

APPENDIX C

Spectrometer settings and data acquisition guide.

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I. Geometry considerations.

The samples must be placed in a consistent manner, as geometry has a fairly significant effect on the peak amplitudes. The vertical plane and the horizontal angle of the sample should be fixed. The horizontal angle is varied by rotating the sample holder. Set it so that it forms a 45 degree angle with both the X-ray tube and the detector, in order to minimize Rayleigh and Bragg scattering.

Low-energy X-rays (those below 4keV) get severely attenuated in air. As such, if you want to see low-energy peaks, such as Si K_{α} , Sn L_{α} , etc. you must try to minimize the detector-sample distance.

II. Checking initial (saved) settings.

Before taking spectra, a quick check of the apparatus settings will save your time. On the PC desktop click on an icon of the ADMCA software . In a selection window click on the “Connect to MCA” option.

In the opened window, find a function DPP in the upper row of options, and in the drop-down menu select “Display Settings”. You will see the configuration of the detector with current setting as in Fig.C1.

If the real values are different from those in Fig.C1, start re-setting the detector manually.

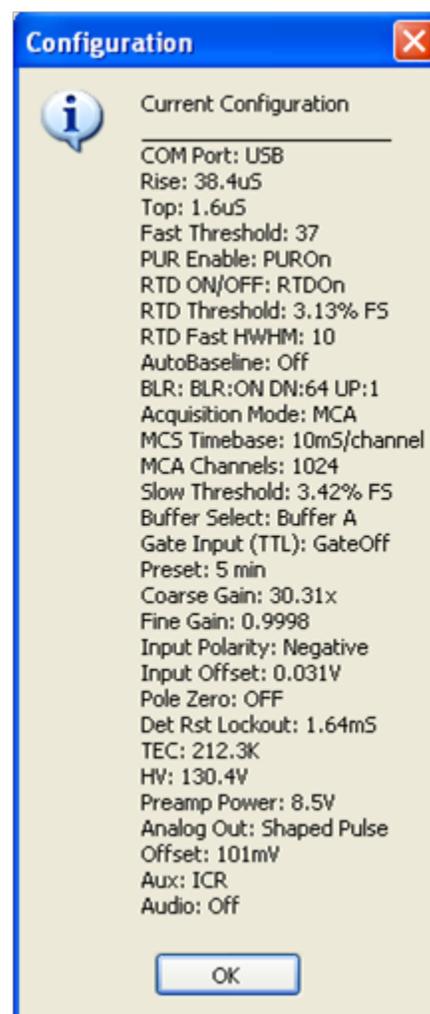


FIG. C1.

III. Detector settings adjustment.

The settings for the detector can be accessed and changed via the Amptek ADMCA software, either by pressing F9, going into the MCA menu and selecting “Acquisition Setup”, or by clicking  in the toolbar. The following window should pop up:

1 – General. Selecting a detector.

The “General” window is shown in Fig. C2.

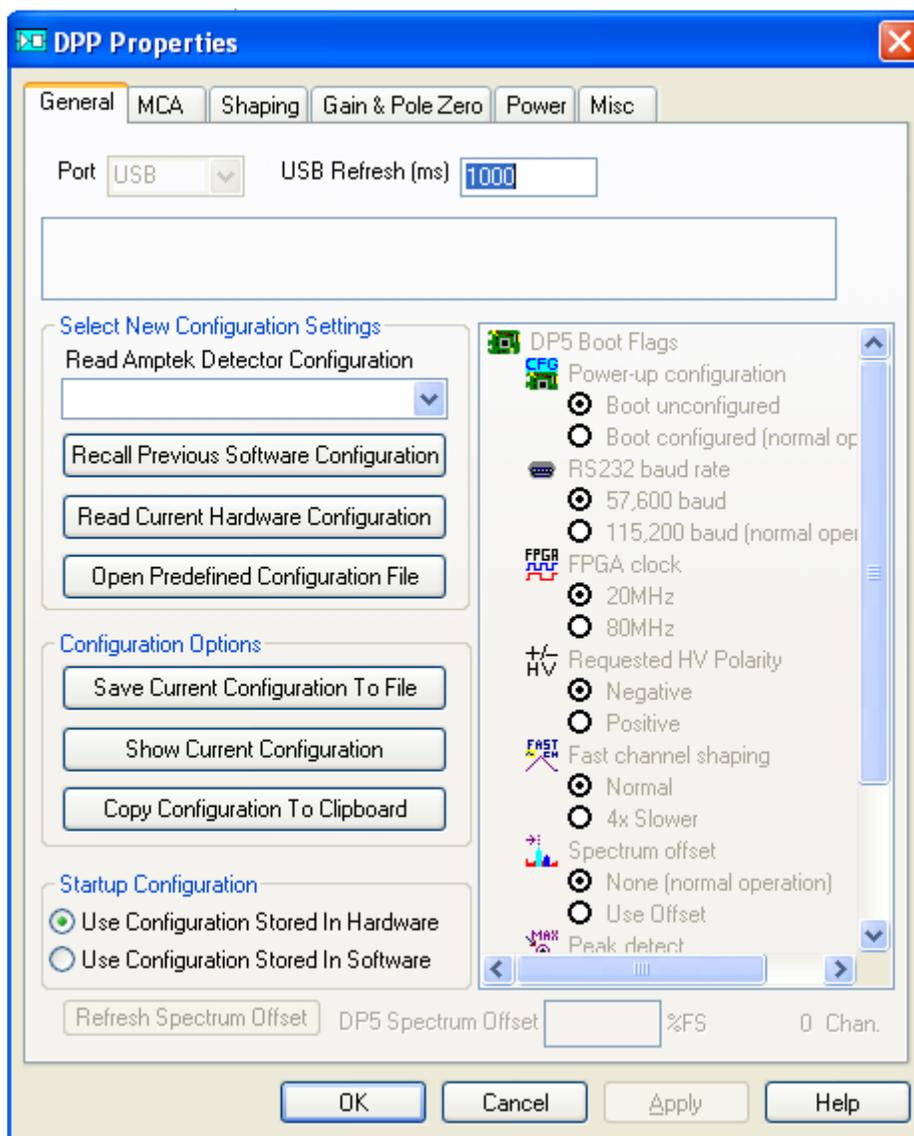


FIG.C2.

In the drop-down menu under “Read Amptek Detector Configuration”, select the Si 13mm²/300μm detector settings. Reload these every time you (re-)open the software, or when you want to revert to the default settings after changing something – these are the working and optimized settings for the detector. Click Apply, click OK.

2 – MCA (MCA settings).

Switching from General to MCA window, you will find three main settings: the “MCA Channels”, the “Preset Time (sec)” and “Slow Threshold” (Fig. C3).

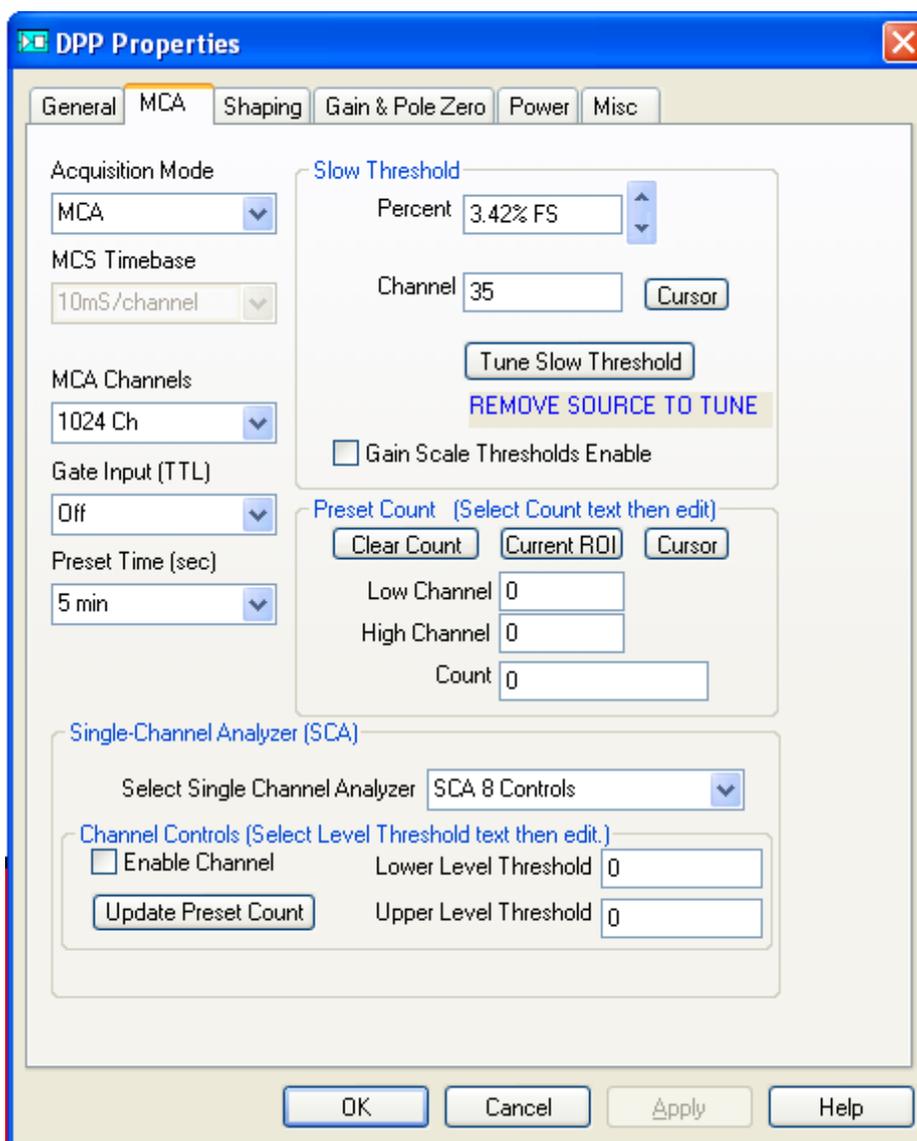


FIG.C3.

MCA Channels

The number of channels does not affect the resolution or energy range of the spectrum; it only makes the spectrum more or less refined. If the peaks being taken are not smooth, then you can decrease the number of channels (or increase the acquisition time). If the FWHM (Full Width at Half Maximum) of the spectrum is less than 5 channels, then increase the number of channels.

Preset Time

Fairly obvious, you can preset the acquisition time here. The acquisition should be run long enough for all the peaks being considered to have a small error, and therefore a large area. An area of a few hundred counts is usually sufficient.

Slow Threshold

The slow threshold is the channel (or percentage of the number of channels) below which no counts are taken. Electrical noise manifests as low-amplitude, low-energy pulses, and is consequently recorded as counts in the low-energy channels. The slow threshold should ideally be set such that it is just above this noise.

The slow threshold can be set automatically or manually. To set it automatically, remove any radiation sources from in front of the detector, start an acquisition, and click “*Tune Slow Threshold*”. It is also tuned when the  button is pressed on the toolbar (again, an acquisition must be running).

To set the slow threshold manually, start a source-less acquisition, click on the first channel, and press F8. This sets the slow threshold to the first channel. You should see the noise peak start growing immediately. Click a few channels above the highest-energy noise, and press F8 again.

You should go through the exercise of manually tuning the slow threshold in order to be able to recognize and remove the noise peak if you see it in the future.

The slow threshold (and fast threshold and input offset) should be retuned every time you change the gain, the peaking time, or the high voltage bias.

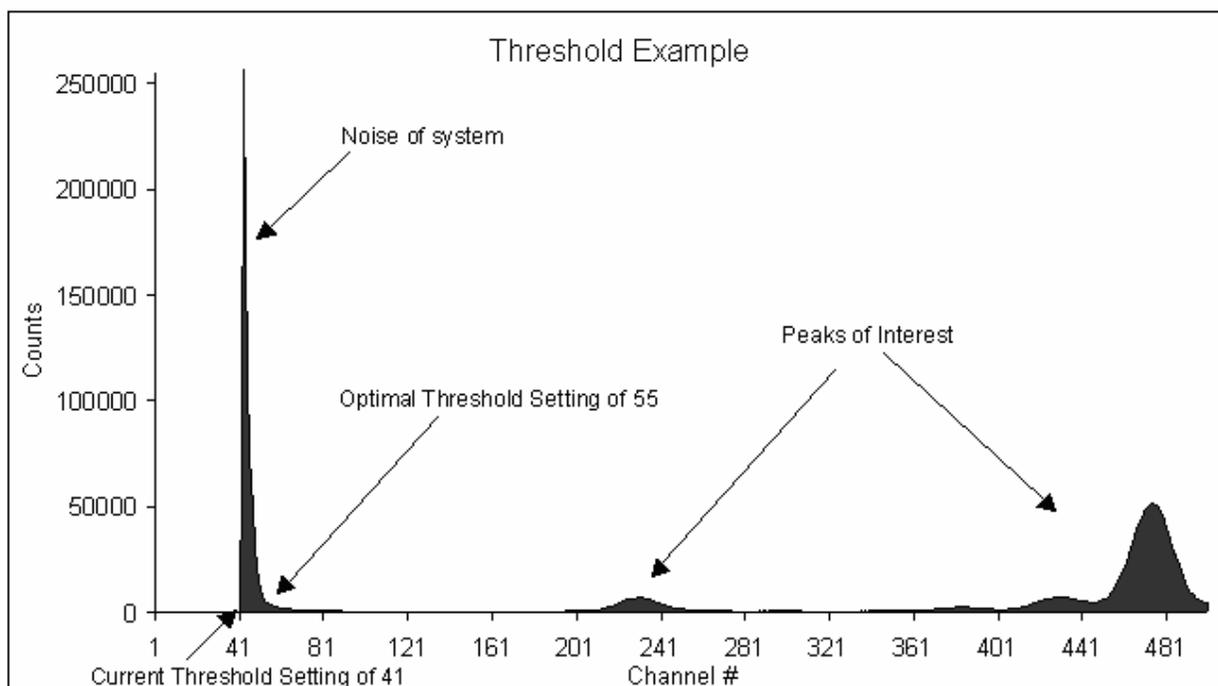


FIG.C4.

3 – Shaping.

The main settings here are the “*Peaking Time*”, “*Pile-Up Rejection*”, “*Fast Threshold*” and the “*Rise Time Discrimination*”, seen in Fig. C5.

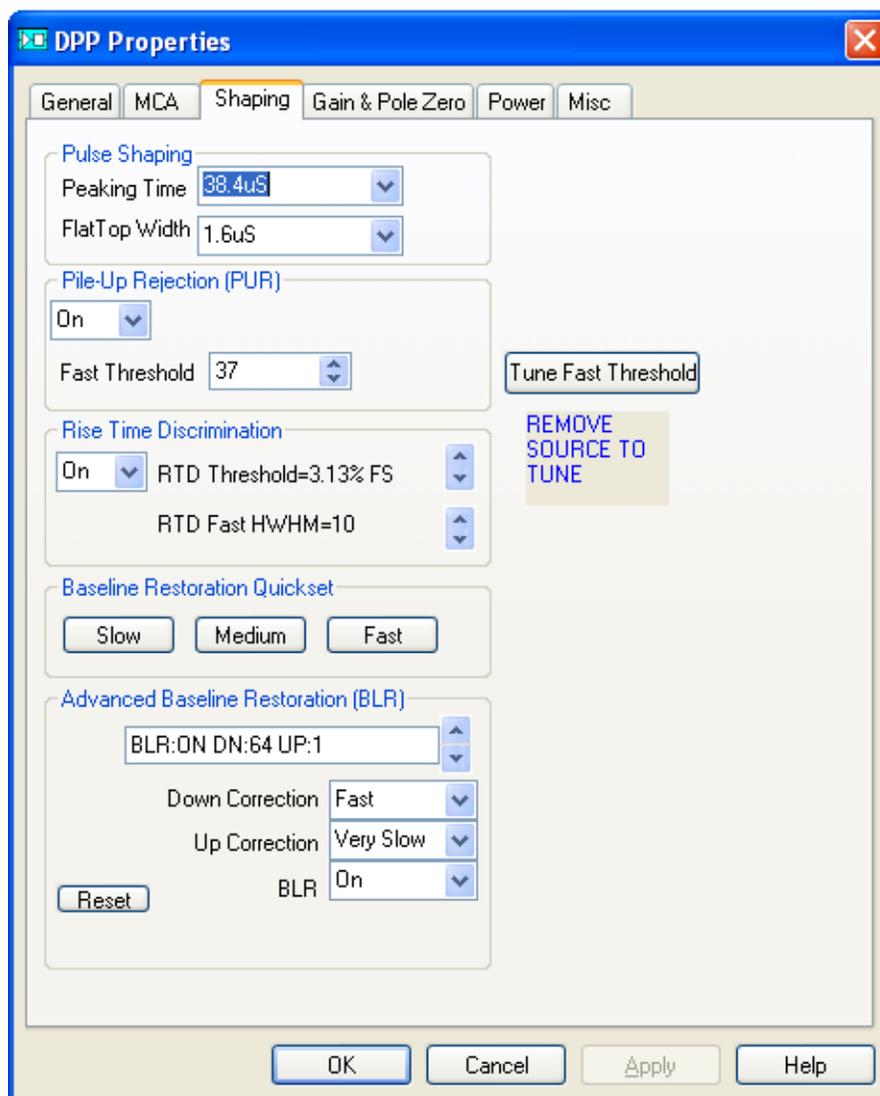


FIG.C5.

Peaking Time

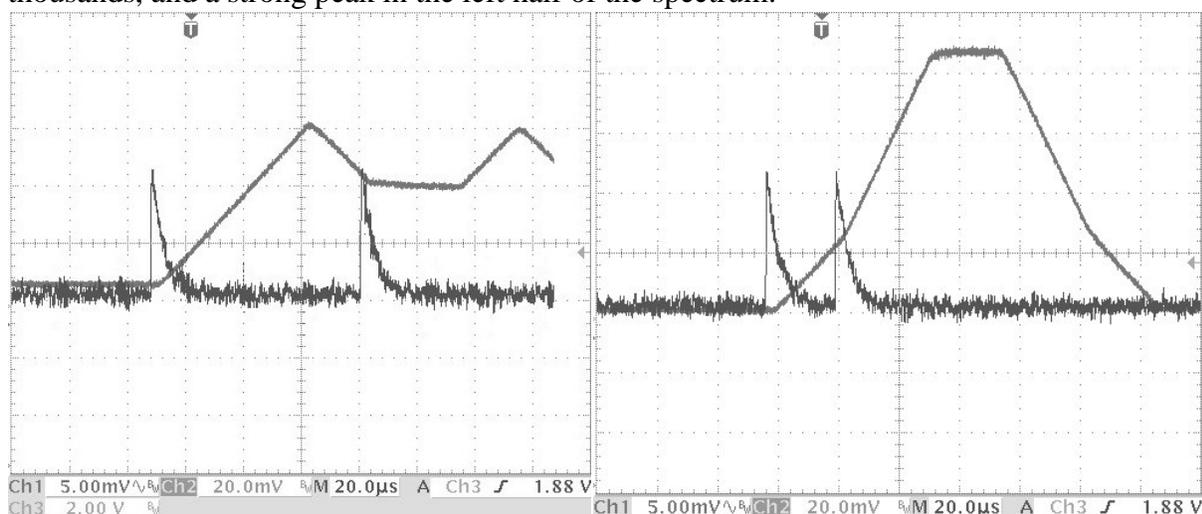
Most likely, you will never change this. The peaking time is directly proportional to the width of the pulse that is recorded by the detector. You want the pulses to overlap as rarely as possible, in order to not have to deal with pile-ups. In this respect, smaller peaking times are better. However, peaking times below $12.8 \mu\text{s}$ begin to decrease the resolution of the spectrum, so only use lower peaking times if the dead time of the spectrum (percentage of discarded counts) is above $\sim 20\%$.

There are other ways to deal with a large dead time: a) decrease the current, b) move the sample and X-ray tube further apart, or c) move the detector and sample further apart. Note that c) will probably cause you to lose low-energy peaks as they are attenuated more in air than are high-energy peaks. Only if these options are exhausted or unavailable should you decrease the peaking time. Also, before doing any of this, make sure your fast threshold is not too low, as this could result in a spurious input count.

Pile-Up Rejection (PUR)

Understanding this requires some knowledge on the inner workings of the detector. It has two parallel processing channels: the fast channel and the slow channel. The fast channel essentially sees every pulse that is separated by at least $0.4 \mu\text{s}$ but cannot accurately measure their amplitude (energy). The slow channel can only resolve pulses that are separated by at least the peaking time, which is larger than the $0.4 \mu\text{s}$ of the fast channel. If two pulses in the slow channel are separated by, for example, $0.6 \mu\text{s}$, the fast channel see two pulses, while the slow channel will only see one pulse with an amplitude equal to the sum of the two pulses. With PUR turned off, this sum-pulse is recorded in a high-energy channel. With PUR turned on, the pulse is rejected. The two fast channel counts are still counted as input counts, but total counts (the number of recorded pulses) are unchanged.

In practice, PUR should always be turned on. The only reason to turn it off would be if you were interested in seeing the pile-up peaks, in which case you need an input count rate at least in the thousands, and a strong peak in the left half of the spectrum.



(a)

(b)

FIG.C6.

Fast Threshold

The fast channel (see PUR) also measures the amplitude, but not as accurately as the slow channel. However, to get an accurate input count, it is still necessary to distinguish electrical noise from actual counts, thus a fast threshold is implemented which is identical to the slow threshold in the slow channel. Fast channel pulses that map to a channel below the fast threshold are rejected – counted as neither input counts nor total counts, even if a pulse occurred in the slow channel. Thus, a fast channel that is too high would cause legitimate pulses in the slow channel to be rejected, while a fast channel that is too low would cause an inaccurate measured input count, as the fast channel would trigger on electrical noise.

The fast threshold can be set automatically or manually. To automatically set it, you can click the “*Tune Fast Threshold*” button in the Shaping tab of the Acquisition set-up, or it is automatically tuned when you click  in the toolbar.

To manually set the fast threshold, remove any source from in front of the detector, start an acquisition, and observe the input counts. You want the input counts to increase by 2-5 counts per second. If they are increasing faster, increase the fast threshold. If they are increasing slower or not at all, decrease the fast threshold.

The fast threshold (and slow threshold and input offset) should be retuned every time you change the gain, the peaking time, or the high voltage bias.

Rise Time Discrimination (RTD)

In this 300 μm detector, the back 100 μm of Si is only partially depleted, so if an X-ray interacts here and creates free electron-hole pairs, the charge is collected / swept away more slowly than it is in the depleted region, causing a slower-rising lower-amplitude pulse. RTD discards such pulses that have a half-width at half-maximum greater than the specified “RTD Fast HWHM”. Via trial and error, the best setting is consistently (for various peaking times and gains) HWHM = 10.

The RTD Threshold is the channel (percentage of total channels) above which RTD begins to get activated.

RTD should be turned on at all times, especially if there are high-energy (above 10keV) peaks. If the dead time is very large, then RTD can be turned off to speed up data collection, at the price of some inaccuracy (in addition to skewing the spectrum slightly towards low energies, it causes the assumed value of the detector efficiency to be incorrect).

4 – Gain and Pole Zero.

The main settings here are “Gain” and “Input Offset” (Fig.C7). The rest of the settings should not be changed.

Gain

Pulse amplitudes are multiplied by the gain before being mapped to different channels. In essence, a pulse at a lower gain will map to a lower channel than the same pulse would at a higher gain.

There is a different channel-energy calibration for each different gain value. They can be found by making a linear fit of at least two known peaks of a spectrum taken at the gain value in question (recommended), or consulting the table below (accuracy dubious, may depend on other settings):

Gain	Calibration at 1024 channels: channel number = const ₁ E + const ₂ (± 2 channels)
91.84	96.088 *E + 12.314
76.68	80.323 * E + 9.8454
60.30	62.996 * E + 7.3636
49.92	52.215 * E + 5.557
41.58	43.465 * E + 4.6135
34.72	36.359 * E + 3.1665
30.30	31.691 * E + 2.8848
25.09	26.227 * E + 2.4313
20.90	21.833 * E + 1.92
17.46	18.24 * E + 1.6674
14.27	15.014 * E + 1.2661
11.82	12.41 * E + 1.1738
9.84	10.327 * E + 0.9639

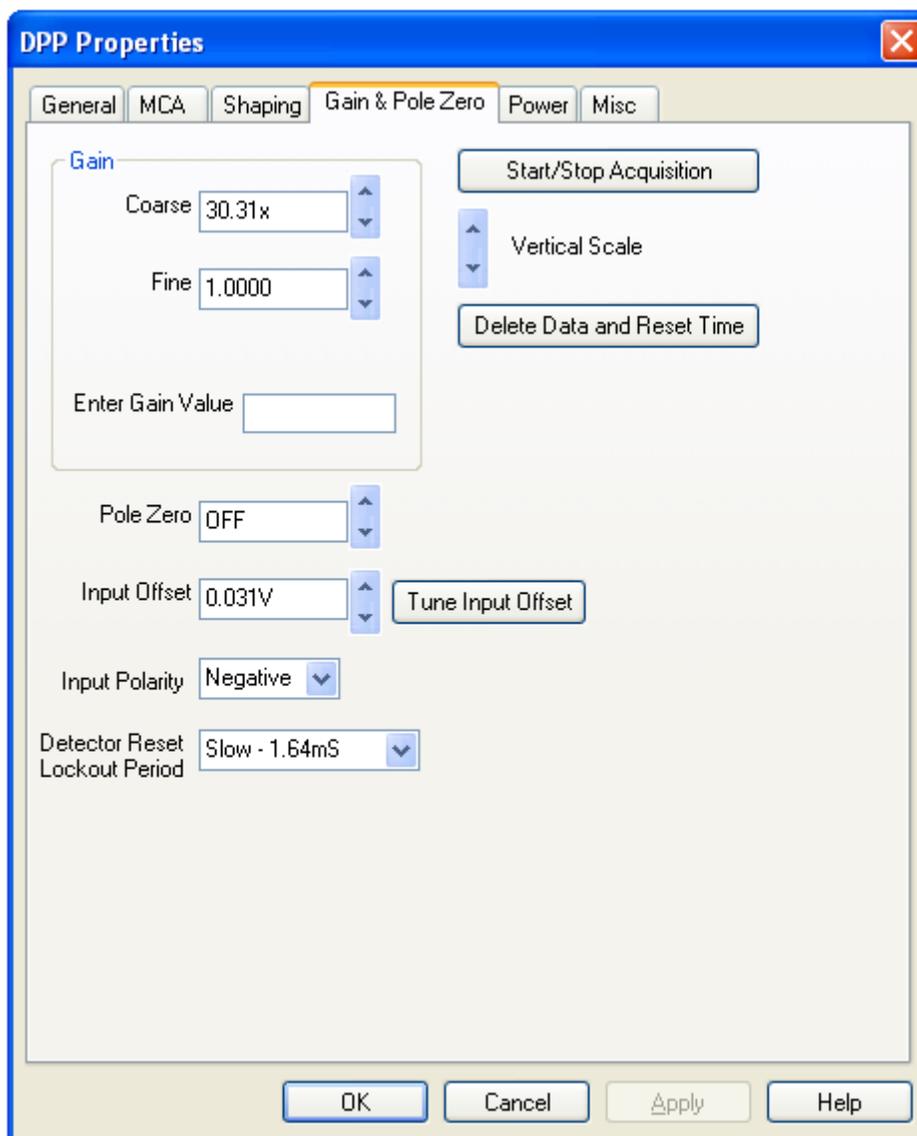


FIG.C7.

The gain should be selected such that the highest-energy peak that you expect to see appears in the spectrum. To see any peak that can be excited by a 30-kV X-ray tube, choose 30.30x gain.

Input Offset

The input offset (and slow and fast thresholds) should be tuned whenever the gain is changed, with no source in front of the detector.

5 – Power.

Relevant settings here are the “Cooler Temperature” and the “High Voltage Bias” (see Fig.C8).

Cooler Temperature

The colder, the better. As it happens, this detector has a two-stage Peltier cooling system, so it can cool itself to 75-80 K below room temperature (i.e. to 213-218 K). As long as the temperature is relatively constant (fluctuations by a few degrees are okay), the spectra will be consistent.

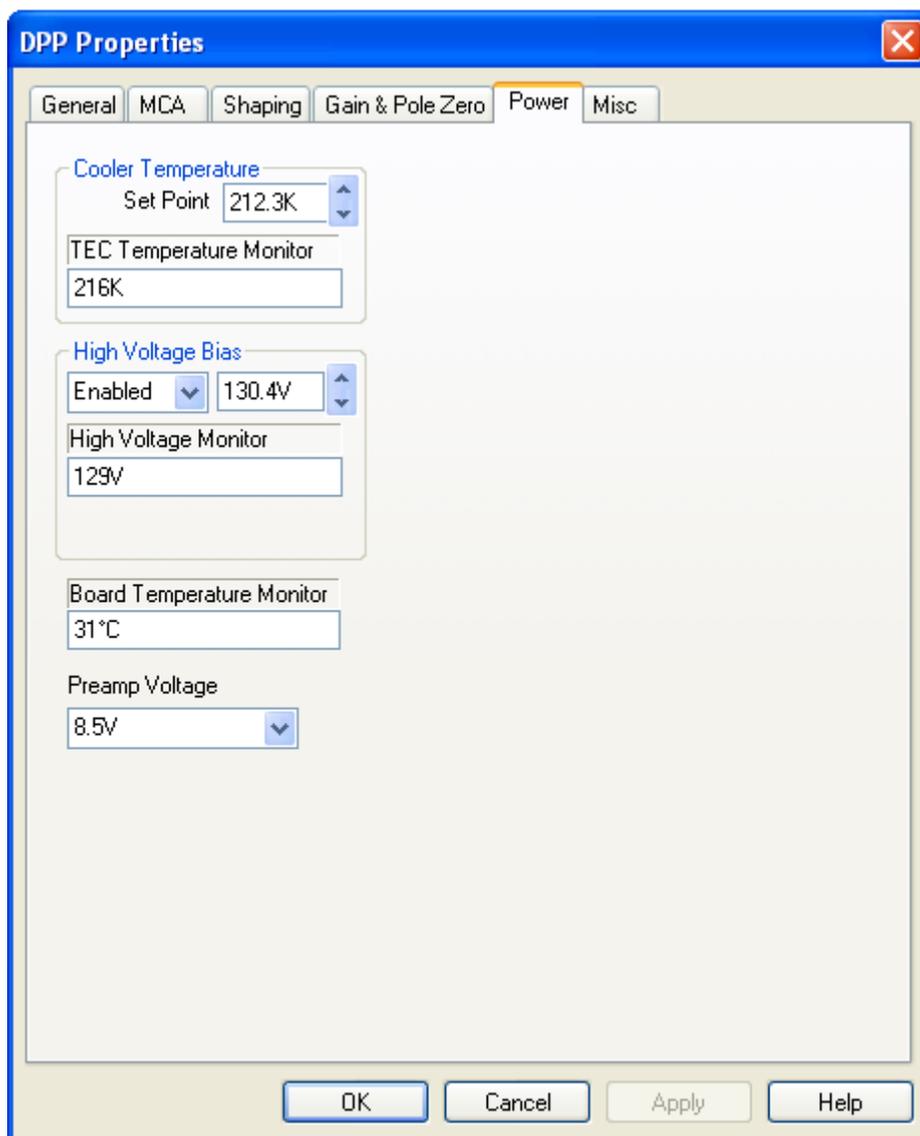


FIG. C8.

High Voltage Bias (HV Bias)

It is not recommended that you change the HV bias from 129 V. If it is decreased, you may lose the pulse and be unable to restore it without turning off the detector, waiting a few minutes, turning it back on and tuning fast and slow thresholds if necessary. If it is increased, you will have decreased resolution, and when decreasing it back later you may again lose the pulse and have to troubleshoot again.

The disappearing pulse issue has never occurred when the detector is turned on and the HV bias is immediately set to 129 V, so that is the recommended course of action.

6 – Misc.

There isn't much of interest in the miscellaneous settings, except just one: the virtual oscilloscope can show whether the signal is being registered by the detector. This can be useful during troubleshooting when the source of absence of a signal is to be determined.