

Standard Operating Procedures for XRF Detector

For a reference of the XOS instrument components, see Figures 1-4 in Appendix A.

1 Pre-Operation Setup

- Check Connections:
 1. Make sure X-ray tube is uncharged, i.e. machine is off. If on, contact user responsible for current experiment; if they cannot be reached, shut off the device via the computer software, and turn off the power supply.
 2. Check that all cables are properly connected for the computer, monitor, interlock box, and power supply for the XRF device.
- Inspect enclosure to ascertain damage or sources of possible leaks.
- Flip power supply switch on, turn on computer if it is not already done.

2 Saftey Procedures

- Obtain and put on personal protective equipment (PPE) as appropriate for the experiment being performed.
- Test interlock box to make sure safeties are functioning properly.

3 Login and Data Acquisition Setup

- Before using the software, insert specimen into enclosure below the source, adjusting the detector/source head so that it is lightly touching the sample surface.
- Close the enclosure doors, then log into the computer and open the Amptek ADMCA software.
- The ‘Starting ADMCA’ window will pop up asking for a device and whether the user would like to open a file or connect. Choose ”DP4/X123” as the device, and hit ‘Connect’. Verify that the USB symbol on the right corner of the status bar (bar at the bottom of the Amptek software window) is green to confirm that the DPP is connected.

- Enter ‘Acquisition Setup’ either by clicking the ‘Acquisition Setup’ icon shown in or by clicking the ‘MCA’ tab at the top and scrolling down to ‘Acquisition Setup’, both shown in Figure 5.
- The DP4/X123/PX4 Properties window will open as shown in Figure 6. Click the ‘MCA’ tab, shown in Figure 7, and perform the following:
 1. Set ‘Preset Time (sec)’ to ‘none’.
 2. Under ‘Slow Threshold’, set ‘channel’ to ‘0’.
 3. Click ‘Apply’, then ‘OK’.
- Click the ‘Start Acquisition’ icon; a large peak should appear in the lowest channels.
- Go to ‘Acquisition Setup’, click the ‘MCA’ tab again, click the ‘Tune Slow Threshold’ button, and then click ‘OK’. Click the ‘Stop Acquisition’ icon (originally ‘Start Acquisition’), then click the ‘Delete Data/Reset Time’ icon.
- Click ‘Start Acquisition’, go to ‘Acquisition Setup’, click the ‘Shaping’ tab (shown in Figure 8), click ‘Tune Fast Threshold’, click ‘OK’, then ‘Stop Acquisition’ and ‘Delete Data/Reset Time’. Go back to the Acquisition Setup window and click the ‘Shaping’ tab. Change the peaking time according to the specimen being used: for bulk specimens, use a low number in the 0.8 - 9.6us range; for thin specimens, use a larger peak time (typically >20us). Click ‘Apply.’
- Make sure the enclosure doors are closed and the indicator light on the bottom lock turns green, then access the XOS control software. A window will appear as shown in Figure 9. Set ‘voltage’ to 50, ‘current’ to 0.4, and click the ‘Start Ramp’ button.
- When the source has completed ramping, click the ‘Start Acquisition’ icon in the Amptek software window, then monitor the dead time on right. If dead time goes above 18%, adjust the peaking time to a lower number. When finished, click ‘Stop Acquisition’.

4 Calibration

- Click the ‘Analyze’ tab at the top of the Amptek software window as shown in Figure 10, and click ‘Calibrate’.
- Choose a well-known peak for your calibration material and select the center of the peak by left-clicking. Adjust the horizontal and vertical scales with the arrows on the bottom (refer to Figure 10) right in order to better locate the center of the peak. Input the energy value of that peak, then click ‘Add’. Repeat this step so that at least 2 points are chosen for calibration.

Note: As a second point, you can always use the Rayleigh-Compton scatter peaks, with the Rayleigh peak on the right. The Rayleigh peak has an energy equivalent to Mo Kalpha, 17.4 keV.

Note: This system will display Cu and Fe artifact peaks as part of the baseline signal since the tip contains Fe and the Cu comes from a shield that is misaligned with the detector.

5 Experimental Guidelines

- When taking x-ray fluorescence measurements, be sure to insert the specimen *before* the source is turned on. After the source is fully ramped, start the data acquisition for the desired preset time as per the experiment, then ramp down the source by clicking the "Turn Off" button in the XOS software window shown in Figure 9. Wait for the source to completely ramp down (voltage and current will lower to 0, the light above the enclosure will turn off, and the radiation symbol in the XOS control window will gray), then open the enclosure to remove the specimen.

6 Emergency Procedures

- For building or laboratory emergencies, click the "Turn Off" button in the XOS software window to shut off the source, then leave the lab.
- In the case that there is a software malfunction, manually switch off the power supply (refer to Figure 4).

7 Maintenance

- Radiation survey for enclosure inspection is performed every 8 weeks to ensure proper radiation containment.

A Appendix A



Figure 1: XRF detector enclosure

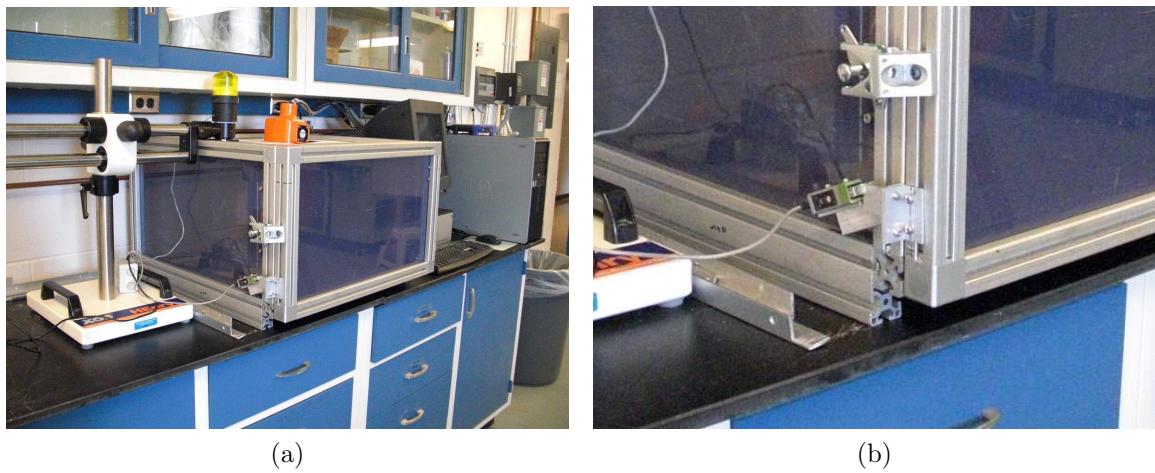


Figure 2: a) the interlock box as seen from the left of the detector enclosure, b) a magnified view



Figure 3: the power supply and MCA with a computer monitor on top and the computer for the MCA on the right



Figure 4: back of the MCA/power supply unit



Figure 5: the MCA tab, Data acquisition and DPP icons (from left to right): Acquisition Setup, Connect/Disconnect, Start/Stop Acquisition, Delete Data/Reset Time

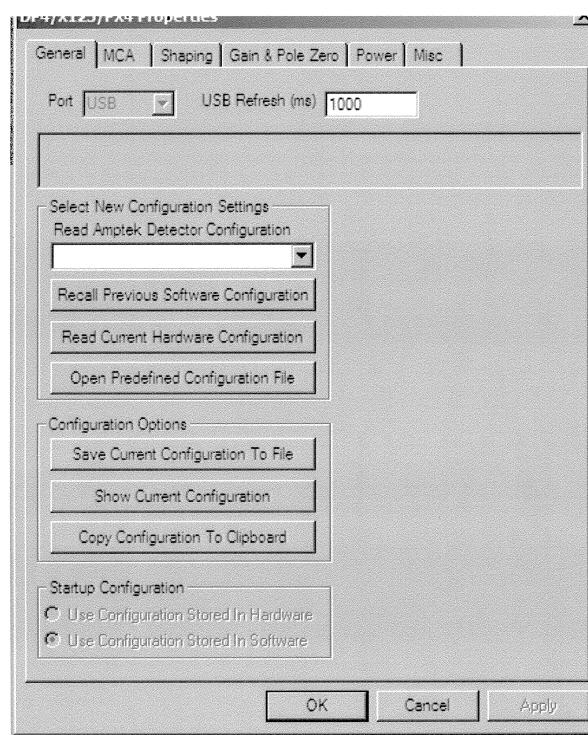


Figure 6: DP4/X123/PX4 Properties window

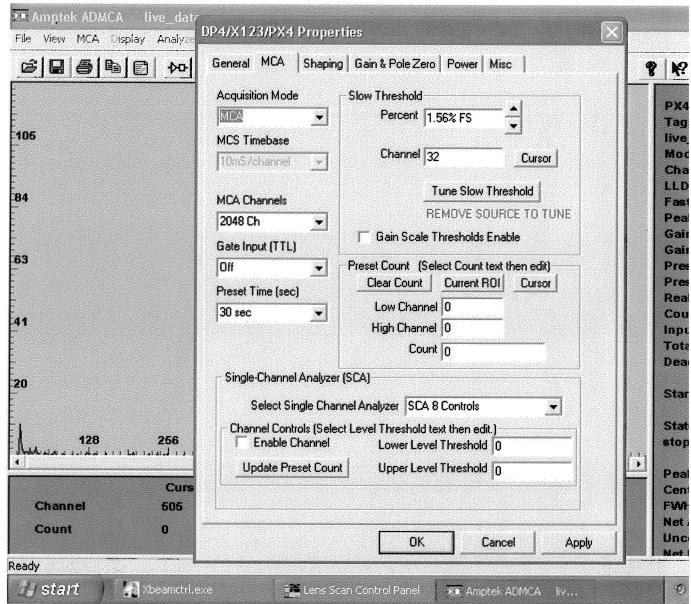


Figure 7: the MCA tab

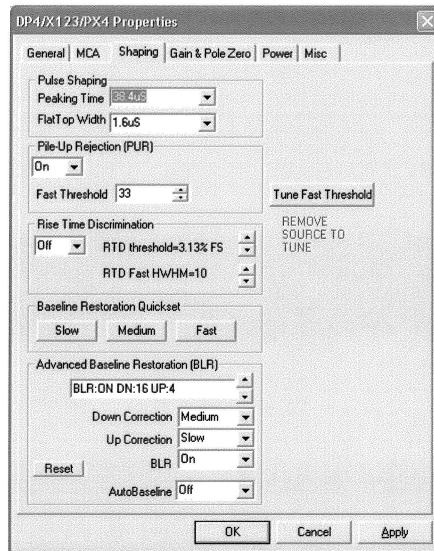


Figure 8: the Shaping tab

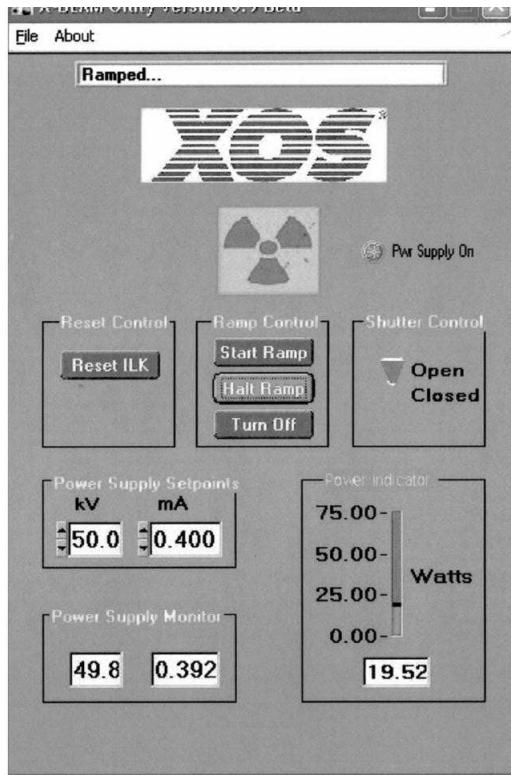


Figure 9: screenshot of XOS software control window

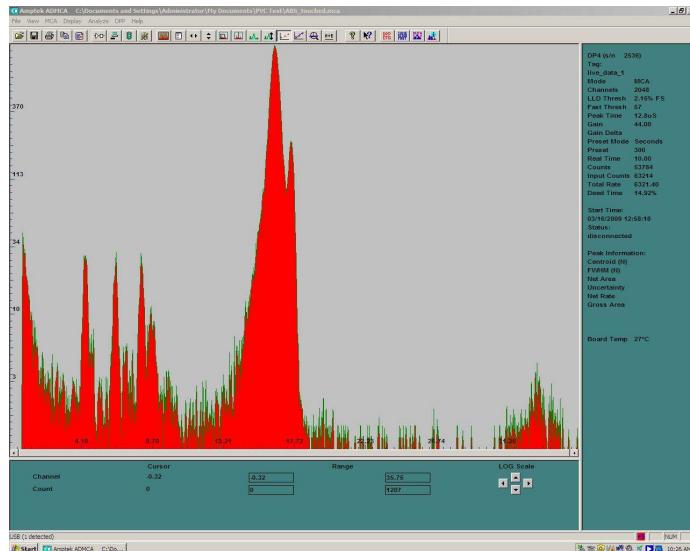


Figure 10: the Amptek software window