

ENERGY CALIBRATION IN DPPMCA AND XRS-FP REV A0

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Energy calibration is very important for quantitative X-ray analysis. Even a small error in the calibration of the energy scale can have significant consequences: if the photopeaks are shifted even slightly from their expected positions, the software may misidentify peaks, compute erroneous photopeak areas, and make significant errors when correcting for overlapping photopeaks, sum peaks, etc.

Amptek's Experimenter's Kit makes use of two different software packages: the DPPMCA software and the XRS-FP software. Both programs include the capability to calibrate the energy scale but they treat the calibration differently. These differing approaches are the source of many errors for users. This application note is intended to describe, very briefly, how to minimize these issues.

Background

For any spectrometer, a key parameter is the "system conversion gain", the eV/channel. It relates the system input, the energy deposited by a particle in units of energy, to the system output, the corresponding channel number in the output histogram or spectrum. Another key parameter is the "offset", the channel corresponding to zero energy. Ideally, the offset would be zero and the gain exactly equal to a convenient value. In real hardware, the gain and offset depend on many things, including parameters the user can change, on component tolerances, and on temperature coefficients of the hardware.

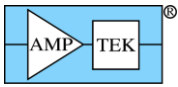
DPPMCA is a "hardware-centric" software package. We recommend using DPPMCA to optimize a wide range of hardware parameters in the spectrometer before you use XRS-FP to acquire and analyze the spectra.¹ The system conversion gain and offset depend on many different hardware parameters, some which a user can adjust and some which vary from one detector to another. The approach of DPPMCA is to set these parameters, then to measure the channels corresponding to the photopeak energies, and then to perform a linear regression, yielding the gain and offset.

XRS-FP is an "analysis-centric" software package; it controls the hardware to take a spectrum but then does extensive spectrum processing. It can read in a spectrum which has been previously acquired with DPPMCA or XRS-FP. For efficient analysis, XRS-FP always assumes that the spectra have zero offset and an exact integer system conversion gain (5, 10, 20, 40, 80 and 160 eV/channel). Since the hardware is not always set to exactly zero offset or exactly this gain, XRS-FP adjusts spectra (i.e. re-bins the data) based on calibrated values and on check boxes which enable adjusting.

You can use either DPPMCA or XRS-FP to change the DPP's gain and to calibrate the energy scale. The interfaces and dialogs are quite different in the two software packages.

Most problems occur when the check boxes in XRS-FP are set incorrectly (i.e. inconsistent with each other or with the settings when the data were acquired) or when the calibration data stored in the file are not consistent with the spectrum adjust options.

¹ Energy calibration and gain are discussed in the "Gain" section of the FAQ in DPPMCA's HELP. Note that the "system conversion gain" (eV/channel) differs from the digital processor's gain (a dimensionless number and one factor of many in the system conversion gain).



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Recommendations

1. Setting the gain

- 1.1. We recommend first setting the DPP gain so that the system conversion gain is close to 5, 10, 20, 40, 80 or 160 eV/channel. If it is close, XRS-FP does not have to adjust (rebin) the data very much, minimizing errors. A typical value for the eV/ch would be 20 assuming you are using 2048 channels. I.e. a range of 40 keV typically contains most if not all the peaks of interest for XRF analysis using Si detectors.
- 1.2. You can set the gain using either DPPMCA or XRS-FP. In both cases, you take an initial spectrum, determine the initial system conversion gain and offset, compute the new DPP gain and offset, and send them to the DPP.
- 1.3. In DPPMCA, the calibration dialog gives you a gain and offset. You must calculate the new values using a calculator or spreadsheet (an example is below).
- 1.4. In XRS-FP, the "Setup", "DPP/MCA" dialog has a utility to quickly compute the new gain (no calculator needed) but only changes the gain, not the offset. A detailed description of its use is given on page 4.

1.5. Notes

- In XRS-FP, under "Setup", "DPP/MCA", you must enter the number of channels (e.g. 2048) and the energy range (e.g. 40 keV). These give the expected gain (e.g. 20 eV/channel). XRS-FP requires the actual gain to be close; if you tell XRS-FP that you have 2k channels, and 40 keV full-scale, but the gain is really 5 eV/channel, it will generate an error message.
- The configuration of the processor is stored in EEPROM in the DPP; when the DPP is powered on, it recalls the configuration in use when it was powered off. If you use DPPMCA, the software simply connects and uses the internal configuration. If you use XRS-FP, it reads in a configuration stored in the DP5.TXT or PX5.TXT file (in the same directory as XRS-FP.EXE) and overwrites that stored in the DPP's EEPROM. Therefore, every time you change the DPP configuration, you should save the new config to DP5.TXT (or PX5.TXT). You can do this using DPPMCA or XRS-FP. The choice of DP5.TXT or PX5.TXT is made automatically depending upon which type of DPP is present. (If no file is found then the XRS-FP software will use the existing DPP configuration, just like DPPMCA.)

2. Calibrating the energy scale

2.1. You can calibrate the energy scale using either DPPMCA or XRS-FP.

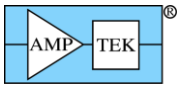
- In DPPMCA, you can calibrate using any number of photopeaks (the software performs a linear regression on the centroids) and using any number of spectra (you can take spectra from several pure sample and then combine the results). When you calibrate in DPPMCA, you must manually transfer the calibration results (gain and offset) into XRS-FP.
- In XRS-FP, you calibrate using two and only two photopeaks and they must be present in the same spectrum. You need a sample with two known, strong, widely separated photopeaks.

2.2. Calibrating in DPPMCA

- A detailed description of DPPMCA's calibration dialog is described in DPPMCA's HELP.
- DPPMCA yields two parameters, denoted "A" and "B", which are the spectrum offset (A) and system conversion gain (B) derived from a linear regression. XRS-FP adjusts the spectrum based on two parameters, the offset (eV) and gain (eV/channel), found under "Calibrate", "Spectrum". You take the values found in DPPMCA and enter them here in XRS-FP. Note that these must be in eV; if you calibrated DPPMCA in keV, then you must rescale to eV. Prior to entering these values ensure that you have previously set the correct target eV/channel, and clicked on the "Reset Gain and Offset" button.

2.3. Calibrating in XRS-FP

- A detailed description is given on page 6.



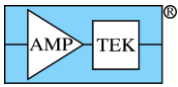
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3. Acquiring data

- 3.1. We recommend enabling the checkbox in "Setup", "MCA/DPP" labeled "Auto Adjust Spectrum during Acquire". When this is checked, and you acquire the spectrum in XRS-FP, it re-bins the data as they are acquired so the spectrum has zero offset and the target integral gain (eV/channel).
- 3.2. We recommend using XRS-FP to acquire the spectra which will be used for analysis. We find there to be fewer errors when XRS-FP is used for all the steps: taking the spectrum, automatically adjusting it, and processing it. Use DPPMCA only to configure the hardware.

4. Opening files

- 4.1. We recommend you do not enable the checkbox in "Setup", "Processing" labeled "Auto Adjust Spectrum Gain & Offset on Load". If this is enabled and the box is enabled to auto-adjust during acquisition, then when you load a file, it is adjusted twice, causing errors. The XRS-FP software will automatically warn you against doing this.
- 4.2. If you have saved a .MCA file acquired in XRS-FP, and it was adjusted during acquisition and is not adjusted on load, the energy scale will be correct.
 - Note that XRS-FP stores the energy calibration values in the XRS-FP.INI file. If you take spectra one day, then adjust the DPP gain the next day, your original calibration values are over-written.
 - We strongly recommend that, for every spectrum you take using XRS-FP, you save not only the .TFR file and the .MCA file but also a copy of the XRS-FP.INI file you used. If you will load a file for analysis, make sure to overwrite the XRS-FP.INI file before launching the software.
- 4.3. If you have saved a .MCA file acquired in DPPMCA, and you did not save the calibration values in the .MCA file, then XRS-FP will not automatically do any adjustments. After you have loaded the file, go to "Calibrate", "Spectrometer", and press "Adjust Spectrum".
- 4.4. If you have saved a .MCA file acquired in DPPMCA, and you calibrated it in DPPMCA, then XRS-FP will automatically adjust (re-bin) the data using the calibration values stored in the .MCA file.
 - Note that XRS-FP assumes you calibrated in keV. If you calibrated in eV, XRS-FP generates an error message (the gain is off a thousand-fold).
 - Note that the .MCA file from DPPMCA does not store the gain and offset for the calibration. Instead, it saves the list of data points used in the calibration. XRS-FP only reads the first two of these. So if you calibrated in DPPMCA using three or more points, the calibration will be off when reading the .MCA file in XRS-FP.



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Detailed Instructions

Correcting the gain using DPPMCA

The plot below shows the result of an energy calibration in DPPMCA. A spectrum was taken from a stainless steel 316 sample, the Fe and Mo photopeaks marked, and their centroids used in the calibration dialog.

As the screen shows, the DPP gain was 30, and the calibration yields a system conversion gain of 20.56 eV/channel. The calibration also yields an offset of -51 eV (this means that channel zero is at -51 eV, or equivalently zero energy is at $(51 \text{ eV}) / (20.56 \text{ eV/channel}) = 2.5$ channels).

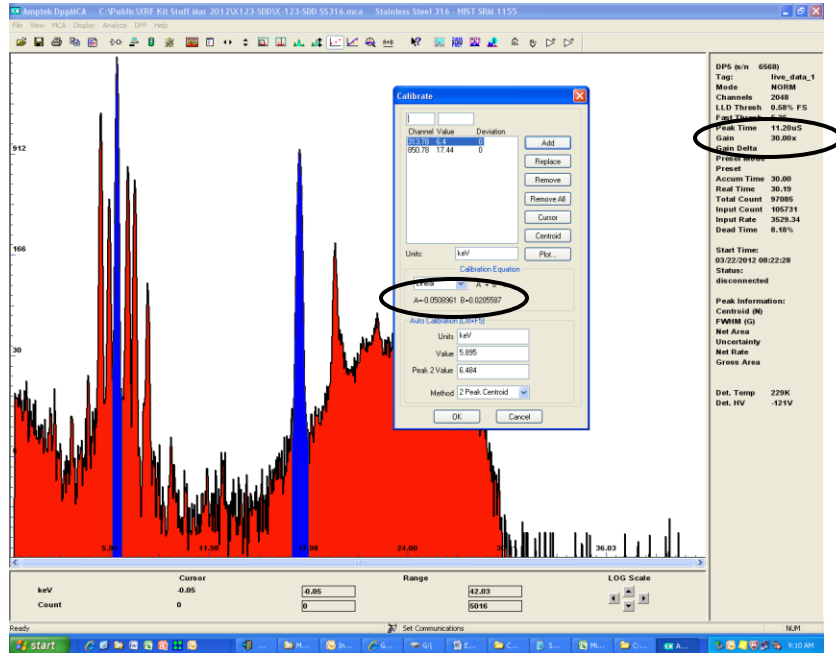
The spreadsheet on the right shows calculations to get the correct gain and offset. To get the correct system conversion gain, the DPP gain should be commanded to

$$(20/20.56) * 30 = 29.18.$$

The spectrum offset should be commanded to

$$-2.5 * (29.18/30) = -2.4 \text{ channels.}$$

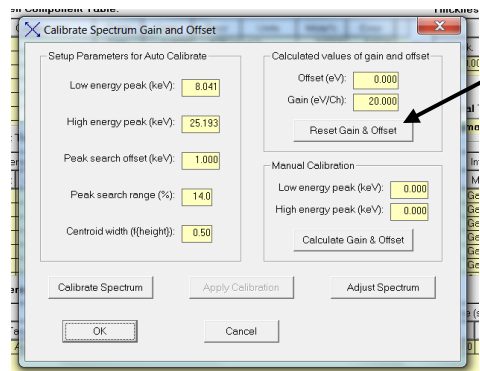
Note that the offset can be commanded to a fractional number of channels.



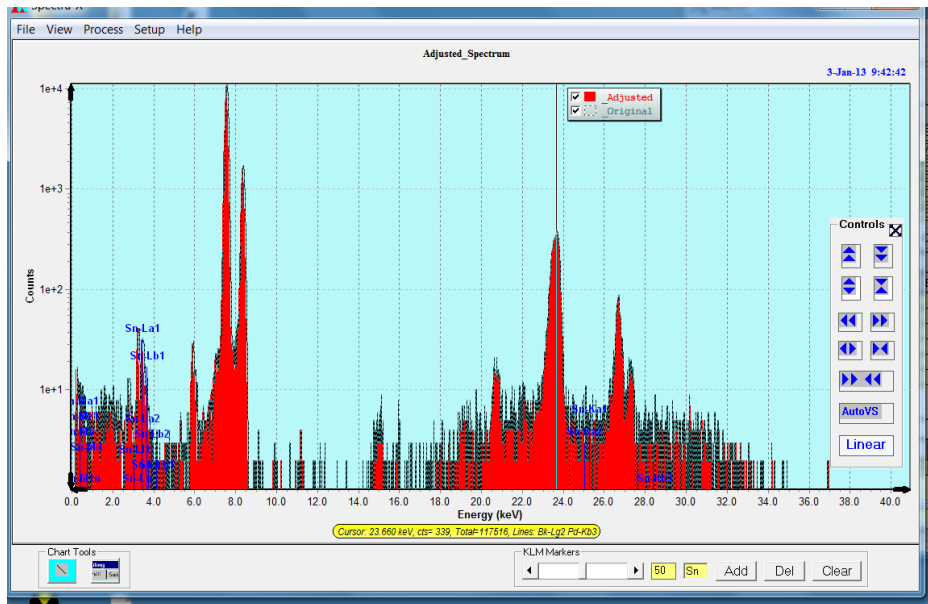
	Initial	Target
DPP Gain	30	29.18
DPP Offset (channels)	0	-2.4
A (measured offset, keV)	-0.0509	0
B (measured gain, keV/channel)	0.020559	0.02
Measured offset (channels)	-2.5	-2.4

Correcting the gain using XRS-FP

- 1) Open "Calibrate", "Spectrum" and press "Reset gain and offset". This sets the offset to zero and the gain to its ideal (10, 20, etc. eV/channel) and ensures that the software is not adjusting the spectrum.

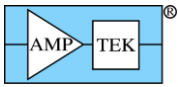


- 2) Ensure the "Auto Adjust Spectrum During Acquire" check box is unchecked in the Setup-DPP/MCA panel. Then acquire the calibration spectrum. The plot below shows one measured from a Cu-Sn calibration sample (on a log scale). Double click on the photopeak, and then drag the cursor to the centroid of the photopeak. You can read the energy of the cursor in the yellow box at the bottom. In this case, the Sn K_{α} peak is at 23.66 keV (it should be 25.193 keV).



- Go to "Setup", "DPP/MCA". Click the "Calibrate" button in the *Gain* section.. In the dialog that appears (shown below), enter in the top box the expected peak energy (25.193 keV for the Sn K_{α} peak in this example). In the middle box, enter the measured energy (23.66 keV in this example). Click "Calculate" and a new DPP gain is computed.

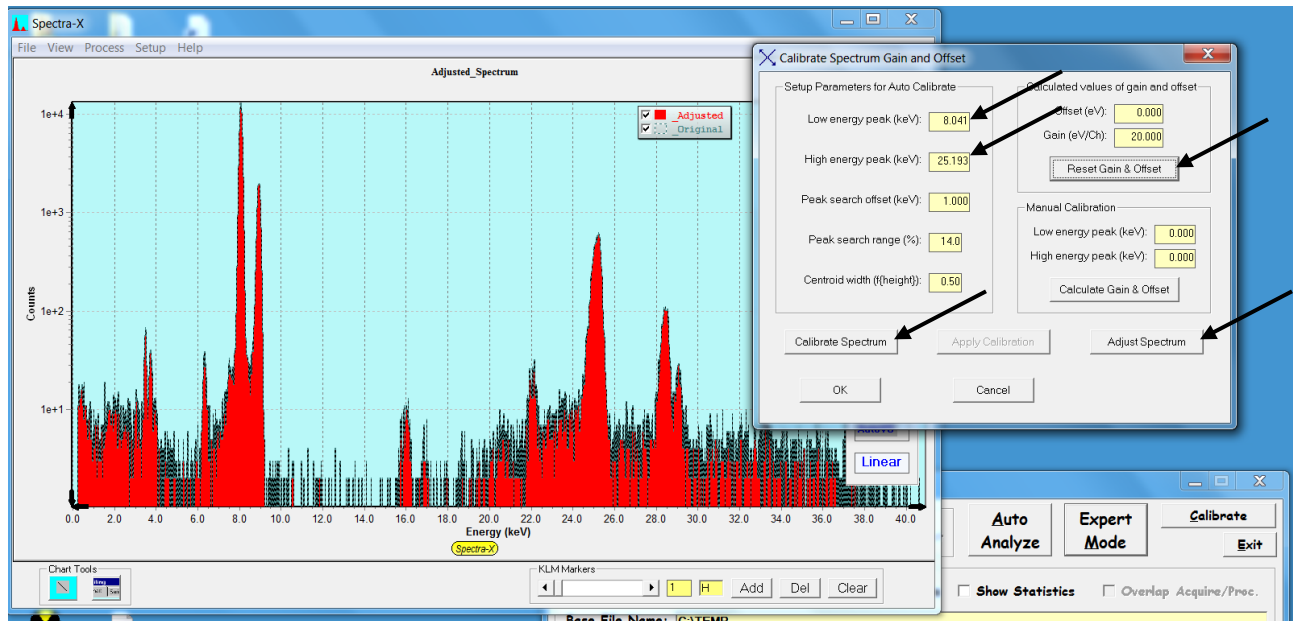
- Click "OK" and the value is written to the main dialog and sent to the DPP. Click "Save DPP Config File" so that the new value will be written to DP5.TXT (or PX5.TXT), and thus be available when you next launch XRS-FP.



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Calibrating using XRS-FP

- 1) Make sure the "Auto Adjust" option on acquire is *off* (in "Setup -> DPP") then acquire the spectrum to be used for energy calibration. Use a sample with two strong, well-defined peaks, well separated from other peaks, and well separated in energy. The example below uses a Cu-Sn sample.
- 2) Open "Calibrate", "Spectrum". First
- 3) Press "Reset gain & offset", to clear these values.
- 4) Enter the correct centroid energies into "low energy peak" and "high energy peak" boxes
- 5) Use the cursor to find the centroid of each peak in Spectra-X. Enter these values as the "Low energy" and "High energy" peak values in the "Manual Calibration" section
- 6) Click "Calculate Gain & Offset" (you will see some change in offset and the gain at this point).
- 7) Now use the automatic calibrate method by clicking on the "Calibrate Spectrum" button to get refined values of the gain and offset. Press "Adjust spectrum" to adjust the spectrum to match the new values. These new values are automatically written to XRS-FP.INI, over-writing any previous values, when the OK button is clicked (the Cancel button will not save any of these values).



You can use this "Auto Calibration" sequence on a routine basis, to correct for spectrometer drifts from one day to the next.