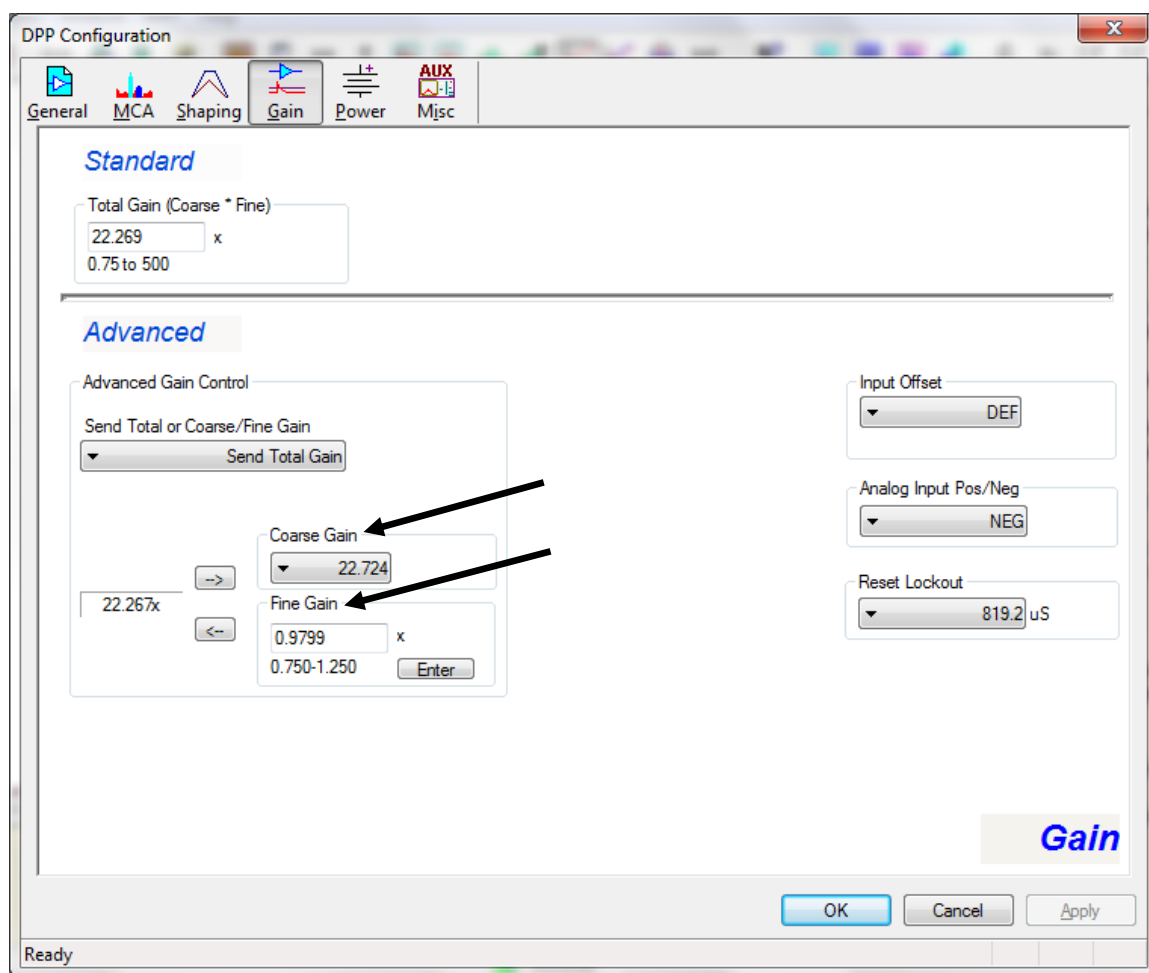
simultaneously). To do this in DppMCA, go to “Analyze->Calibrate”. This will display the dialog shown in Figure 2, part A. Change the units from “Channel” to “keV” to allow the spectrum to be displayed in keV. Then set the scale by “Adding” the appropriate channel and energy values (see Figure 2 below). 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 (or ADMCA) and acquire a spectrum.

- c) Still in the Amptek software (DppMCA or ADMCA) 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  $K\alpha$  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 is 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-FP 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.

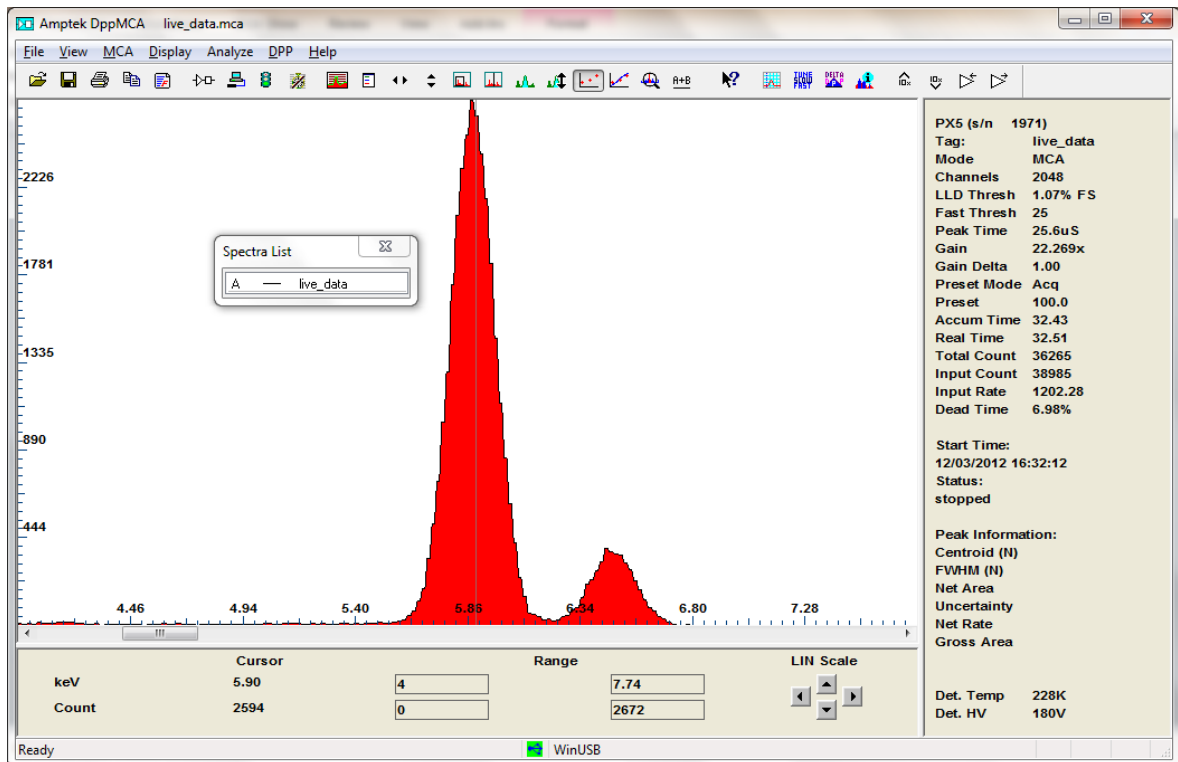


**Figure 3. Amptek’s DppMCA Software – Gain Panel.** DPP Configuration-Gain panel, showing the course and fine gain settings. Note that the threshold settings are located in the DPP Configuration-Shaping panel, should they need adjusting.

- 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  $K\alpha$  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 K $\alpha$  value of 5.895 keV (see Figure 4).

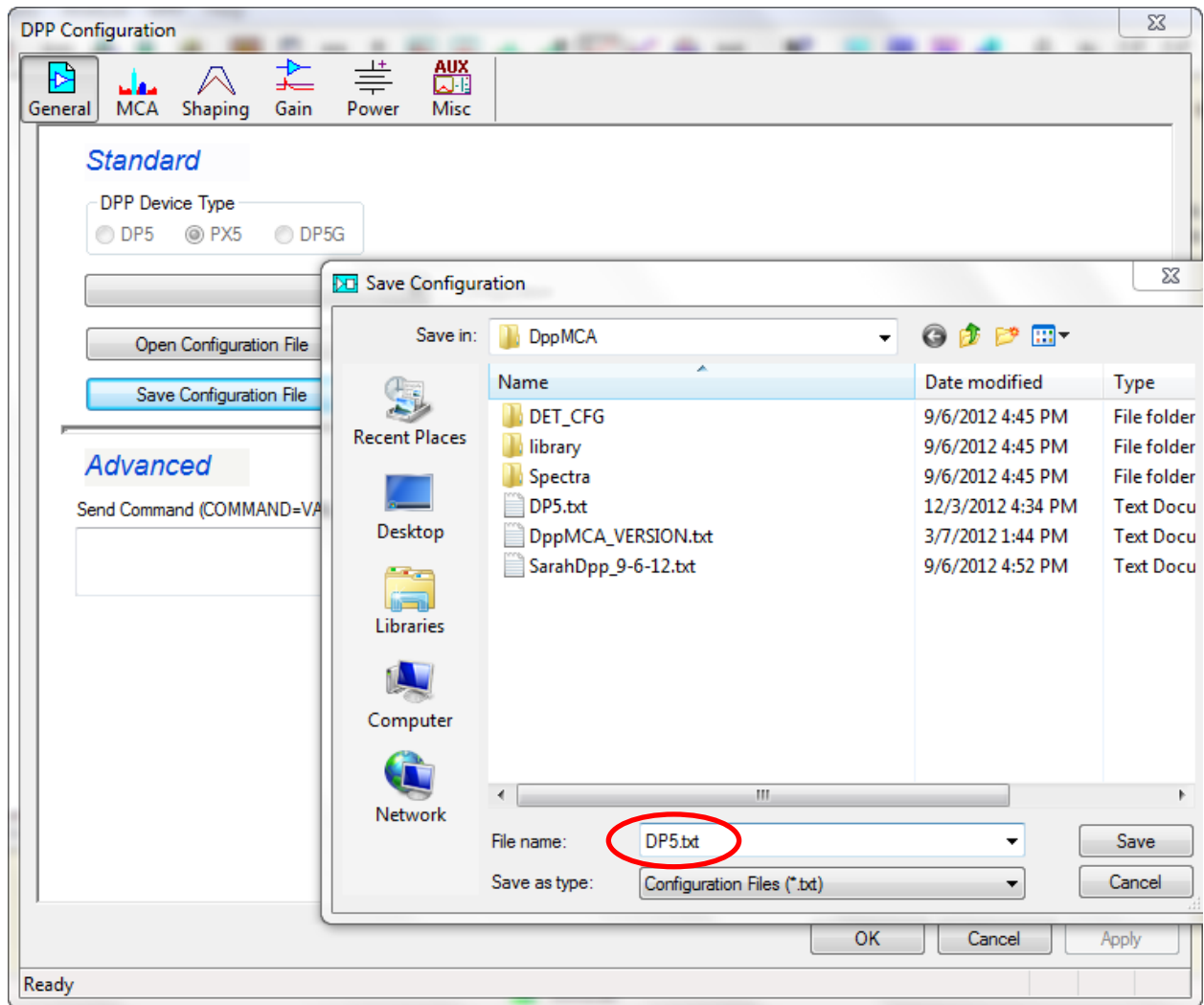


**Figure 4. Amptek's DppMCA Software – Spectrum.** Well calibrated Mn K $\alpha$  peak after adjusting the course and fine gains and tuning the slow/fast thresholds. The obtained Mn peak centroid is 5.90 keV, very close to the known Mn K $\alpha$  value of 5.895 keV.

- e) Next save the configuration file in the Amptek software. Assuming you are 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).

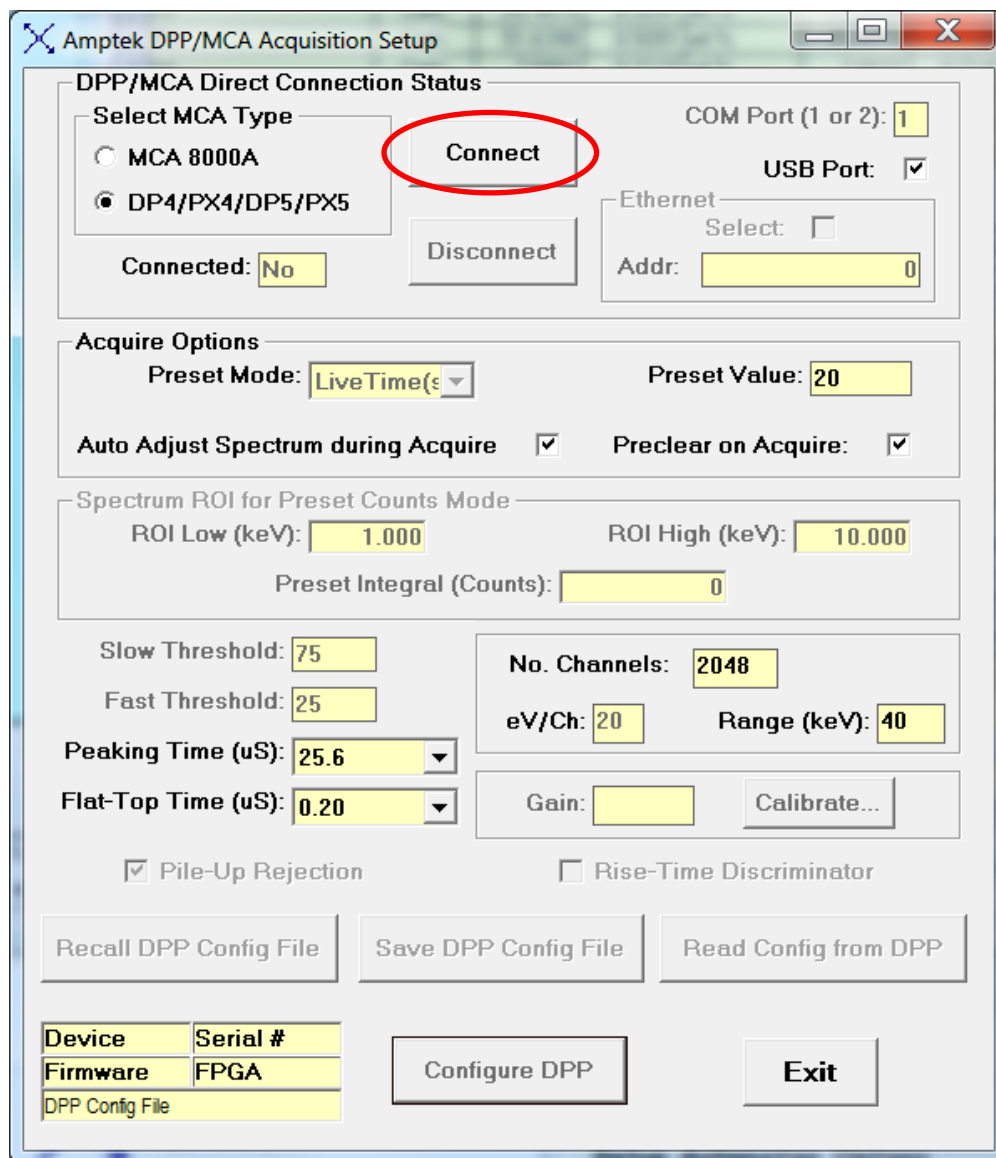
It is very important that the filename match the DPP device (e.g., DP5.TXT for a DP5 or X-123 module), because there are different parameters for each of these devices. Also, the XRS-FP software will later automatically recognize the type of DPP and then load the appropriate \*.TXT configuration file.

Note that there will be similar steps required for the older FW5 DPP devices. In this case, the ADMCA program should save the configuration files as DP4.CFG or PX4.CFG. Note that a DP5 with FW5 essentially emulates a DP4 processor, hence the naming convention.



**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.

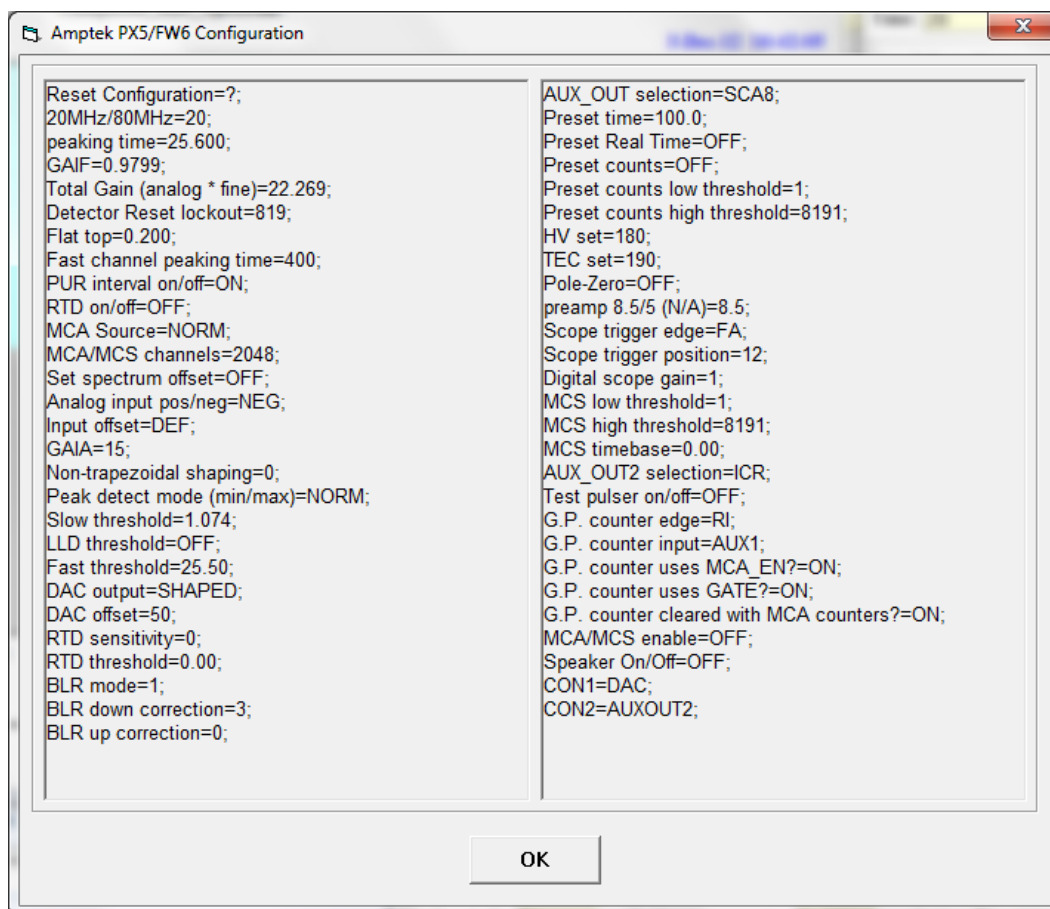
- f) Close the Amptek software at this point. It is important that DppMCA (or ADMCA) is **not** running simultaneously with XRS-FP, because the DPPs cannot be connected to more than one program.
- g) Now copy the configuration file (i.e. DP5.TXT or PX5.TXT, etc.) saved above, in step (e), to the XRS-FP software folder location (e.g., C:\CrossRoads Scientific\XRS-FP).
- h) Load the XRF-FP software, go to “Expert Mode,” and then open the “Amptek DPP/MCA Acquisition Setup” panel using the “Setup ->MCA/DPP” menu option. 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 or ADMCA software. See the example in Figure 6 below.



**Figure 6. XRS-FP 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-FP.INI file, except the Preset Value, which may have come from a TFR file.

Next click on the “Connect” button (Figure 6) and, after the connection is made with the DPP (using the USB interface in the example above), the XRS-FP software will load, apply and display (as shown in the example in Figure 7 below) the configuration file (PX5.txt) that was saved from the Amptek software (as shown above), and copied to the XRS-FP software directory. You should check that the parameters are set to the same values (e.g. total gain, number of channels, thresholds, peaking times, etc.) as seen in DppMCA.



**Figure 7. XRS-FP Software – Amptek Hardware Configuration Dialog.** This dialog 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-FP folder location.

Figure 8 below shows the XRS-FP “Amptek DPP/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).

Also note, in the bottom left corner of this panel, that the hardware parameters of the connected DPP are displayed. The example below 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.

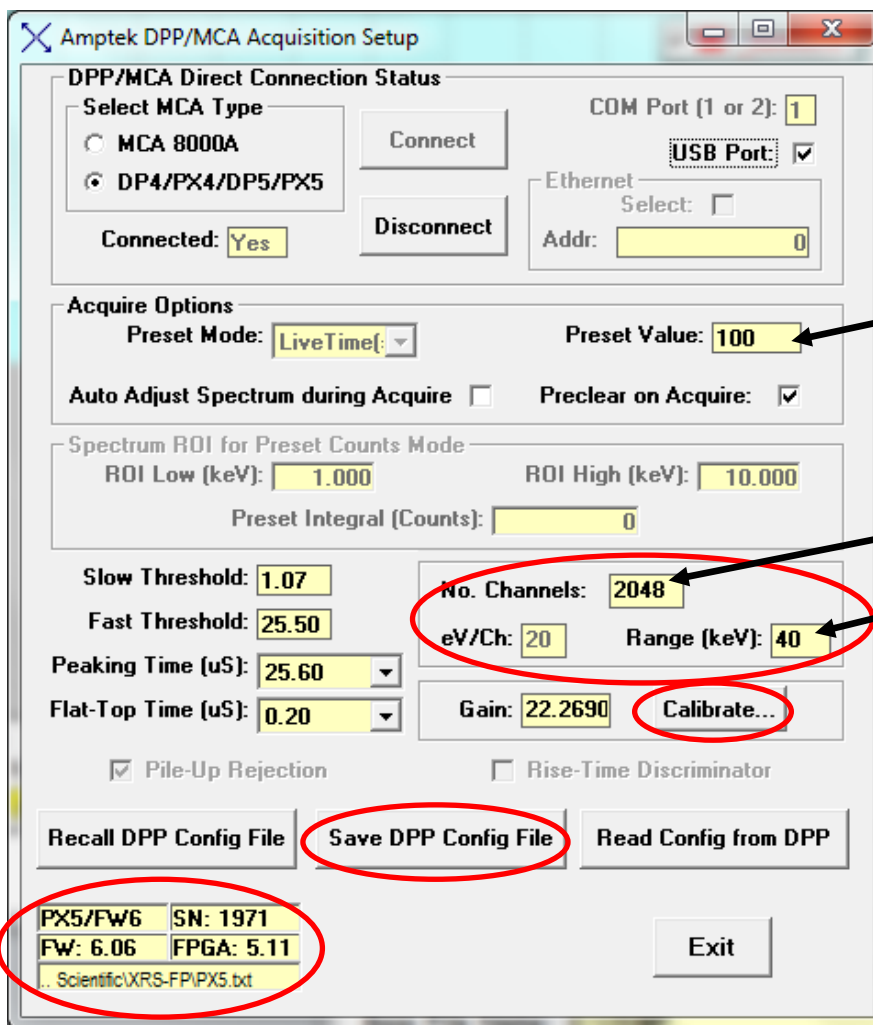
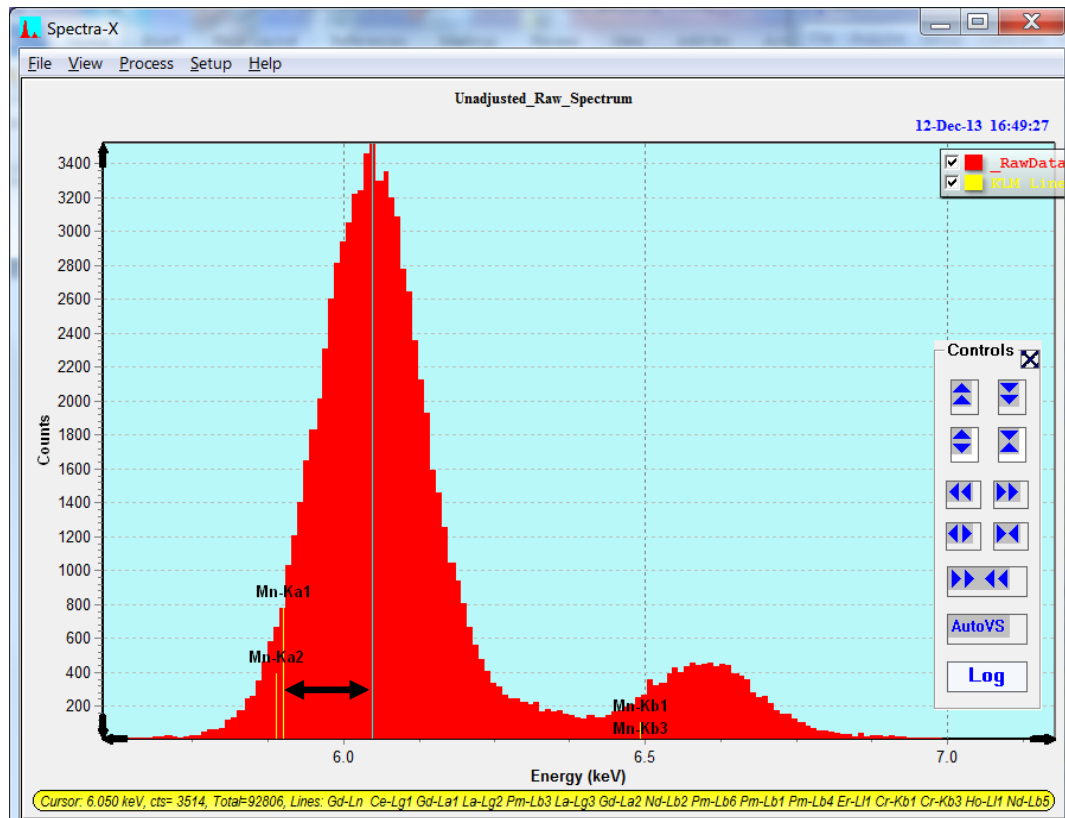


Figure 8. XRS-FP Software Setup-MCA/DPP Panel. Amptek DPP/MCA Acquisition Setup parameters after loading the DPP configuration file.

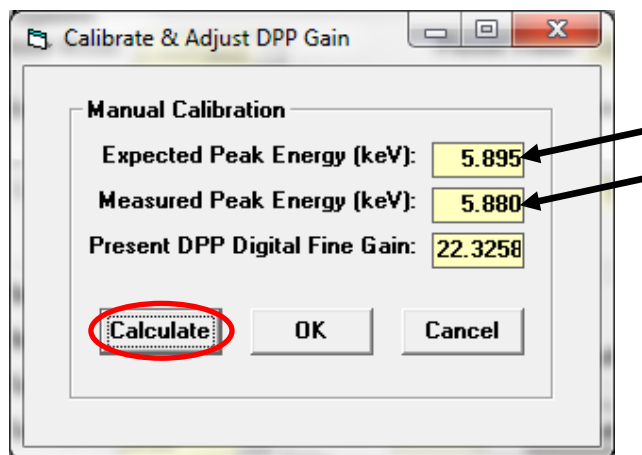
- j) Before leaving this panel there are two extra steps that should be done, although the first of these is optional. This first step (optional) is to fine tune the Total Gain value that was entered in DppMCA. This is, again, an approximate adjustment as we will then proceed to the software calibrate that will calibrate *both* the Gain and Offset. Until this point we are assuming that the offset is zero eV, which is rarely the case in practice. To fine tune the firmware Gain we will use the “Calibrate” button in the *Gain* section of the dialog (see Figure 8 above), but first we need to acquire a spectrum. Before doing any acquisition, make sure that *all* Adjust options (both in Setup-DPP and Setup-Processing) are turned **OFF**. This is very important.
- k) 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 in XRS-FP to collect a spectrum for about 30 seconds. Compare the peak

location to the expected location. In the example (Figure 9 below) the Mn-K $\alpha$ , $\beta$  peaks are shown along with the Mn-K line markers.



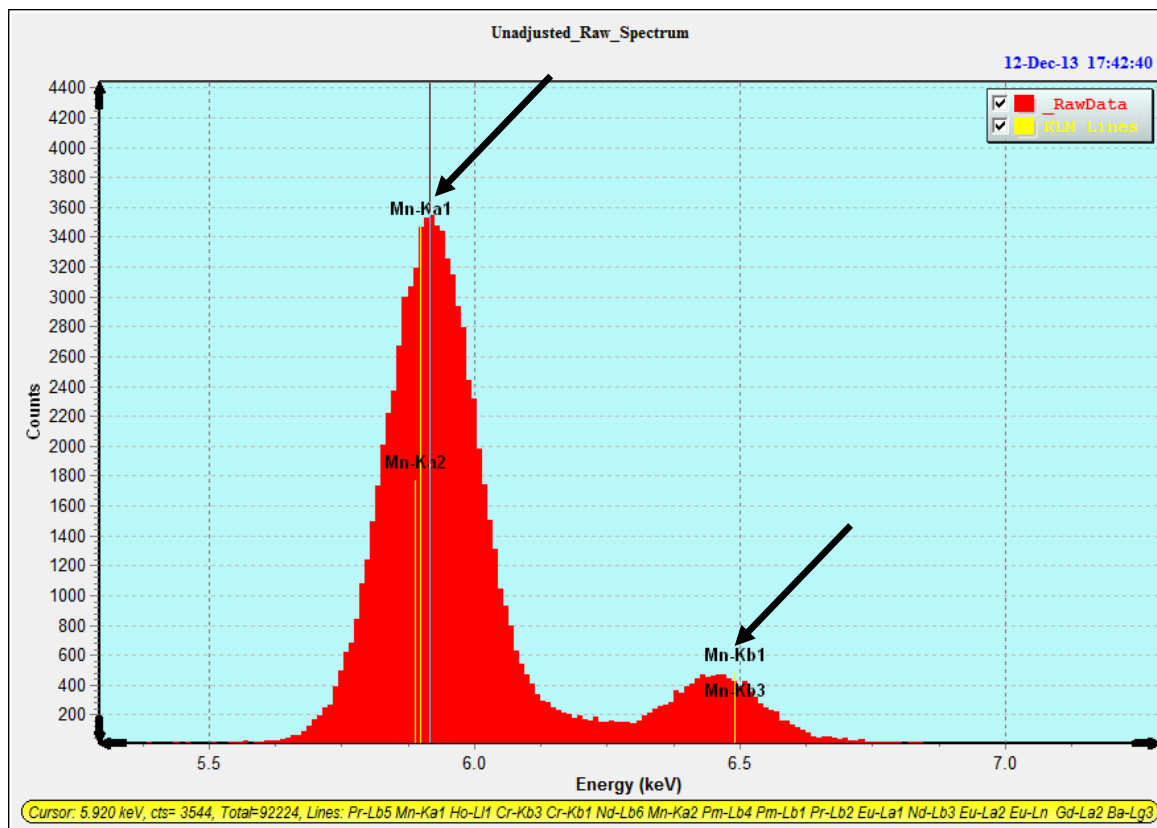
**Figure 9. XRS-FP Software – Spectra-X.** Compare the actual & expected peak locations.

- l) Now go back to the “Setup->DPP” panel and click the “Calibrate” button in the *Gain* section (Figure 8). This will display a new panel as shown below in Figure 10. Enter both the expected centroid energy of a known peak (e.g. Mn-K $\alpha$ ) in the spectrum, and the measured value as seen with the cursor in the Spectra-X panel (Figure 9). Then click “Calculate”, which will update the 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-FP Software – Calibrate & Adjust DPP Gain.** Enter the expected and measured peak centroid energies then click ‘Calculate’. This will give you an approximate but closer starting point for getting calibrated spectra.

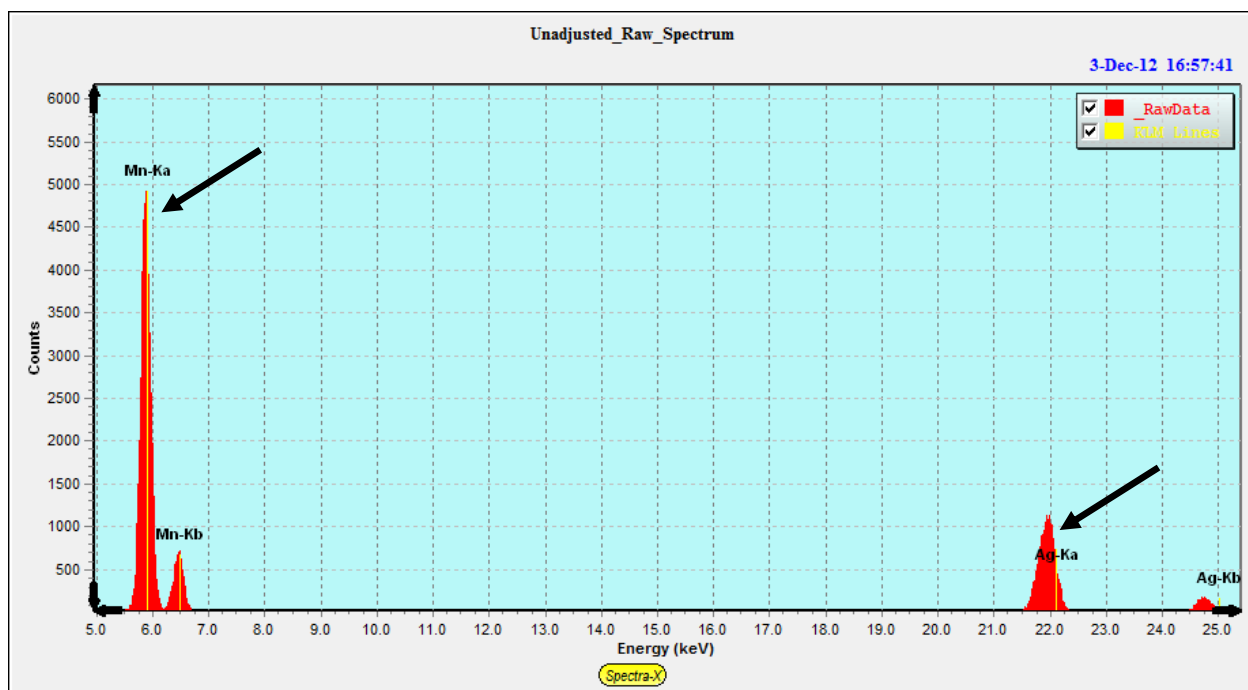
- m) Now click on the “OK” button and return to the Setup->DPP panel. Next click on the “Save DPP Config File” (Figure 8) and “Save” to the default DP5.TXT or PX5.TXT file. Now re-acquire a spectrum to check the peak locations and parameters. Using the previous conditions this might look like Figure 11 below. Notice that the Mn-K peak centroids are not perfect but much closer than before.



**Figure 11. XRS-FP Software – Spectra-X.** Spectrum after hardware gain calibration.

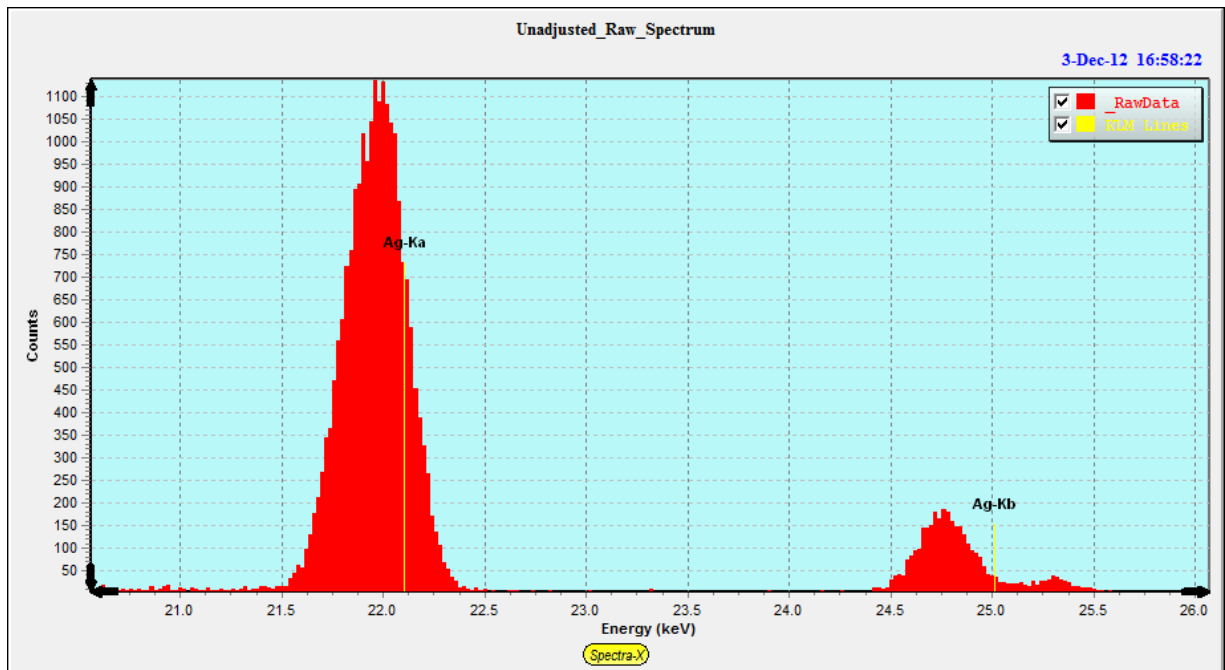
Note: This completes the hardware (and firmware) calibration. All necessary parameters are now set in the configuration file that controls the hardware. You are now ready to move onto the software calibration at this point (see steps below).

- n) Now we are ready to start the XRS-FP software gain and offset calibration. In XRS-FP, make sure the “Auto Adjust” option on acquire is **off** (in “Setup->DPP”). Now Acquire a spectrum from a sample that has two well-separated  $K\alpha$  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. 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 Mn  $K\alpha$  and the Ag  $K\alpha$  peaks are reasonably close to their known centroid values of 5.895 and 22.104 keV, respectively. Figure 13 displays the silver peaks from the full spectrum shown in Figure 12. This shows that the silver peaks are not well calibrated.



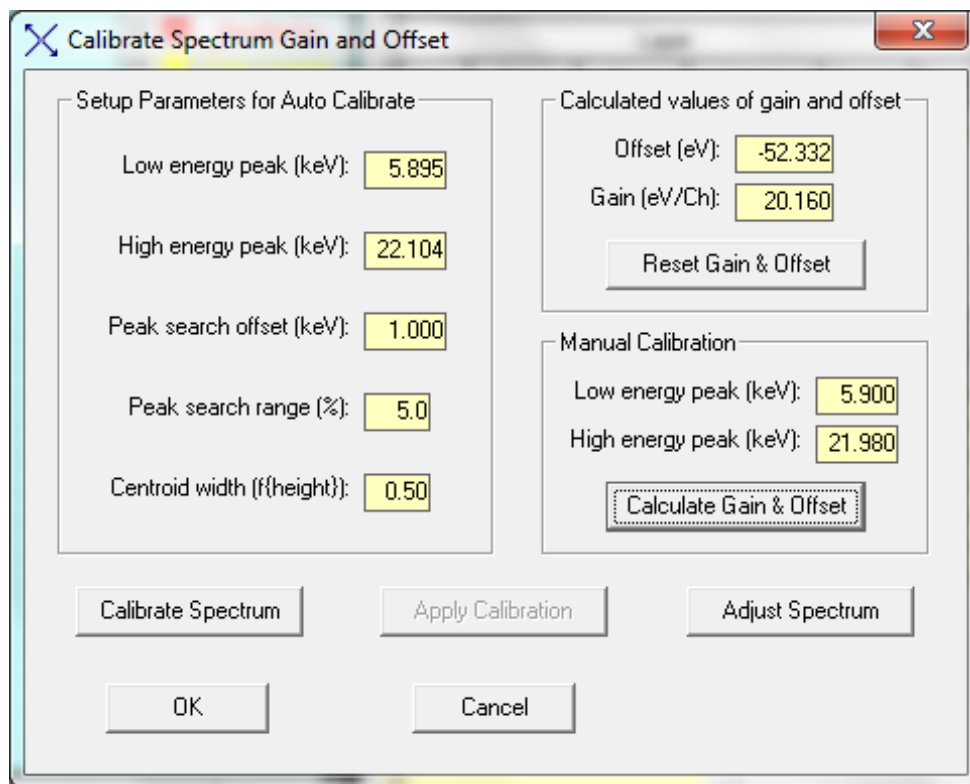
**Figure 12. XRS-FP Spectra-X.** Acquired spectrum showing 2 well-separated and well-defined peaks (i.e. Mn- $K\alpha$  and Ag- $K\alpha$ ) for use in the 2-point software calibration.





**Figure 13. XRS-FP Spectra-X – Example Spectrum.** Spectrum showing that the silver peaks are not calibrated correctly.

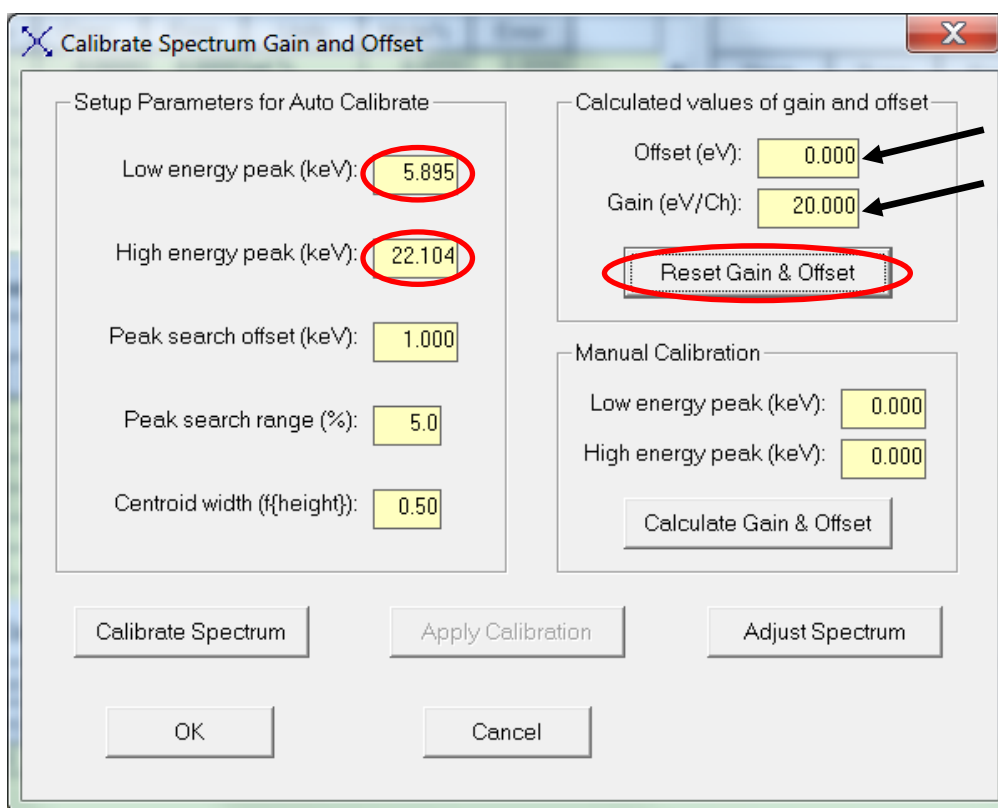
- o) Now “Exit” from the Acquisition-Setup panel and then display the Calibrate-Spectrum panel via the main menu. An example of this panel is shown below in Figure 14.



**Figure 14. XRS-FP Calibrate-Spectrum Panel.** Example of the Calibrate Spectrum Gain and Offset panel in XRS-FP before Resetting Gain & Offset.

To complete the software calibration we will only use the Calibrate-Spectrum panel.

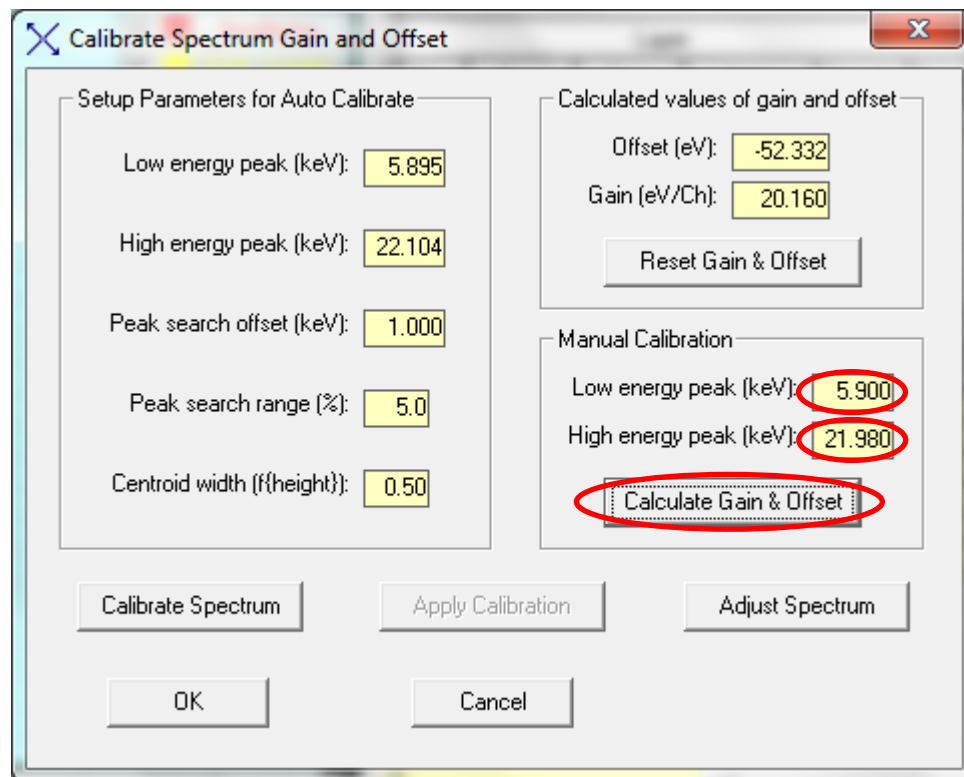
- p) Next click on the “Reset Gain & Offset” button. This ensures that any previous XRS-FP software calibration is reset (cleared) to the target values of a zero Offset energy (eV) and Gain (channel width in eV). In our setup so far, this will result in the following screen, as shown in Figure 15. Note that the target eV/ch changed from 20.160 to 20.000, and the Offset reset to zero, in our example. After this, you may optionally click on the “OK” button and the changes so far will be saved in the XRS-FP.INI file. Of course, you must then re-load the “Calibrate->Spectrum” panel if you do this!



**Figure 15. XRS-FP Calibrate-Spectrum Panel.** The screen from Figure 14 after clicking on the “Reset Gain and Offset” button.

- q) Enter the centroid ( $K\alpha$ ) values of the Low and High energy peaks used for calibration in the “Setup Parameters for Auto Calibrate” section. For example, in the calibration used here, the centroid values for Mn- $K\alpha$  and Ag- $K\alpha$  were entered as the Low and High values, respectively (see Figure 15 above). 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.

- r) Next we will do a “Manual Calibration”. Use the cursor to find the centroid for each peak in Spectra-X. Enter these values as the “Low energy” and “High energy” peak values in the “Manual Calibration” section (see Figure 16 below). In the example here, we typed in 5.900 keV for the Low energy peak, and 21.980 keV for the High energy peak. Now click on the “Calculate Gain & Offset” button. After this you should see changes in Offset and the Gain values (see Figure 16 below). The Offset has changed from 0 to -52.332 eV, and the Gain from 20.000 to 20.160 eV per channel.

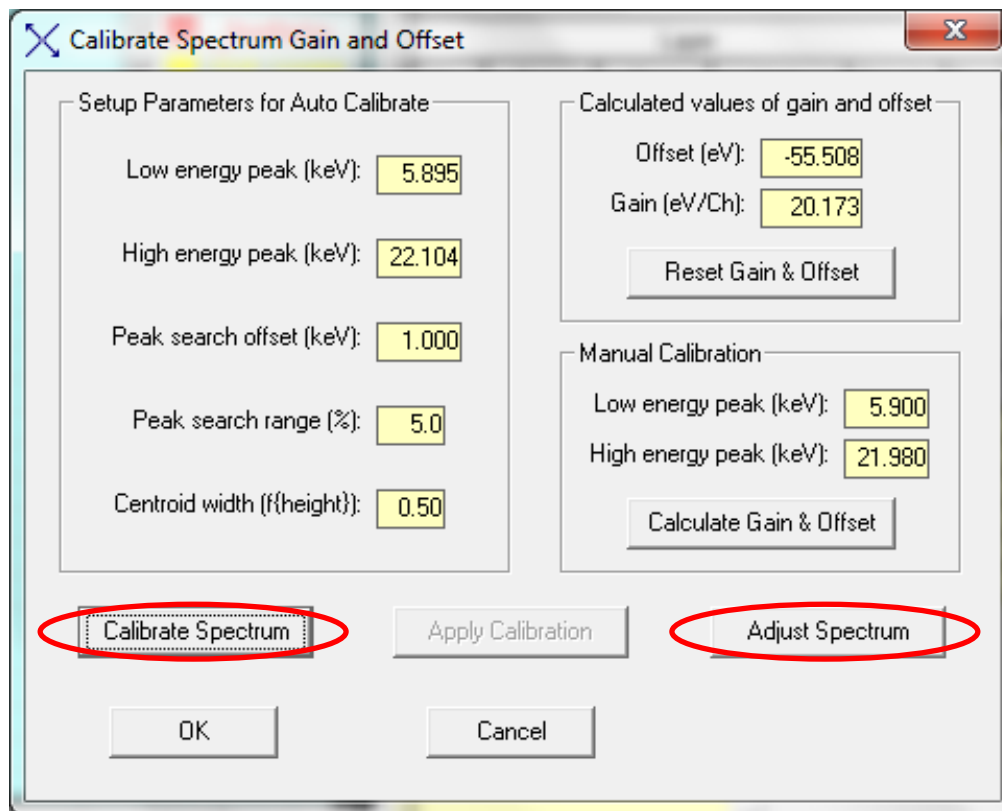


**Figure 16. XRS-FP Calibrate-Spectrum Panel after Manual Calibration.** The screen from Figure 15 after clicking on the “Calculate Gain & Offset” button.

- s) 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 or ADMCA), otherwise the hardware setup will never match the intended software setup.
- t) 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-FP 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 for each of the two peak energies.

- u) Now click on the “Calibrate Spectrum” button to perform the automatic calibration step. This 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 17 below. Note: that you will see some change in offset and a slight change in the gain at this point.



**Figure 17. XRS-FP Calibrate-Spectrum Panel after Automatic Calibration.** The screen from Figure 16 after clicking on the “Calibrate Spectrum” button.

Notice that the Offset has changed from -52.332 to -55.508 eV, and the Gain from 20.160 to 20.173 eV per channel. Because the manual calibration was already close the automatic values are not that different.

- v) Next click on the “Adjust Spectrum” button (Figure 17). 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 18 and 19 below, which show the adjusted and original spectra collected for the example used in this guide (i.e. the adjusted and original peaks for Mn and Ag).

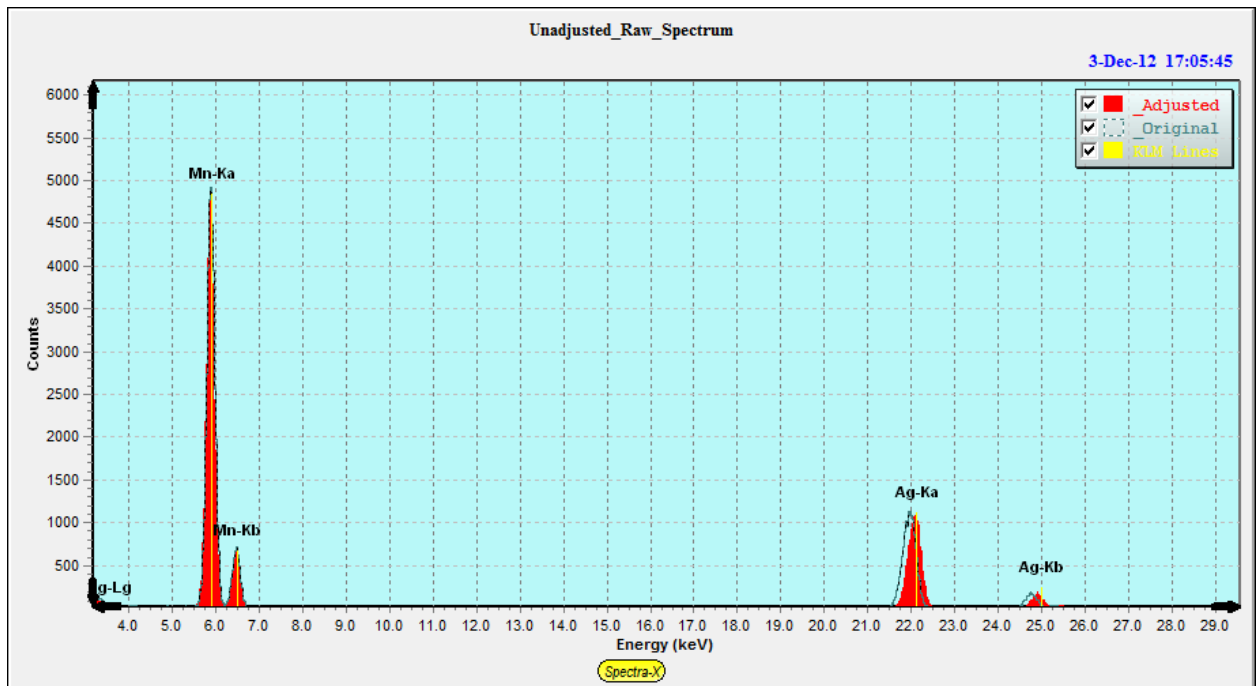


Figure 18. XRS-FP Software – Spectra-X. Note that the peak locations (centroids) now fall at the correct energies in the adjusted spectrum (red) as compared to the original spectrum (dashed) collected before calibration.

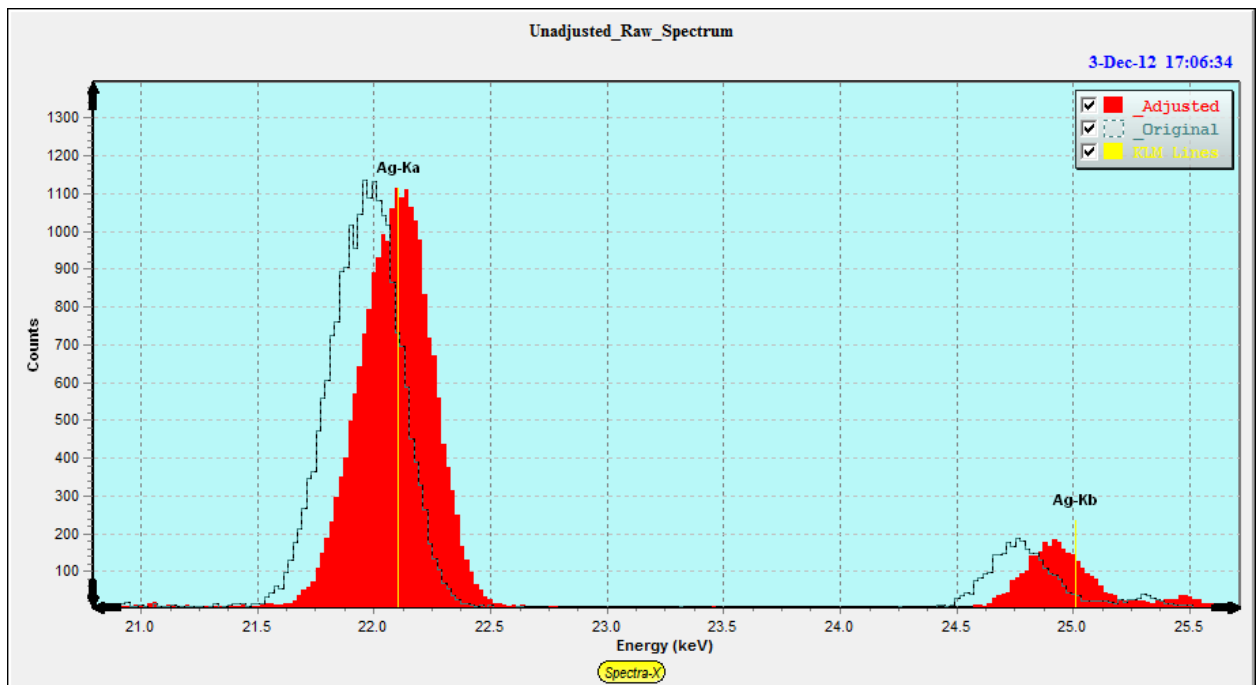


Figure 19. XRS-FP Software – Spectra-X. Detailed view of the Ag peak used in the example calibration showing the original and adjusted spectra.

- w) If you are happy with the resulting spectrum after the Adjust step, then click on the “OK” button (Figure 17). This will save the Offset and Gain values in the XRS-FP.INI file for future use. Each time the software is loaded these values will be loaded for subsequent Adjust operations on spectra to ensure that they are converted into the calibrated form that is required by the XRS-FP software for spectrum processing.
- x) Finally re-open the “Setup->DPP” panel in XRS-FP. Now select the “Auto Adjust Spectrum during Acquire” option in the “Acquire Options” section (see Figure 20 below).

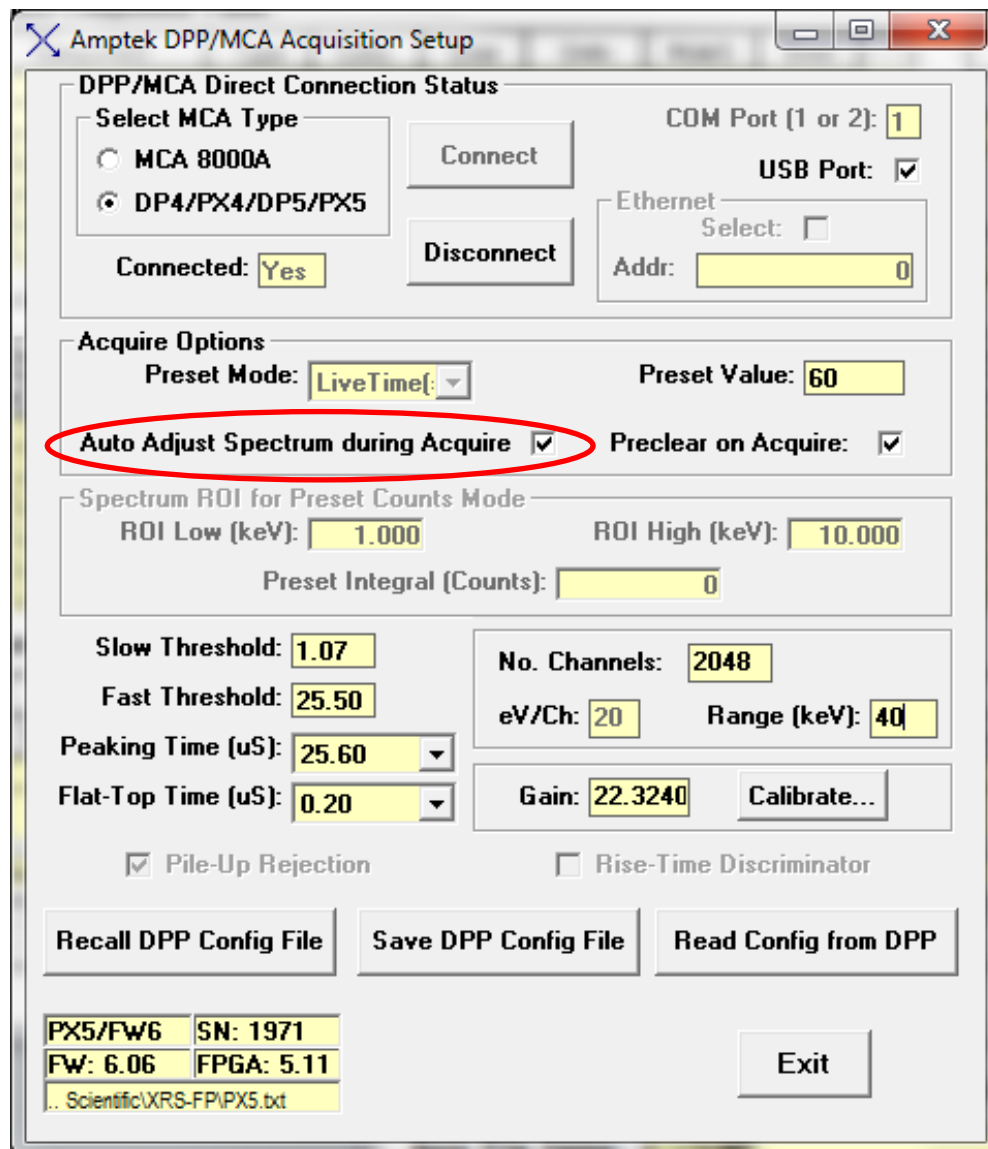
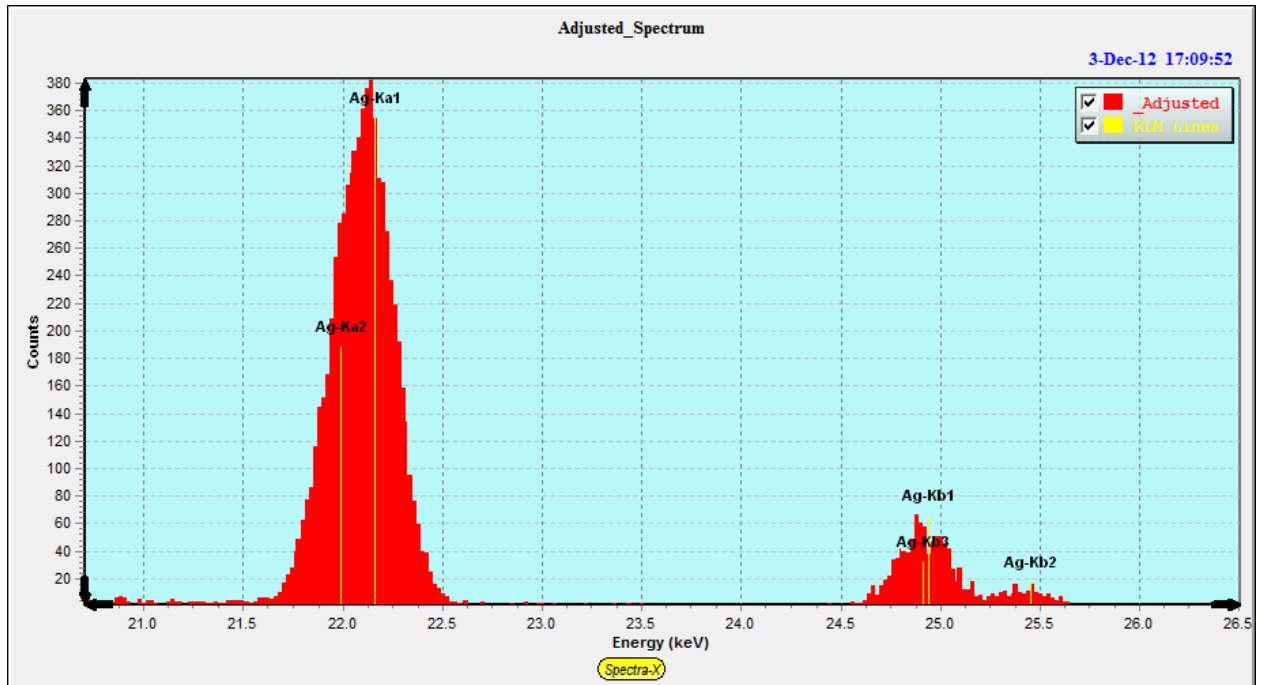


Figure 20. XRS-FP Software Calibrate-Spectrum Panel. Select the “Auto Adjust Spectrum during Acquire” option.

- y) Now, "Acquire" a spectrum. You should see that the acquired spectrum is now well calibrated (see Figure 21 below).



**Figure 21. XRS-FP – Spectra-X.** Acquisition of a well-calibrated spectrum having followed all the calibration steps above.

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