Recommended Practices for X-ray Fluorescence with Amptek Systems

Once a user has his XRF system setup and is beginning to take data, we are often asked if we have any recommendations on how to set up or operate to obtain the best results. The following is a list of suggestions which we have developed over the years.

1. General advice for XRF analysis

1.1. Reproducibility is the key to accuracy.

XRF is a comparative technique: the measured intensity of the characteristic X-ray peaks are related to the concentrations of the elements in the sample by a set of parameters which depend on the details of the instrument, source-detector geometry, etc. Even the "Fundamental Parameters" analysis uses instrument dependent parameters.

- Calibration samples and unknowns MUST be measured under IDENTICAL conditions.
- Calibration samples and unknowns should have the nearly same composition.

  For the best results, calibrate using reference materials with composition nearly that of the unknowns. For second best results, calibrate using several single element standards. If you must, use standardless analysis, but it will only be approximate: it uses approximate physical models for the values which are obtained during calibration.

- Calibration samples MUST be measured long enough that the random errors in their measurement are much smaller than will be accepted for the unknowns.

1.2. To improve precision, increase the photopeak counts.

- To improve precision two-fold, increase the counts four-fold. To do this, you have three options:

  Increase measurement time. This is simple and direct but often not desirable.

  Increase count rate. This usually reduces energy resolution and/or impacts dead time and pulse pile-up corrections. It may require a change in source strength.

  Increase detector area. This usually reduces energy resolution.

- The importance of energy resolution depends on the measurement. As a general rule, if peaks overlap or are lost in the continuum, then improving energy resolution will help. If not, improving energy resolution is not useful while increasing count rate is likely very useful.

1.3. Errors and measurement uncertainty can be minimized but never eliminated.

1.4. Never report a value without reporting the measurement uncertainty.

"No measurement is meaningful unless accompanied by an estimate of its uncertainty". This is a general rule of scientific measurements, not unique to XRF, but is often neglected.

2. Optimize the set-up.

2.1. Carefully design the sensor head

- Ensure that the geometry of sample, detector, tube, collimation, etc. cannot change. If the distance or angle to the target change, the calibration parameters will be incorrect, degrading accuracy. See 1.1 above.
- Keep the distance from the tube to the sample and from the sample to the detector as small as possible, while preventing the tube from directly exciting the detector. This maximizes count rates and minimizes losses in air.
- Ensure that the materials used in the sensor head will not produce characteristic X-rays which interfere with your measurement, termed "contaminants".
- Filter the X-ray tube to minimize background in the energy range of primary interest.

2.2. Use good heat sinking on the detector

- In Amptek's XR100 system, the detector is mounted on a Peltier cooler, to reduce electronic noise. The Peltier cooler generates a temperature differential of up to 80 deg C between the detector and the back of the detector hybrid and its mounting stud. About 0.5 W must removed from the hybrid. If the back of the hybrid warms up, the noise will increase.
- In the XR100 and X123, the hybrid is mounted directly to the preamp. The preamp package is the heat sink so must be kept as cool as possible. Do not hold it in your hands. Instead, mount it to a metal plate. If you must mount it in an enclosure, provide a good thermal path to ambient.
- In some OEM applications, the preamp is a printed circuit board so has no heat sink. You must then provide a good heat sink, mounted directly to the back of the detector hybrid.

2.3. Eliminate or reduce external noise sources

- Use a single point ground for the system. Consider ground return paths, particularly those near the detector and preamp and those involving high power devices (often the HV supply for the X-ray tube or the supply in a PC or laptop).
- We have often found the charger in laptops to inject significant ground noise. Using a 3 to 2 prong adapter to float the laptop can be very helpful in these cases.
- Keep the preamplifier and DPP far from CRTs and switching power supplies.
- In OEM applications, if the preamp is a printed circuit board then it has no shield. You must provide an appropriate shield and must be very careful with the ground connection between the detector and electronics.

2.4. Optimize the DPP configuration parameters

- Peaking time: The best energy resolution is usually found at a relatively long peaking time. Shorter peaking times accommodate higher count rates but degrade resolution. The optimum depends on what you are trying to do: the more closely spaced elements, the more important resolution will be.
- Try to keep the dead time under 50%.
- Set the gain so that the highest energy you need to measure is just a little under full scale.
- Set the number of channels so the lowest energy peak you measure is ~10 channels FWHM.
- Set the detector temperature as low as possible, with 5°C margin at the highest ambient.
- When all other configuration parameters are set, then set the thresholds. Either use AutoTune or manually set the fast and slow thresholds to get 1-2 cts/sec with the tube off.

3. Optimize the analysis software parameters

There are two classes of parameters in Amptek's XRS-FP software package: some which describe the instrument (the geometry, the resolution, etc) and some which define the processing steps (the smoothing algorithm, the peak shape model, etc).

3.1. Make those parameters which describe the instrument as accurate as possible.

- Even small changes in these parameters may significantly impact the analysis result for certain photopeaks. It is sometimes tempting to put in a rough value but this may cause problems. These parameters have a "right" value and it is best to use it.
- The geometric details (distance to source, angle to target, window thickness, etc) are critical.
- Carefully set the information on the detector, the signal processor, and the radiation source (X-ray tube or radioisotope).

3.2. Emprically optimize those parameters which define the spectrum processing.

- There is no single "right" value for the number of iterations of background removal and other processing parameters. Changing these parameters may improve or degrade accuracy, or may add significantly to processing time. Using a nonlinear deconvolution, for example, allows the system to accommodate small changes in the gain or resolution of the spectrometer, but it adds considerably to processing time and the algorithm can converge to an erroneous value.
- Use a few samples of known values and empirically refine the parameters until you obtain an optimum combination. Play with the settings and see what they do.