**BioCAT: Basic Techniques for EXAFS**
Revision date 8/11/88 G.Bunker†

## Sources of Noise in EXAFS Experiments

Noise in EXAFS experiments has a number of sources, among them unavoidable random fluctuations in the number of photons absorbed by the detectors (“photon counting noise”), electronic noise, and noise that arises from sensitivity to fluctuations of the x-ray beam. In the typical EXAFS experiment, one acknowledges the fact that the incident x-ray intensity varies with time, and attempts to compensate for it by dividing the measured signal by the measured incident flux, each integrated over the same time interval. A number of experimental problems often interfere with exact compensation for such beam intensity fluctuations. Most of these problems can be avoided if care is taken, but they can (and often do) cause glitches, noise, and distorted amplitudes if they are ignored. The most important of these problems can be summarized in the mnemonic HALO: *Harmonics, Alignment, Linearity, Offsets.* It is a good idea to keep these in mind during the experiment, so they can be systematically dealt with.

In order to precisely compensate for the incident intensity fluctuations, it is necessary that the $I_0$ and $I_f$[1] detectors “see” exactly the same beam, in the same way. If harmonics are present in the incident beam, the $I_0$ and $I_f$ detectors will in general measure different proportions of the harmonic radiation and the fundamental, *even if the detectors are identical*. The reasons are quite simple. The $I_f$ detector senses the fluorescence photons (which are essentially of a single energy), plus scattered background (most of which is elastic scattering at the excitation energy, but some of which is inelastically scattered radiation at lower energies), plus scattered harmonic radiation. On the other hand, if the $I_0$ detector is an ion chamber, it measures the x-ray intensity at the excitation energy (the fundamental), and also some small fraction of the harmonic radiation. Thus the $I_0$ and $I_f$ detectors, in effect, see different beams. Even if one uses a scatterer and detector as shown in figure 1, instead of using an ion chamber for $I_0$, the $I_f$ and $I_0$ detectors “see” beams of different spectral content. The result is that fluctuations in the incident beam intensity do not divide out perfectly. For this reason it is essential to minimize the harmonic content of the beam, either by detuning the monochromator, or by using a harmonic reject mirror. This is the H in HALO.

---

† Acknowledgement: I am indebted to Professor Edward Stern and the rest of the UW EXAFS group, especially C.E. Bouldin, B. Bunker, W.T. Elam, S. M. Heald, and K. Kim for many discussions.

[1] In this discussion, $I_0$ denotes the detector that measures the incident flux, and $I_f$ denotes the fluorescence detector. Athough direct reference is made only to fluorescence measurements, many of the points apply to transmission experiments as well.

EXAFS experiment with scatterer as flux monitor

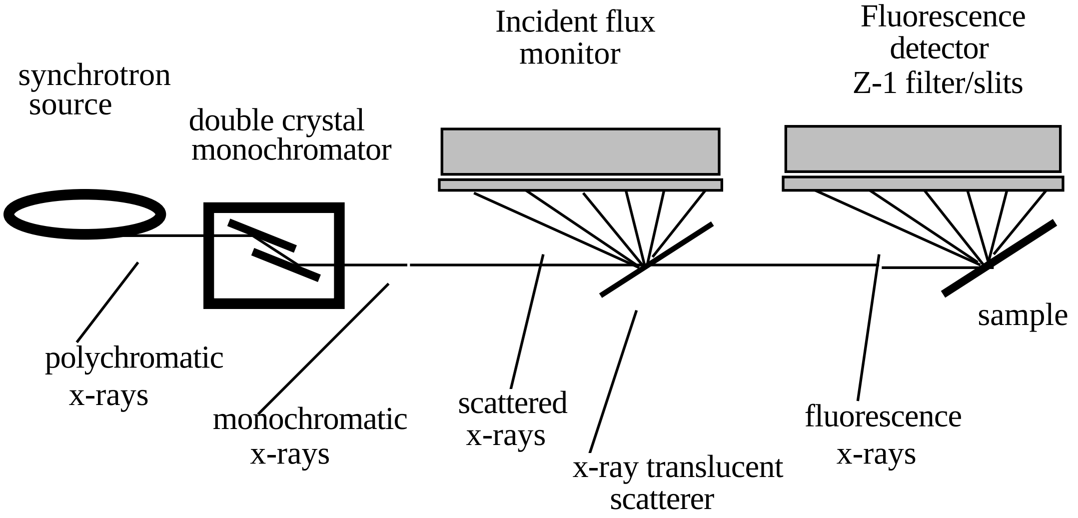

Figure 1

The second major reason why the two detectors may "see different beams" is that the sample alignment may be incorrect. The x-ray beam size and shape should be defined by a slit placed *before* the $I_0$ detector, and nothing except for spatially uniform objects such as smooth windows and a homogeneous sample should intercept the beam after that point. If something does clip the beam between the front detector and the sample, whether it is a lead mask, the edge of the exit aperture of the front ion chamber, the edge of a cryostat entrance window, or even merely a wrinkled cryostat entrance window, then small fluctuations in beam position may cause noise, glitches, or other distortions. Improper alignment is one of the primary causes of glitches in experimental data.

The L in HALO stands for linearity. The intensity fluctuations will divide out only if the detectors and electronics are linear, that is, their outputs are proportional to the inputs (also a constant offset is okay, as long as it is subtracted out). If ion chambers are used, the applied voltage must be high enough that they are in their "plateau region", where the output for a given x-ray flux is independent of high voltage. The value of the plateau voltage depends on the construction of the ion chamber, the photon flux, the fill gas(es), and other variables. Typical values for the plateau voltages are 300–400 V for transmission ion chambers with incident fluxes of about $10^9$–$10^{10}$ photons/sec, and 50–100 V for fluorescence ion chambers absorbing $10^6$–$10^7$ photons/sec. If other types of detectors are used, it is crucial that their linearity also be checked. Finally, the electronics (e.g. amplifiers, voltage to frequency converters) should be operated over their linear ranges by suitably adjusting the gains and offsets. When dealing with small signals, it is often wise to set the amplifier offset to one volt or so, so the signal always is much larger than electronic noise and drift. This offset should be measured and subtracted out by the data collection computer program. For the equipment in common use (e.g. Keithley 427 amplifiers, and Analog Devices V/F converters) it is best to keep signals in the range 1–10 volt.

Finally, it is very important to periodically measure the “offsets” (i.e. the signal produced when the x-ray beam is off) so that they can be subtracted out. Fluctuations in incident intensity will not divide out between $I_0$ and $I_f$ if the proper offsets have not been subtracted. Sometimes offsets can drift, so it is a good idea to check them regularly. If the detectors are sensitive to visible light as well (such as PIN diodes and scintillator/PMT detectors), then the detectors should be well shielded from the light, or at a minimum, the ambient light should be kept as constant as possible.

These are the four aspects of “HALO”. Even after these precautions are taken, some noise is, of course, still present in the data. Some noise is a consequence of residual nonlinearities, which require more advanced modulation techniques (which are beyond the scope of this brief note) to eliminate. Other problems also may potentially exist, such as differing sensitivities of the $I_0$ and $I_f$ detectors to beam position. If one uses an ionization chamber to detect $I_0$, and the average position of the beam (i.e. centroid of the intensity) fluctuates horizontally, the $I_f$ detector will be more sensitive to the fluctuations than will $I_0$, which will result in increased noise. This problem can be corrected by using the symmetrical detector configuration shown in figure 1 (and/or by using modulation techniques).

For dilute specimens, however, the noise is usually dominated by fluctuations in the number of detected photons. If N photons on the average are absorbed per unit time, and they are absorbed independently of each other, and N is much large than one, general statistical arguments indicate that the number of photons actually absorbed per unit time fluctuates by about $\sqrt{N}$ . For example, if one million photons are absorbed on the average, one would expect (root-mean-square) fluctuations of about one thousand.

Such fluctuations are a major source of noise for EXAFS studies on dilute samples. The measured signal consists of the desired fluorescence photons ($N_s$) and scattered background $N_b$ (plus refluorescence from the filter). The noise arises from fluctuations in the total number of photons: noise $\sqrt{N_s+N_b}$ , and thus the signal to noise ratio is given by: $S/N = N_s/\sqrt{N_s+N_b}$ . It is more convenient to deal with the square of the signal to noise ratio, called the “effective number of counts” $N_{eff} = N_s^2/(N_s+N_b) = N_s/(1+N_b/N_s)$. If you were to measure $N_{eff}$ signal photons with no background, you would have the same signal to noise ratio as you obtain with $N_s$ signal photons and $N_b$ background photons. Calculating $N_{eff}$ during the experiment is quite straightforward: $N_s$ is the difference in the number of photons above (A) and below (B) the edge, and $N_s+N_b$ is just the value above the edge; thus $N_{eff} = (A-B)^2/A$. When optimizing the experiment (for example when determining the best filter thickness to use), it is quite practical to (for example) add or remove a filter, move the monochromator above and below the edge, measure A and B, and calculate $(A-B)^2/A$, and use whichever combination of filters that gives the higher number. It is important to optimize the right expression. Although intuitively plausible, it is NOT correct to optimize the ratios A/B or (A-B)/B. Optimizing the wrong function will give degraded results. For example, choosing a filter by maximizing (A-B)/B in general results in a filter that is too thick.

Typically the amplitude of the EXAFS over the data range is only a few percent of the size of the edge step (A-B). If we want our EXAFS data to have a signal to noise ratio of 1–10%, then, relative to the edge step, we must obtain a signal to noise ratio of .01–.1%, i.e. $S/N \approx 1\text{–}3 \times 10^3$. Attaining such a signal to noise ratio requires $10^6$–$10^7$ effective counts total per energy point. This rough criterion allows us to estimate how long it will take to obtain the data during the experiment. If the time required exceeds the time available, it may be wise to reduce the energy range of the scan, because the data at high k probably would not be useful anyway. The time T available until the next fill of the electron beam is given by $T = \tau \ln (I / I_{min})$, where I is the present current, $\tau$ is the beam lifetime, and $I_{min}$ is the current at which the ring is refilled.

**Example:**

Suppose you are using a fluorescence ion chamber at the iron K edge, the amplifier is set to $10^{11}$ gain, and you are getting 260,000 V/F output pulses in 2 seconds integration time below the edge, and 520,000 above the edge. Many V/F converters in popular use put out 100,000 pulses/sec for one volt input, so below the edge we have 1.30 V, and above the edge we have 2.60 V coming from the amplifier. Since the signal and background are equal (1.30 V), the effective voltage $(A-B)^2/A$ is .65 V. Using the conversion factor[2] of $3 \times 10^6$ photon/sec for 1 volt at $10^{10}$ volt/amp gain at the Fe K$\alpha$ fluorescence energy, and accounting for the difference in gain, we obtain $N_{eff} \approx 3 \times 10^5 \times .65 \approx 200{,}000$ effective photons/sec. To obtain statistics of 2 million effective counts it would take 10 seconds integration time per energy point. If you are integrating for 2 seconds/point in a scan, and each scan takes 30 minutes, it would take roughly 5 scans and 2.5 hours to obtain the required data. If the electron beam lifetime is short, you may wish to include the effect of reduced beam current as well. If the beam lifetime is $\tau$ minutes, and the present beam current is I, the average current for the next T minutes is: $I (\tau/T) (1-\exp(-T/\tau))$. The average current until the next fill is: $(I-I_{min})/\ln(I/I_{min})$, where $I_{min}$ is the current at the next fill will occur.

In summary, it is well worth the trouble to memorize the equation:

$$N_{eff} = \frac{(N_{above} - N_{below})^2}{N_{above}}$$

as well as the mnemonic “HALO”: Harmonics, Alignment, Linearity, Offsets. In most cases one should obtain enough scans that $N_{eff} \approx 1\text{–}3 \times 10^6$ for the sum of all scans. In combination, these simple rules can be a great help in diagnosing problems, in planning experiments, and in making effective and efficient use of beam time.

---

[2] See “Estimating photon fluxes from ion chamber currents” in this series of notes: “Basic Techniques for EXAFS”.