STABILITY IN AMPTEK'S DETECTORS AND DIGITAL PULSE PROCESSORS

Stability is an important concern in X-ray spectroscopy. Anything that changes the photopeak count rates, other than the intensity of the characteristic X-ray lines to be measured, can cause errors. If the gain, offset, or energy resolution change, then regions of interest will be incorrect, peaks may be misidentified, overlaps may not be correct, etc.

When properly configured, Amptek's family of solid state detectors and electronics are extremely stable. The goal of this application note is to explain how to obtain stable operation and then to quantify the expected drifts. Note that scintillator/PMT systems will exhibit important changes with temperature, time, and other parameters. This application note applies only to semiconductor detector systems, including Si-PIN, SDD and CdTe detectors.

Summary

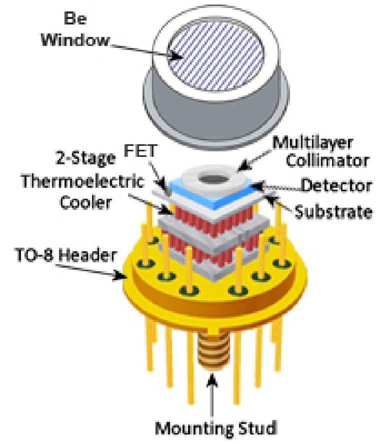
- The key to stable operation is the detector temperature. The detector temperature dominates the temperature coefficients and long term stability: if the detector temperature changes, then the energy resolution, system conversion gain, and offset will change significantly.
- If the detector temperature is held constant, i.e. the temperature controller is always regulating, then the rest of the system contributes coefficients of tens of ppm per °C (the exact value will vary due to component tolerances). This is small but nonzero so users must prepare for such drift.

	Typical gain temperature coefficient (ppm/°C)	
	T_{DET} fixed	T_{DET} not fixed
X-123 (all detector types)[1]	0 to 50	100 – 150
DP5	0 to 40	100 – 150
DP5 & XR-100 (all detector types)	0 to 50	100 – 150
PX5	0 to 40	100 – 150
PX5 & XR-100 (all detector types)	0 to 50	100 – 150

- Amptek's processors are designed to minimize changes in the spectrum with count rate. In general, a properly configured X-ray spectrometer below 50% dead time will exhibit small but nonzero shifts in gain, offset, and resolution. At input count rates above 50% dead time, changes in the spectrum are more likely to be important.
- If any configuration parameter is changed, then the energy resolution, system conversion gain, and offset may change. Users sometimes change a parameter and assume it is negligible, but this should never be taken for granted.
- Users may also make changes to the system, external to the spectrometer, without realizing the consequences. Photopeak count rates are very sensitive to changes in the sample to detector geometry. Photopeak count rates may depend on the overall input spectrum, depending on the algorithms used.

[1] For the purposes of this application note, the AXR detector with PA-210 or PA-230 preamplifier and DP5/PC5 digital pulse processor and power supply are considered equivalent to the X-123.

Thermal stability



As shown on the right, Amptek's detectors use a thermoelectric cooler to minimize electronic noise without the use of cryogenics. Our power supplies permit control of the detector temperature. To minimize drifts with time and temperature, the user must (1) set the temperature so that the detector remains at a constant, regulated temperature and (2) properly heat sink the detector.

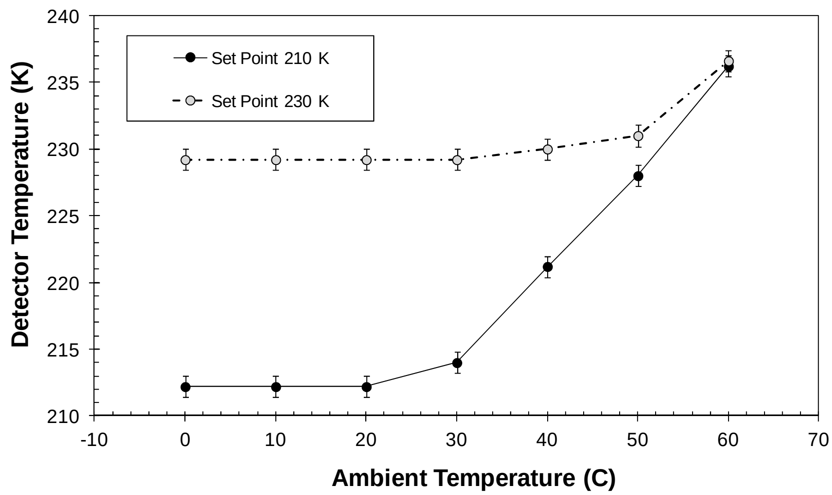
The thermoelectric coolers can achieve a maximum temperature differential of >75°C. To set the temperature properly, first determine the maximum temperature at which the detector will be used. This is the temperature of the detector's mounting stud, which is often higher than ambient. A detector mounted in an enclosure can easily run 10°C above ambient. Set the detector temperature 65°C lower than this maximum, leaving at least 5°C for margin. The system can now regulate the detector temperature over all ambient conditions.

The very best resolution is obtained at the lowest temperature, thus some users set the temperature at its minimum, e.g. 200 K. This ensures that the cooler is always running at full capacity. This setting achieves the highest resolution, but sacrifices stability when ambient temperature rises. The user must choose which is better for the particular application. Some analytical software, such as Amptek's XRS-FP, compensates for gain drifts and changing resolution, while other programs software cannot accommodate such changes. The user must understand the system requirements and then select the best configuration.

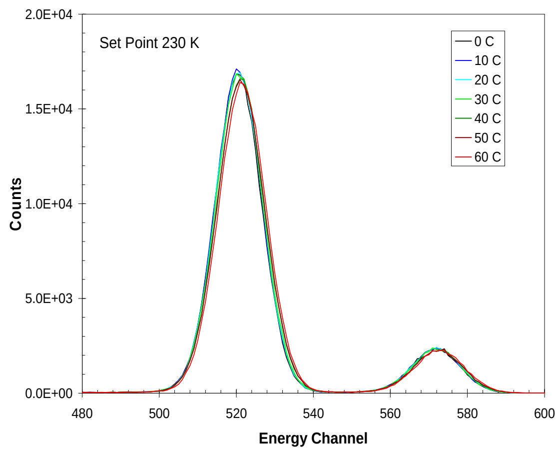
The thermoelectric cooler removes heat from the detector and transfers it to the mounting stud and the back of the detector hybrid. The detector must be connected to a heat sink, which must be kept as cool as possible to achieve the best performance. Thermal resistance between the mounting stud and the ambient must be as low as possible. Users sometimes attach the stud to a metal plate, but then put this plate in an enclosure with no airflow or external heat sink, thermally isolating the detector from the outside air.

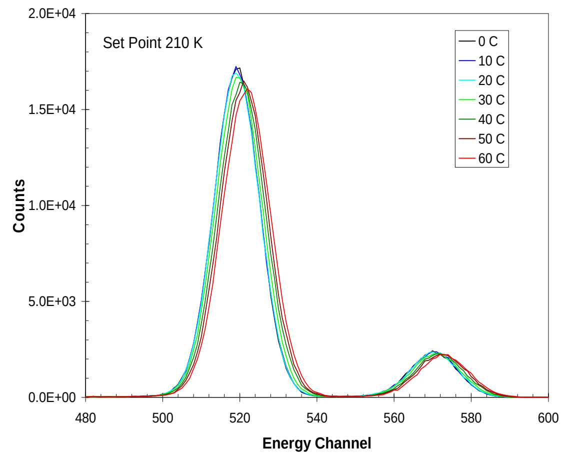
Thermal stability and gain

The figure below shows the detector temperature versus ambient temperature for two different detector temperature settings. When set to 230K, this unit maintained a constant detector temperature up to ambient temperatures of 50°C. When the set point was lowered to 210K, it was stable to 20°C but not at higher ambient temperatures. Note that the "ambient" is the temperature of the detector heat sink; this is often located inside an enclosure so is warmer than the room temperature.



The plots below show the consequences of the gain shifts for spectroscopy. The plot on the left shows the 5.9 keV ^{55}Fe photopeak over a range of ambient temperatures, with a set point of 230K. The peak shift is negligible. The plot on the right shows the results for a set point of 210K and the peak shift is quite noticeable.



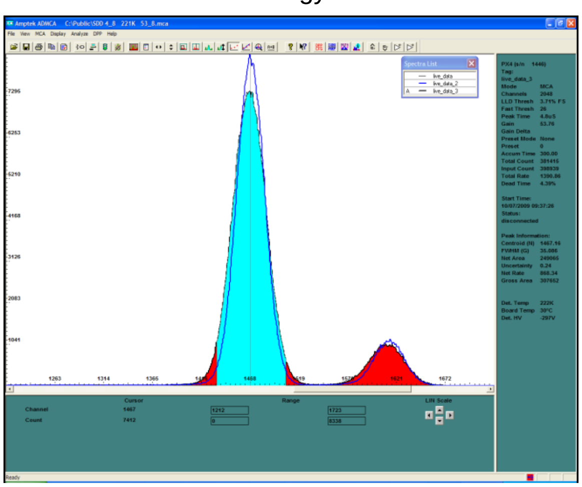


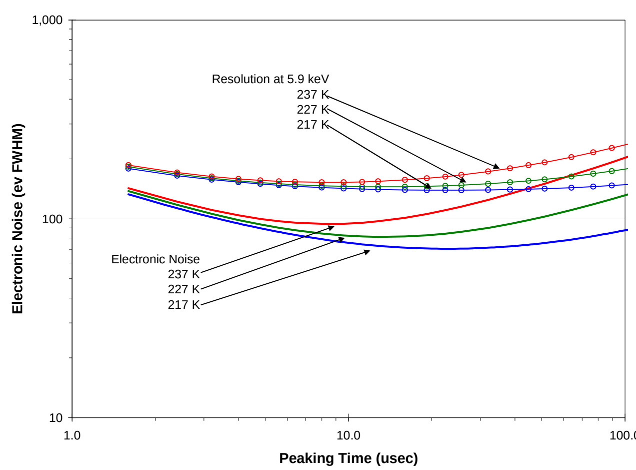
If detector temperature is held constant, then gain shifts are much smaller but are still nonzero. Consider a specific example: at 25 keV, the resolution of an SDD is about 250 eV. The signal processor may have a tempco of 50 ppm/^{o}C, and in a handheld instrument, the temperature may change by 20^{o}C. This shifts the centroid by 25 eV or 10% of the FWHM which is likely to be important.

Thermal stability of energy resolution and spectrum offset

It the energy resolution of a detector changes, then the photopeak count rate computed from a region of interest will likely change. This is illustrated in the spectrum below, on the left: if the peak is broader and one integrates the counts within a fixed window, the computed count rate decreases as FWHM increases.

The plot on the right below shows the electronic noise and the energy resolution at the Mn K_{α} line (5.895 keV) as a function of detector temperature and of peaking time for a given detector. If the detector is operated at full cooling and the optimum T_{peak}, and the detector temperature changes, electronic noise changes quite noticeably. The impact on resolution is less due to Fano broadening. At shorter peaking times the temperature coefficient is reduced. If the detector temperature is held constant (i.e. the set point is adjusted for stability as discussed above), the change in the temperature of the electronics has no measurable effect on energy resolution.

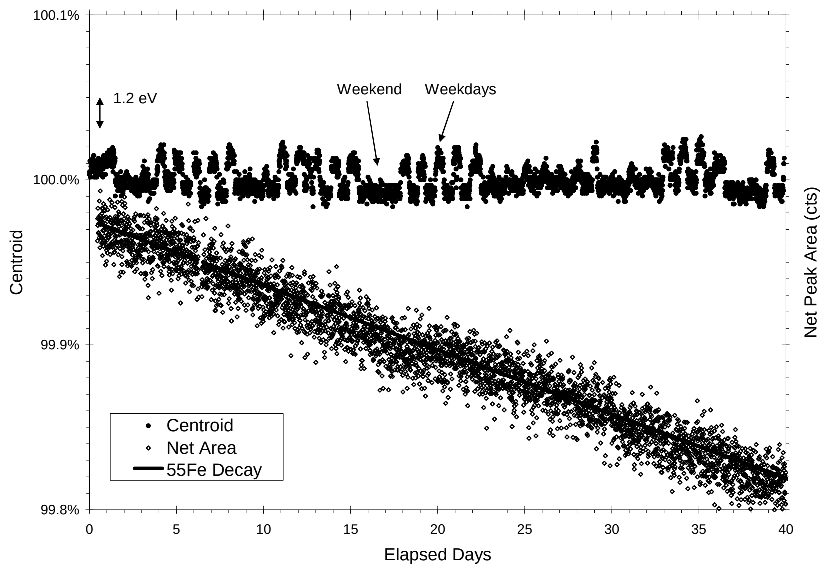




Many customers assume that the spectrum has no offset, i.e. that channel zero corresponds to zero energy, but this is not true. Any real electronic system has some offset. In Amptek's digital processors, the "zero energy" point will be greater than channel zero by about the FWHM of the noise. If the electronic noise changes for any reason, the offset will change commensurately. If detector temperature changes, then electronic noise changes, and spectrum offset changes. This is one of the reasons why turnkey ED-XRF analyzers have a detector "calibration" (or standardization) process.

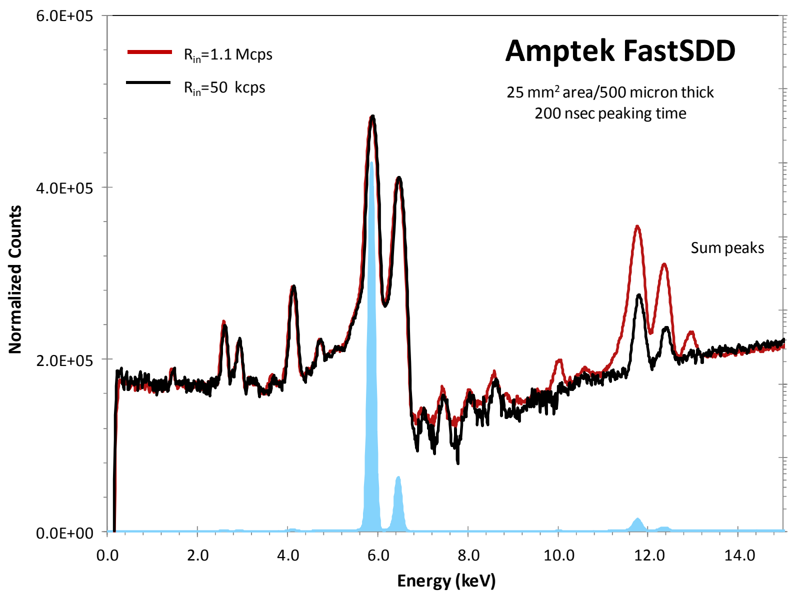
Drift with time

There is no measurable long-term drift in the gain of Amptek's systems. The plot below shows a 7 week measurement of ^{55}Fe centroid and count rate, with a fixed detector temperature. The centroid exhibits a diurnal variation of +/- 160 ppm, or 1 eV. During the work week, the thermostat was set back by 5°C overnight, while over the weekend, the thermostat is constant. The 160 ppm shift clearly results from a 32 ppm/°C tempco of the signal processor, which is expected. The slope of the centroid versus time is zero (the uncertainty in the regression is several times larger than the slope). The plot also shows that the count rate decreases with time, according to the half-life of the ^{55}Fe source.



Stability with count rate

The plot below shows two spectra measured from a 99.9% pure Mn target using an Amptek FAST SDD™, at T_{peak} of 200 ns. One is measured at 5x10^4 cps input count rate and the other at 1.1x10^6 cps input count rate, with a dead time of 66%. The main change in the spectrum is the increased sum peak at the higher rate but the Mn K_α line also shifted down by 30 eV. If the rate is increased enough, the centroid will shift more and the FWHM will also degrade.

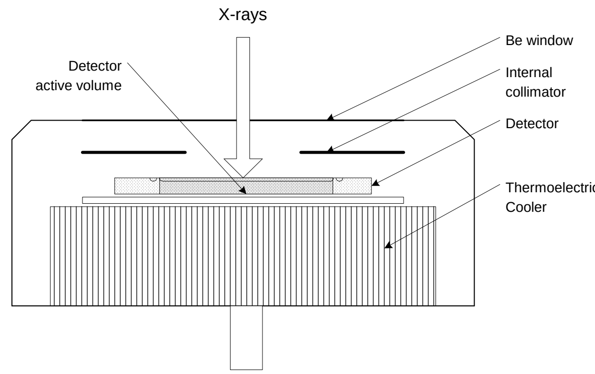


Other changes in measured count rates

There are many reasons why the count rate in a system may vary even when the spectrometer, the detector and signal processor, are stable. Users often attribute changes to the spectrometer when other causes are at work so it is useful to consider some of these other causes.

Geometric sensitivity of the detector

Some customers have asked if the geometric sensitivity of the detector itself can change. The answer is "No". The detector's sensitivity is determined by its active volume and by the transmission of X-rays through the Be (or C-Series) window. These are geometric quantities, determined by the thickness of the silicon wafer, the size of the collimator, and the thickness of the detector window. These will not change over time, temperature or anything else.



System geometric stability

Changes in the geometry of the X-ray source, sample, and detector can have a large effect on count rates. In a backscatter geometry, the count rate varies as $1/r^4$: moving the sample away reduces the flux on the sample by $1/r^2$ and then the flux from the sample to the detector by $1/r^2$. If your sample is 1 cm away and you change the position by 0.5 mm, the count rate changes by 20%. If you change the position by 50 microns, the count rate changes by 2%.

Source stability

With a radioactive source, the intensity will vary with time according to the half-life of the source. This is very predictable so corrections are quite straightforward. In the seven week measurement above, notice the clear change in count rate in just one day, for a source with a 2.7 year half life.

Most X-ray spectroscopy applications use X-ray tubes or electron beams as the source due to their greater intensity but they are less stable than radioactive sources. There are many possible causes for X-ray tube flux to vary: changes in power voltage, noise on control lines, thermal expansion of electron optics, tube aging, etc. For debugging purposes, use an isotopic source.

Changes in the spectrum

A photopeak is nearly always superimposed on background in the spectrum. The background spectrum may change, e.g. due to changes in beam energy or the matrix of the sample. If the background were perfectly removed, the shape of the background spectrum would not affect the calculation of photopeak area. In practice, background removal algorithms are imperfect so changes in the background spectrum may result in change in the calculation of the photopeak area.

Processor configuration

Users sometimes change a parameter in the DPPMCA software and expect no change in the gain or offset but this should not be assumed. A change in peaking time will affect the noise and this will affect the offset. A change in HV bias can affect collection time and thus system gain.

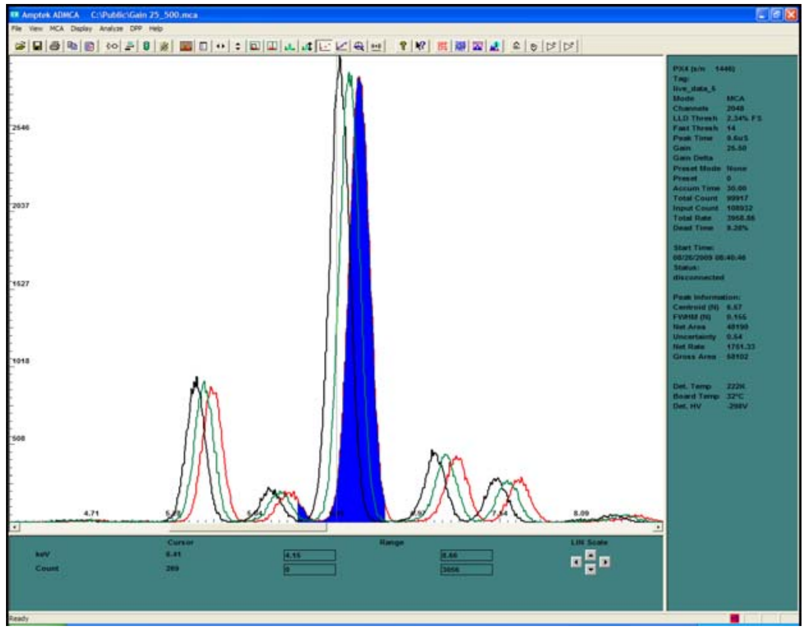
Whenever the Amptek processor is reconfigured, the baseline restorer has to find and reset the baseline and there can be variations in the baseline to which it settles. Some customers reconfigure the hardware for every spectrum, an operating mode likely to cause variations. The long term stability shown above is only assured without reconfigurations.

Interference

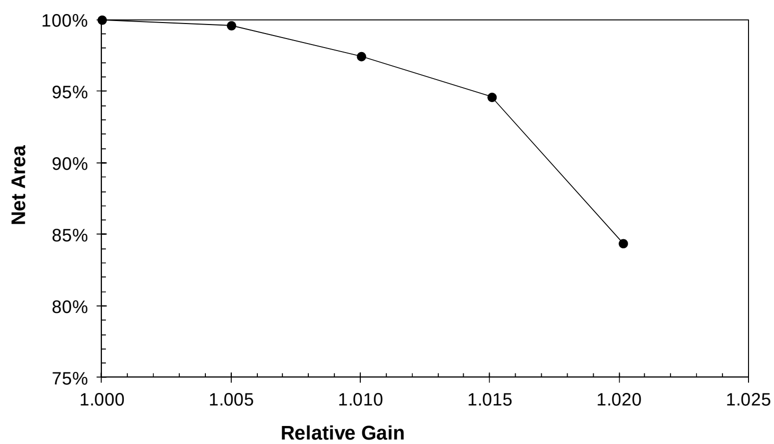
An X-ray spectrometer is often used around other equipment: high voltage power supplies for X-ray tubes, vacuum pumps, etc. This equipment often produces a small level electromagnetic interference which produces a small amount of noise in the detector. It may also produce acoustic vibrations which induce a microphonic response in the detector. A change in external equipment, i.e. turning on a nearby air conditioner, may cause a change in the noise of the spectrometer. This, in turn, will affect both resolution and spectrum offset and cause a change in the calculated photopeak counts.

Why does stability matter?

The plots below show the consequences of changing gain. The plot on the left shows steel spectra in which the gain changes in steps of 1% (with resolution unchanged). The region of interest (ROI) for the Fe K_α peak was marked for the lowest gain. With a 2% change, the ROI clearly includes counts from the adjacent Cr K_α peak and misses counts on the upper portion of the Fe K_α peak. Note that the number of counts in the peak channel decreases as the gain increases. The total counts are actually the same, but are spread across more channels. The plot on the right shows the net area in the photopeak versus gain.



Clearly, the number of counts within a fixed ROI will change as either gain or resolution changes. Since analysis software often uses counts in an ROI to indicate elemental intensity, these results would change. If one uses the centroids of the peaks to identify the elements in a sample, then if the centroid shifts enough, the peak will be misidentified. If the software can rescale the ROIs to match the changing gain, or if each spectrum is recalibrated, then gain shifts become much less important.



How important is stability?

This is a difficult question to answer because it depends very strongly on the details of an application and on the software used for analysis. If one has several closely spaced peaks and the counts in a fixed ROI are used to determine concentration, stability can be very important. One the other hand, if the spectrum is simple and ROIs can be set wide, then the spectrum can shift some with no consequence. Amptek's XRS-FP software performs a Gaussian fit to the peaks, and if the nonlinear deconvolution is selected, it will adjust the gain, offset, and widths of the peaks (within user-defined limits). Spectrum shifting can be readily accommodated. Since the most stable configuration will, in general, have higher electronic noise, the user needs to determine what matters most for the application.

Understanding gain stability

The system conversion gain is the ratio of the output (in MCA channels) to the input (in energy). If an X-ray deposits energy E in the detector, the output is a count in channel N_{CH}, given by

$$N_{CH} = \left(\frac{E_{dep}}{\varepsilon_{pair}}\right)\left(\frac{1}{C_F}\right)\left(G_{AMP}G_{ADC}F_{Norm}G_{MCA}\right)$$

where ε_{pair} is the energy needed to create one electron-hole pair (3.6 eV in Si), C_F is the feedback capacitor in the charge sensitive preamplifier, G_{AMP} is the voltage gain of the analog amplifiers, G_{ADC} is the ADC's conversion gain, F_{Norm} is a purely digital normalization factor, and G_{MCA} is the MCA conversion gain (the number of channels). The product $G_{ADC}F_{Norm}G_{MCA}$ has units of channels per volt.

Many users are surprised to find that the most temperature sensitive term is ε_{pair}, the energy required to create an electron-hole pair in silicon, which has a temperature coefficient of about 100 ppm//°C. Many users think that ε_{pair} is a physical constant. But when an X-ray interacts in the detector, it produces a high energy secondary electron which then scatters from the lattice and electrons, producing lattice vibrations (or phonons) and electron-hole pairs. As the temperature increases, there are more phonons, thus the secondary electrons scatter off more phonons, yielding fewer electron hole pairs.

With Amptek's design, the rest of the system has little temperature drift. The feedback capacitor is located in the detector hybrid so its temperature is stabilized along with the detector. The analog amplifiers use very low tempco resistors along with a circuit topology which maximizes stability. Due to variations in the resistors, the tempco of G_{AMP} is not zero but is tens of ppm/deg C. The ADC utilizes a voltage reference which is stable to 20 ppm/deg C. The other terms in G_{MCA} are purely digital so exhibit no temperature drift.

Understanding resolution stability

The resolution δE is given by

$$\delta E^2 = 2.35\left(\varepsilon_{pair} q_e F_{Fano} E\right) + \left(2q_e I_{dark}\right)\left(A_{step}\tau_c\right) + \left(b_n + a_n C_{IN}^2\right)\left(A_{1/f}\right) + \left(\frac{4kT}{\gamma g_m}\right) C_{IN}^2 \left(\frac{A_{delta}}{\tau_c}\right)$$

where F_{Fano} is the Fano factor, E is the X-ray energy, ε_{pair} is the energy to create an electron-hole pair, q_e is the charge on an electro, I_{dark} is the dark current through the detector, τ_p is the peaking time, C_{IN} is the input capacitance, k is the Boltzmann constant, g_m is the transconductance of the input FET, b_n and a_n characterize 1/f noise sources, and A_{step}, $A_{1/f}$, and A_{delta} are constants which characterize the pulse shaping network. There are several key points from this simple equation and plot.

- The first term is not noise but arises from statistical fluctuations in the charge generation process. For many energies, this dominates the resolution, in which case neither the peaking time nor the temperature makes much difference. One can go to fairly short peaking times and high temperatures with little resolution degradation.
- The electronic noise is the sum (in quadrature) of three terms: white parallel noise increases with τ_p, white series noise decreases with τ_p, and 1/f noise is independent of τ_p. The noise therefore must exhibit a minimum at an optimum peaking time (the noise corner).
- At peaking times shorter than the noise corner, the noise is dominated by white series noise from the preamplifier. This has only a weak temperature dependence. Since throughput is improved at short peaking times, we usually suggest using the shortest peaking time which provides the needed resolution. This will improve both throughput and stability.
- At peaking times beyond the optimum, the noise is dominated by shot noise from the detector's leakage current. This is an exponential function of detector temperature. If one operates at the noise corner, the optimum peaking time, then temperature variations are quite important. Note that the optimum peaking time decreases at increasing temperature.