

X3B Operating Instructions

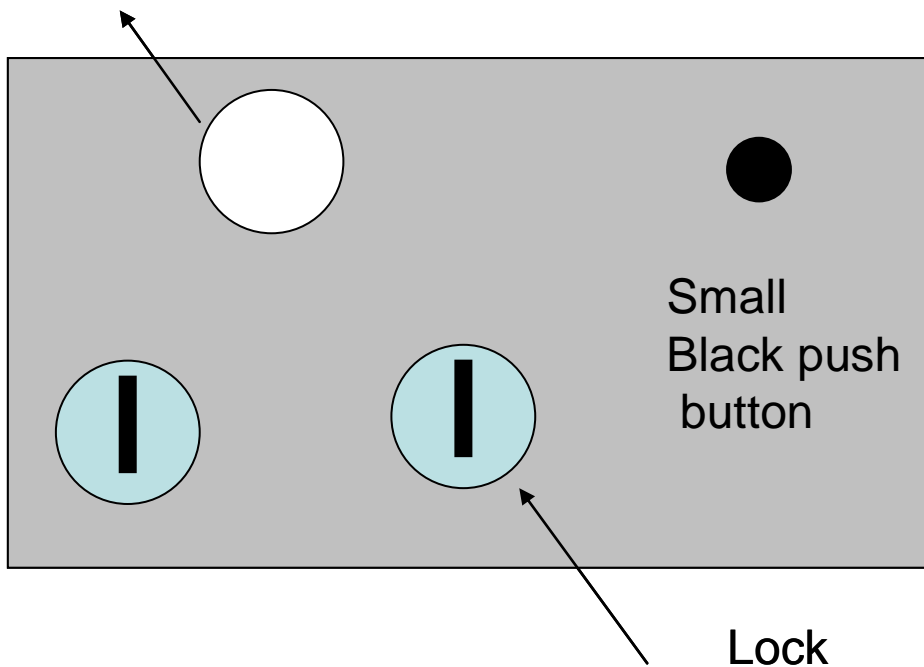
1. General safety procedures

Safety checklist. Walk around the back of the X3B hutch and walk in toward the synchrotron storage ring as far as possible. BS1 is all the way at the far end. Check to see that all of the bremsstrahlung shields BS1 through BS6 and all of the exclusion zone covers, EZ1B through EZ8B are in place. There is one padlock to check in the monochromator door labeled PL1B. The last exclusion zone is in the X3A hutch. Check when the beamline padlock was last inspected (see 'Padlock Log' on the door to the X3B hutch), and put the validation date on the form. Sign and date the form and give it to the 'op co' (operations coordinator). (The instructions for calling the operations coordinator are at the hutch.)

How to Open the Hutch

1. Close the photon shutter. It is located on the system reset box and is clearly indicated. When the shutter is closed, the green enable light goes out and the BEAM ON sign above the door also goes out.
2. Press the small black button while removing the interlock key. (See Figure below.)
3. Use the key to open the hutch door.

Large White light



NOTE: The locking procedure has been reversed as compared to X9b hutch. The lock is on the right slot instead of left slot.

How to Close the Hutch Before a Run

1. Check to MAKE SURE that no one is in the room. Press the interlock button on the left hand side wall of the hutch. (It is a large black button on a small gray box and is labeled *Personnel Check Station*.) A red light inside the hutch will come on.
2. Once this button has been pressed, you have 30 sec to close and lock the hutch door and push the external interlock button. (This is a large black button on a small gray box (by itself) to the left of the lock out key box.) If this button has been pressed in time, the INTERLOCKED light will come on.
3. Put the hutch door Kirk key into the lock (see Fig above), and turn it 180° clockwise.
4. When the alarm stops sounding, open the photon shutter, which is located on the system reset box.



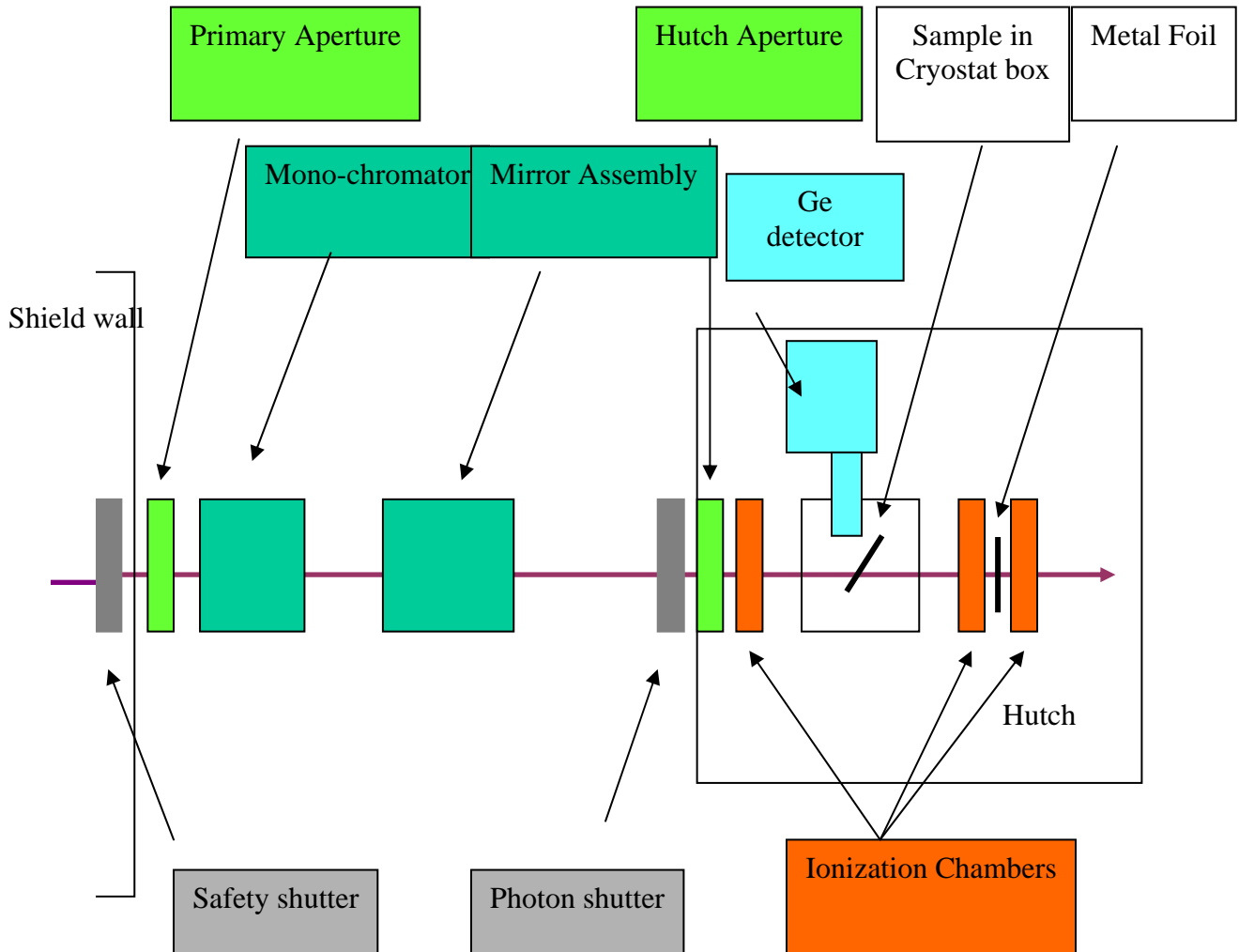
Note: In order to run, all of the lights on this reset box must be green.

What to Do If You Are Trapped Inside the Hutch

First, pound on the door. If this doesn't work, there is a large RED EMERGENCY STOP PUSH button near the door that can be pressed. They will shut down the entire ring so don't push either one unless it is absolutely necessary. This button is on the front wall to the right of the door as you face it to leave the hutch.

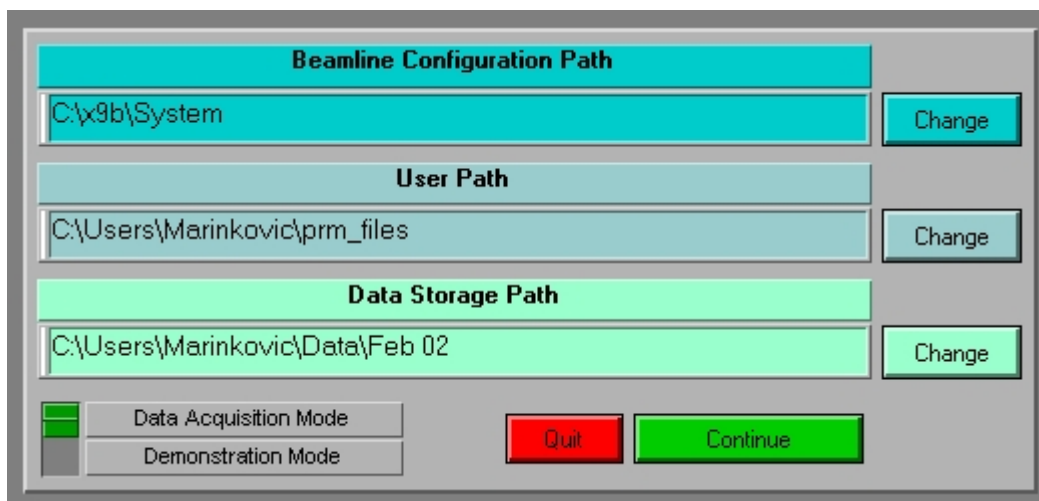
2. Beamline Schematic

Make sure that you understand beamline configuration and its optics. Schematic is given below. The synchrotron beam from the storage ring goes first through primary aperture (PA) slits, then through a monochromator containing Si(111) crystals, reflects off of a Ni mirror to cut off the second harmonic of the monochromator energy, and enters the hutch through a set of hutch aperture slits (HA). The beam goes further through primary ionization chamber (Io), through the sample in the cryostat chamber, and then through first ionization chamber (I1), metal foil, and second ionization chamber (I2). The cryostat chamber with sample is bolted to a motorized x-z table, which is in turn bolted to a pedestal. The height and tilt angle of the pedestal can be adjusted. The two ionization chambers (I1 and I2) are also bolted to the pedestal through a jack. Ge detector is resting on a separate height-adjustable table. For a successful run, each of these elements should be adjusted. Step-by-step instructions are given below.



3. Storage Path Setup

1. NSLS DAC program is usually running at the beamline computer, but you need to close it to be able to set your own storage paths. Find and write in your notebook the energy Mono Position (in the left top corner). Then press Quit. A window with "Quit DAC?" pops out. Click Yes.
2. Call the newest version of NSLS DAC program (at the desktop of the beamline computer). A new window appears.



3. **Do not change Beamline_Configuration_Path!** Click on Change next to User Path, and find your prm_files folder in C:\users\your_directory, then click on Use Current Directory. Then click on Change next to Data Storage Path, go to C:\users\your_directory and make a folder for the current month and year (for instance, August 2002 you can call Aug02). Click on Use Current Directory, then click Continue.
4. When the main window appears (**Fig. 1**), compare the Mono Position with the one you wrote down. If it is not the same, go to *Troubleshooting* section or call beamline staff for help.

4. Monochromator Focusing Procedure

The monochromator consists of Si crystal. The curvature of the second crystal can be adjusted to focus the beam horizontally. This is usually set by beamline staff to a relatively tight focus at 300 eV below E0 for the element used. The focus is then turned off, so that the beam expands slightly at higher energies. Therefore it is necessary to adjust hutch aperture horizontal slits so that a portion of the beam is cut off (see *Beam Fine-tuning*, page 8). When changing to a different energy range (i.e. for a different element) the focus setting should be moved together with the energy. The following procedure explains how that is done.



Note: The force constant of crystal has been calculated by measuring the best focus position at various energy in the range of 6000 eV to 10000 eV. On change of energies, the best focus position is automatically calculated and used.

1. (Do not change element yet). In Monochromator control part of the main window type in -300 eV in the red-outlined box and click Move. When the Monochromator stops moving, write down the energy under Mono Position.
2. In the main screen below click on Edges. Click on the element to be investigated and pick its energy transition. For instance, click on Mn and K α line. Click on Save and exit.

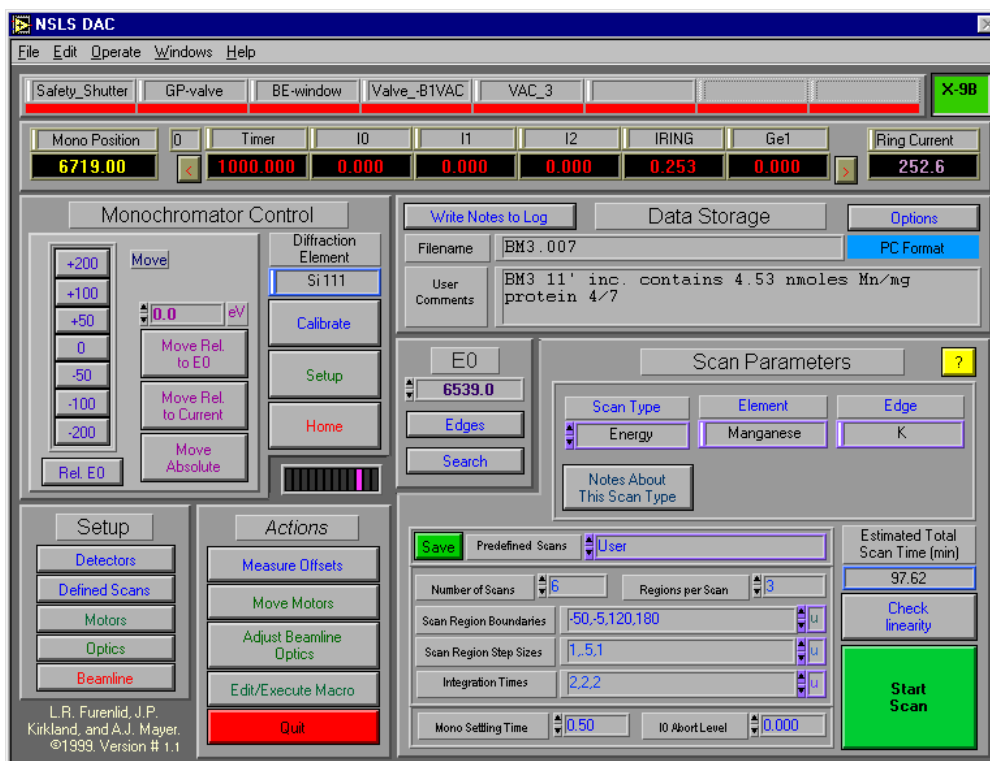


Fig.1.

3. In the main window, choose Setup. A new window pops out. In its lower right corner find the mono focus (in steps) and write it to your notebook.
4. Click on Focusing On, write down the number that appears in the box next to Focusing (that is the focus point in mm from the hutch windows), and click on Save and Exit.
5. In Monochromator control window (top left of the main window) type in -300 eV in the red-outlined box and click Move.
6. Click on Setup. The focusing should read the same number you had in step 1. If not, do the procedure A (below).
7. Click on Focusing Off, and then on Save and Exit.
8. Do piezo calibration (*see page 8*).

Procedure A

Move the monochromator to the energy you wrote in Storage Path Setup, step 1. That is, in Monochromator Control of the main window type in the energy in the red outlined box and click Move. Click on Adjust Beamline Optics. In the window that appears (see Fig. 2) click on the box under Optics Select and hold. Choose Mono Focus. In the box under Destination, type in the Mono Focus position (in steps) that you wrote down in step 3 and click Move Absolute. Then click Exit. Repeat steps 5-6. If the Focusing (in step 6) still differs from that you found in step 4 call beamline staff for help.

5. Print Optic and Motor Positions

This optic and motor positions are necessary as a point of reference, before you start adjusting optics (next sections). They can be discarded upon completing the setup procedure, i.e. after printing the new set (*see page 22.*)

1. In Main Window (Fig. 1) Click Adjust Beamline Optics. A new Window appears (Fig. 2).

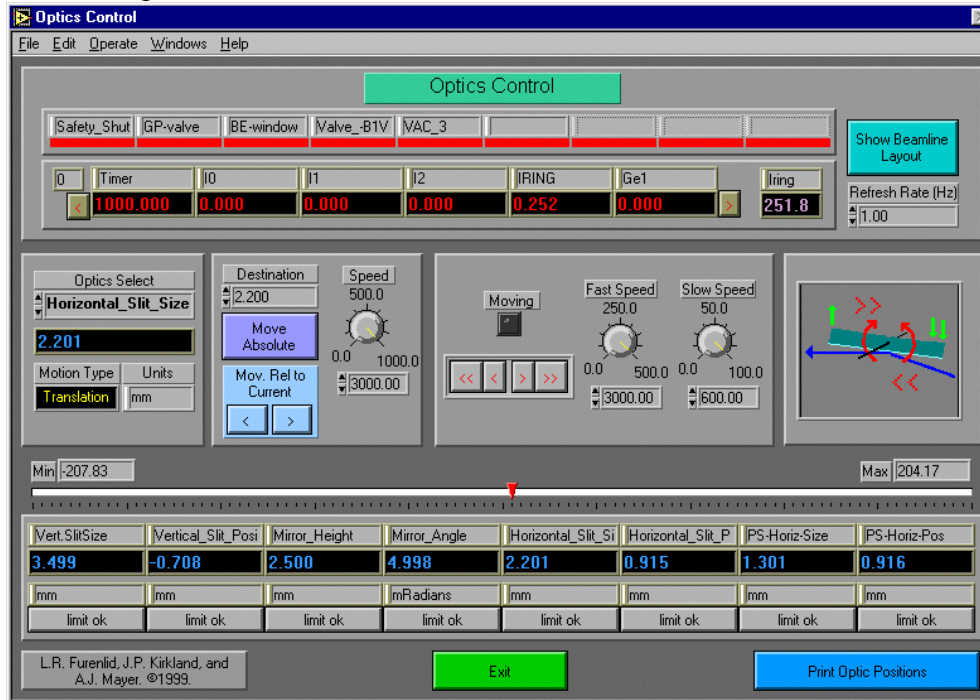


Fig. 2

2. Click on Print Optics Positions. Then click Exit.
3. In Main Window (Fig. 1) Click Move Motors. A new Window appears (Fig. 3).
4. Click Print Motor Positions, and then click Exit.

Motor Control

For ordinary run, everything that you need can be accessed from Adjust Beamline Optics. I unlikely event that you need to enter Motor Control, the window is given below.

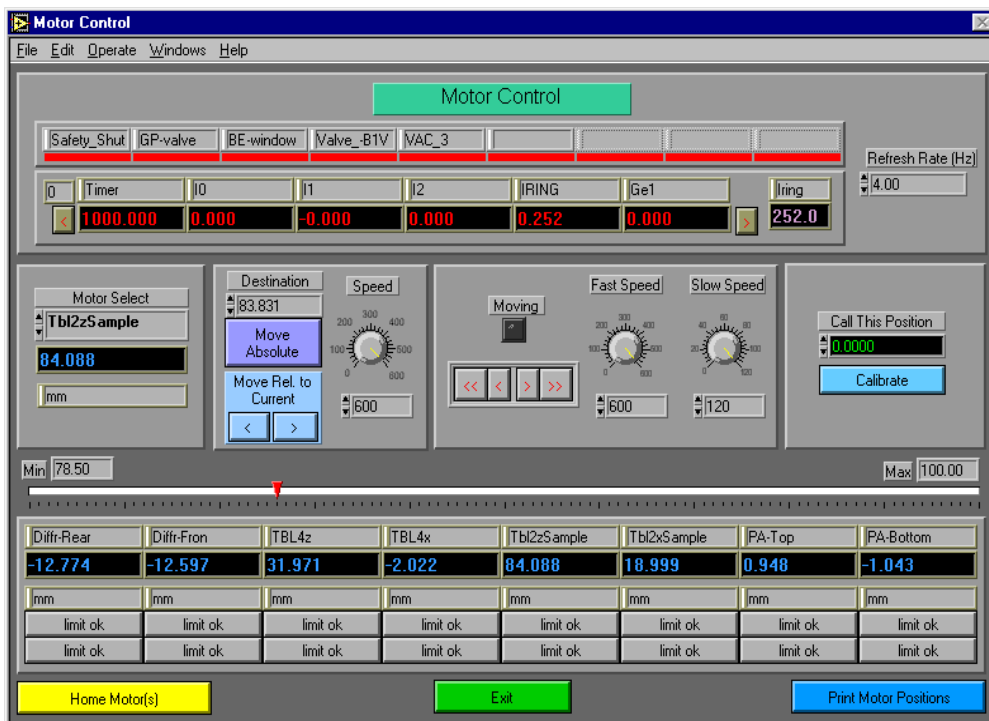


Fig. 3

6. Mirror Angle Adjustment

Ni mirror should be set to an angle that will give the highest reflectivity in the energy range you will be using, and minimal reflectivity in the range of the first harmonic, i.e. in the energy range that is twice the one you need.

1. Make sure that piezo calibration has been done upon changing the focus. If not sure, repeat piezo calibration (*see p.8*).
2. Place phosphorous screen in the sample holder. Phosphorous screen is a plastic 1' x 1" square, intensely yellow on one side. Place it in the sample holder so that the yellow side is facing you. Point the camera to the screen. After you turn the beam on, you should see a clear bright spot where the beam is hitting the screen.
3. Locate Ni reflectivity vs. Energy figures (elsewhere in this folder) for the energy range you will be using. For instance, if you are running Mn, you will need to choose the mirror angle that gives the highest reflectivity in the range of 6.5 to about 8 keV (for EXAFS) and zero reflectivity in the range of the first harmonic, i.e. 13-16 keV. Therefore, the mirror angle should be set to 4.5 mrad or higher.
4. In Optics Control Window (Fig. 2) click on the box under Optics Select and hold. Highlight Mirror Angle. In the box under Destination, type in the desired mirror angle and click Move Absolute.



Note: Remember that the beam moves vertically by about 3.5 mm at the sample for one mrad mirror angle change. For instance, if you increased the mirror angle from 3.5 to 4.5 mrad, the beam is going to hit the sample at the spot that is about 3.5 mm lower than before. You will have to lower the sample table and move the vertical hutch apertures accordingly. **Calculate** how much – you will need it later.

5. Check the reading at Io on the first meter reading (refer to *Meter Reading* section). If it shows a number close to zero, and/or you no longer see the bright spot on the phosphorous screen, the beam no longer enters the hutch. To get it back, go to Optics Control Window (Fig. 2), click on the box under Optics Select and hold. Highlight HA_Vertical_Slit Size. In the box under Destination, type in a large number (for instance 6mm) and click Move Absolute. The reading on Io should increase and the beam should be (at least barely) visible.
6. In Optics Control Window (Fig. 2), click on the box under Optics Select and hold. Highlight Hutch Aperture Box-Vertical. In the box under Destination, type the number you calculated in step 3 (Note), and click on the arrow under Move Relative to Current. For positive values, press the right arrow (>), and for negative press the left arrow (<). Refer to *Motor Movement* section for further details. You should have a clear spot on the phosphorous screen and a high value on Io meter. Its height will be adjusted in section 7.
7. Fine-tune the HA Aperture Box-Vertical by slitting down the Vertical Slit Size to about 1 mm and moving the HA box up or down until you maximize the Io. Refer to steps 5 and 6 of this section for adjusting the slit size and moving HA box, respectively.

7. Pedestal adjustment

The height and the tilt of the pedestal that supports the two ionization chambers and the cryostat chamber with the sample should be set in order to align the beam downstream from the hutch apertures.

1. Locate four screws at the pedestal. They lock the pedestal at a certain height. Loose them.
2. Change the height of the pedestal by the amount calculated in Mirror Angle Adjustment section by rotating the crank at the foot of the pedestal. Rotate it clockwise to increase height.
3. Retighten the four screws.

The tilt of the pedestal table now should be adjusted to the angle of the Ni mirror. Rotating two thumbscrews underneath the pedestal table sets it.

4. Find the small protractor with micrometer and level, usually at the top shelf of the cabinet in the hutch. From the table at the protractor read the correct micrometer height for the mirror angle, and adjust the micrometer. Place the protractor at the table. Watch the level. If the bulb in the vial is outside of the two center markings, adjust the two thumbscrews under the pedestal table to move the bulb into the center.

The beam should be coarsely aligned now to pass through the sample and ionization chambers.

5. To check, turn the beam on and watch the position of the beam at the phosphorous screen. The beam should be vertically aligned.
6. Take out the phosphorous screen and place a small piece of Kodak photo paper between the two ionization chambers. Notice the reading of I1 and I2. Turn the beam on and watch the readings of I1 and I2. They should increase. After a minute, turn the beam off and take out the piece of Kodak photo paper. A clear black spot should be visible at the paper, indicating the position of the beam.

- If the beam is not visible at the photo paper, or if it is well outside the center of the ionization chamber's window, you may optimize the height of the I1 and I2 by adjusting the height of the jack that holds them.

8. Beam Fine-tuning

- Make sure that the energy of the monochromator is set to 300 eV below E0. Open the primary apertures horizontal size fully (for instance 20 mm).
- Watch I₀. Reduce the PA horizontal size by 1 mm.
- Repeat step 2 until you notice that the I₀ shows a decrease of a few percent.
- Open the horizontal slit size to about 15 mm.
- Watch I₀. Reduce the PA horizontal size by 0.5 mm.
- Repeat step 5 until the I₀ is reduced by about 20%.
- Close PA vertical slit size to 0.4 mm
- Move PA box-vertical by 0.05 mm up or down to maximize the reading on I₀.
- Open PA vertical size to desired size.

Note: Reduction in PA vertical size increases energy resolution, but reduces the flux of the beam. You may need to compromise.

- Repeat steps 7 and 8 with HA vertical size and HA box – vertical. HA size has no effect on resolution.

9. Dark Current (Offset) Measurement

- Close the Photon Shutter to turn the beam off. In the main window (Fig. 1) click on Measure Offsets. A new window appears (Fig. 4).

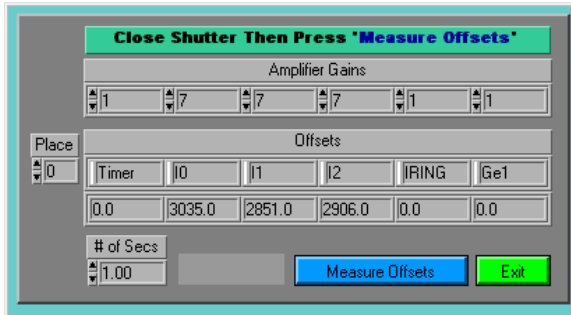


Fig. 4.

- Press Measure Offsets. “Reading..” will lit. After it disappears, click Exit.

10. Piezo Calibration

A full Piezo calibration should be done after each beam dump and every 6 hours thereafter. In addition, the control screen should be set to update the calibration after every few scans, so that it is updated approximately every 2 hours. (Note: Automatic updates sometimes causes problems, see Troubleshooting for details).

Note: If you do not do a full calibration at the start of a new fill, the beam can be so out of alignment that you will get no beam at I₀.

1. On the main window (Fig. 1) Click the Calibrate button in the Monochromator Control section. A new window (Fig. 5) will appear.

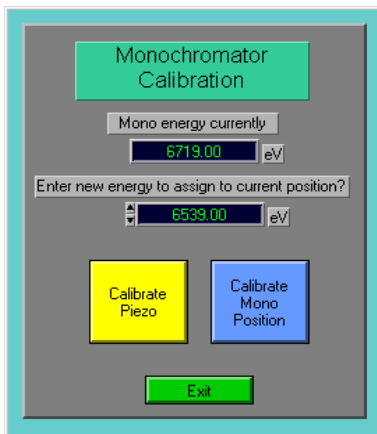


Fig. 5.

1. Click the Calibrate Piezo button. **Do not click Calibrate Mono button!** A new window appears (Fig. 6).
2. Make sure that E0 (eV) is set to the correct energy for the element. Make sure that Automatic is chosen in Piezo Control and Update Piezo calibration every --- scans is set to the desired level (see note at the beginning of this section). 3-5 scans seems a good initial number.
3. Fill in Calibration Points (relative to E0) either manually, or use Predefined points for EXAFS (-300 to 1050 eV) and XANES (-200 to +200 eV). Click on desired button. Full calibration and Update calibration buttons will lit.
4. Click Full calibration button. Observe I₀ while the calibration is taking place. The calibration is supposed to maximize I₀ at each of the energies used. Generally, the shape of the Piezo curve vs. Energy looks similar to that in Fig. 6, except at the end of the useful range of Si(111) crystal (above 11 keV).
5. When the calibration is complete, click the Print button followed by Save and Exit button.



Note: Piezo can vary from 0 to 600V, ideally it should be in the center (around 300 V)

On LOW VOLTAGE PIEZO TRANSLATOR CONTROL:

→ Reverse and Forward direction represents the movement of piezo from center (300 V) towards 600 V and from 300 V towards 0 V, respectively

→ Down and up speed represent movement of piezo by 6 and 12 V increments

Example: In the middle of scan if piezo voltage goes wrong. The piezo is off with a value say 100 V the way to optimize it is as follows:

1. Maximize I₀ by where it is. If there is very low or negligible I₀, change gain
2. Keeping track of I₀ move the speed in 6V increments (remember that you are moving from 100 to 300 so the direction should be reverse (from 300 to 600 V) if piezo reading was 500 V the direction should have been forward)
3. Maximize I₀ by turning voltage control on Piezo control card
4. Repeat procedure until the voltage is around 300 V with a maximum I₀

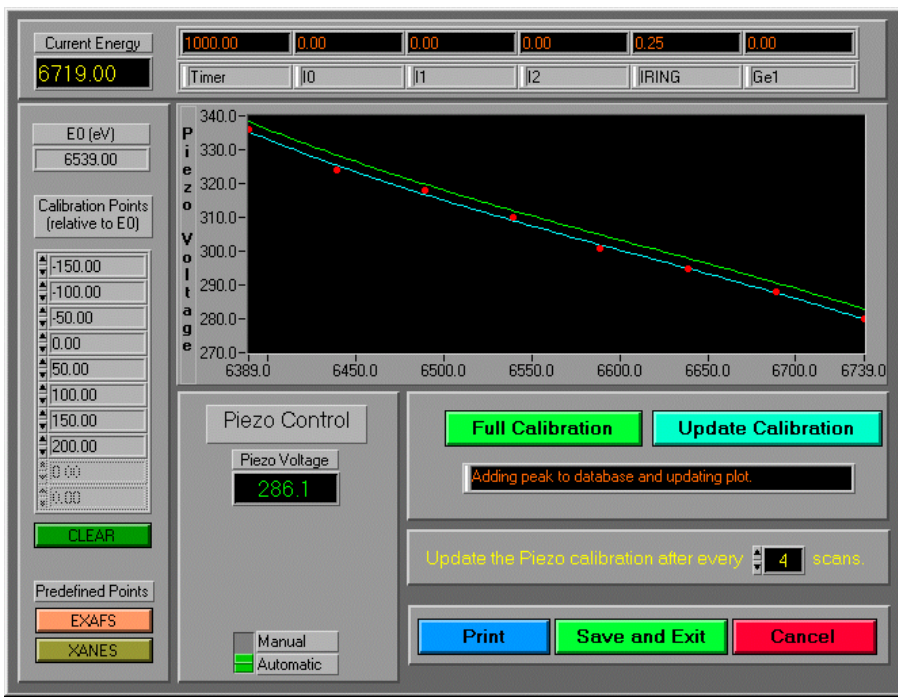


Fig. 6

11. Monochromator calibration

1. Make sure the appropriate metal foil is in position. **The metal foil must be no thicker than 10 μm .** Thicker foils will result in too big a decrease in I2, which will require such large amplifier gains that noise will be too large. A metal powder standard can be used instead of the metal foil, as long as its thickness is uniform throughout the standard and the particle size are ca. 6 μm or less. Take out the sample from the cryostat chamber. Make the HA slits at least 1.5 in the z direction, and 2.5 in the x direction.
2. Turn beam on and on the main screen make sure that the scan type is Energy & element is of the metal foil. Click on User and choose mid_edge scan, choose 1 scan and click Start Scan. A new window appears (Fig. 7).
3. In the top left corner, under Detectors, check the boxes next to I1 and I2. Watch the blue box above. Make sure it indicates I(1)/I(2). Then press Y'(X) button. The blue box should read d/dx[I(1)/I(2)]. After the scan is done (Green Continue button flashes), move the cursor to the peak of the first maximum, which is E0, press the Move to Cursor Position button and then press the Calibrate Position button. A new screen will come up (Fig. 5). Press the Calibrate Mono Position button. You will be asked 'Are you sure?'. Press Yes and then press Exit.
4. Repeat step 2-3, except run a near_edge scan. This is to further refine the proper location of E0.

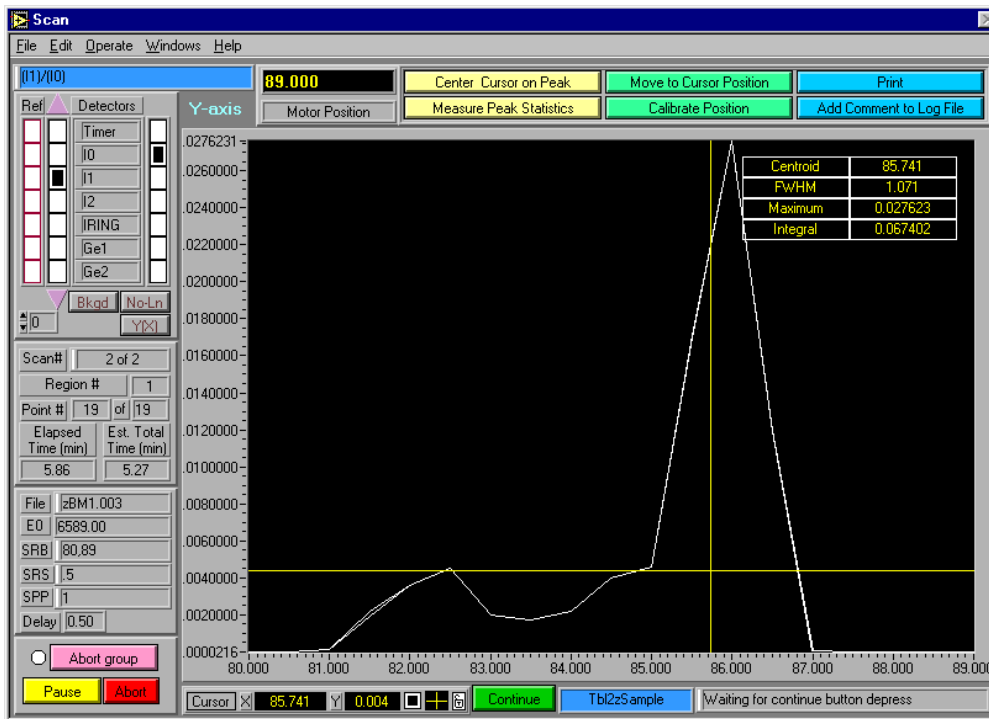


Fig. 7.

12. General Information

To understand future sections, first read the following info on location of meters, controllers etc.

Motor Movement

Never move any motors (except the slit width motors) before pulling the detector away from the sample holder!

In order to move the motors, click on the **Move Motors** button in the main menu. Each time a new sample is placed in the holder, both x and z alignment should be checked and the beam centered in the middle of the sample. Very often, it is unnecessary to calibrate in the z direction more than once for two reasons: (1) the beam height is always less than 2 mm, irrespectively of HA slits width; (2) the height of the standard sample holder is 5 mm and so even if the beam is not perfectly centered in a particular sample, it is unlikely to hit the edges of the holder unless the sample was set in nowhere near the top of the holder. However, a given sample might have bubbles in it in which case it would make sense to check the z alignment and perhaps move the center of the beam off the center of the sample in order for the beam to go through the thickest part of the sample.

x-z sample alignment

To locate the proper x and z position for the beam, following the instructions [for x and z alignment](#) on page 20, but expand the region to include the movement needed to center the beam on the sample holder's hole. The preliminary scan can use a relatively large step size to speed up the process.

Location of the beam using photo paper

If by some chance, no beam goes through the sample during the x or z alignment, the beam must be located. In this case, cut a little bit of paper from the roll of Kodak Linagraph direct print paper. (This paper is inside a small cardboard box on top of the computer). The active side of the paper is yellow, so the yellow side of the paper should face the beam. Place one piece of paper on the outside of the sample chamber where the beam enters the chamber. This is called the nose cone. Tape another piece of paper on the back side of a sample holder facing the beam. (Don't cover the burn region with tape because the tape will prevent the paper from darkening.) Open the horizontal and vertical slits to about 5 mm so plenty of beam energy is available for burning the paper. Expose the paper to the beam for about one minute. Check the paper on the nose first and see if the burn area is centered on the nose. Next, remove the sample holder from the sample chamber and look for the burn mark in the back of the sample holder. If no burn mark is visible on either paper, shine a flashlight on the paper. If a burn mark exists, the flashlight will cause it to darken enough to become visible. If no burn mark appears on either paper, you have real problems and should call for help. If the mark on the nose is off-center and no mark appears on the sample holder, you should probably still get help. However, if marks appear on both, hold the sample holder up to the light to determine where the burn mark is relative to the hole in the sample holder. This can give a relative idea of how much x or z distance must be scanned in order to locate the hole in the sample holder. When deciding in which direction to scan the beam, remember that positive x increases toward the controlling computer station, i.e. on the right when standing behind the sample table and facing the beam input, and decreases away from the controlling computer station. Z increases in the up direction and decreases in the down direction.

Moving the Detector

Once the sample holder is in the proper location for the first run, it may be found that the barrel part of the detector no longer fits into the window in the side of the sample chamber. If this is the case, it will be necessary to adjust the x, y and z location of the detector so the detector can fit into the window in the side of the sample chamber.

The detector moves freely in the x direction but its movement is limited by a pair of set screws. The set screw in the - x direction basically keeps the detector from coming off of the rails. The set screw in the +x direction is very important. It stops the detector from going too far into the sample chamber and breaking the window. There is black tape around the barrel of the detector. The +x set screw should be adjusted so the detector goes into the window just up to the black tape but no further.

The screw for the y axis moves the detector in the y direction and should be adjusted so that the detector barrel is centered in the window in the sample chamber.

If the detector will still not fit into the window in the sample chamber, it will be necessary to adjust the height of the detector so that the barrel of the detector can be centered inside the window in the sample chamber. To change the height of the detector, change the height of the table. This table is basically a large hydraulic jack and works just like such a jack does. That is, it has a knob to lock the jack in position, a handle for

pumping the table up and a release screw to lower the table by letting hydraulic fluid out of the jack. Look under the table the detector is on. There is a black knob about halfway up the center support for the table. Turn this knob counterclockwise to remove the lock from the jack. If the table needs to go up, gently pump the lever under the table until the detector is at the desired height. If the table needs to come down, gently turn the release screw counterclockwise until the table level is correct. Once the z position is such that the detector can slip into the window in the sample chamber, turn the black knob under the table clockwise to lock the vertical position of the table.

Turning on the Helium Cryostat

The helium cryostat is controlled by the large silver box at the back shelf. The side of the box to the back of the sample table has a water flow meter. Water must be flowing through this valve so that the meter registers 50% flow (.5 on the flow meter). If not, adjust the flow until flow is 50%.

Turn power on. The power button is the only switch at the front.

Once the power is on, follow the instructions on the side of the beige box that is located on the shelf facing the beam. This box displays the current temperature in the sample chamber and is used to set the sample chamber temperature. The lower limit of the cryostat is 11°K, even though the cryostat control box can be set to lower value.

Location of Meters

EXAFS/XANES data collection are done using the beamline control computer and the germanium detector computer. Above the control computer are a series of meters (see Fig. 8).

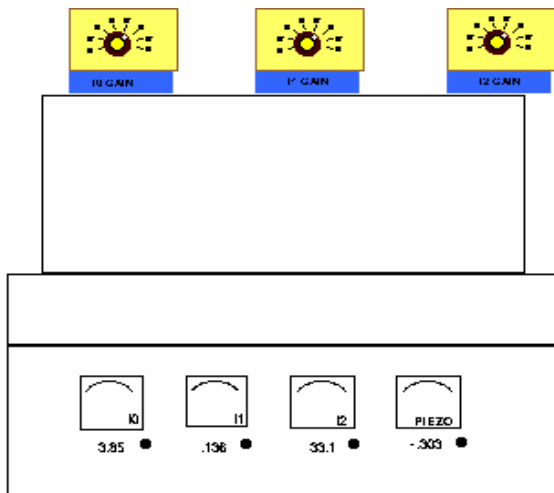


Fig. 8.

The meters labeled I0, I1, I2 show the currents I₀, I₁, I₂ in mA. I₀ is the beam current before the sample, I₁ is the beam current after the sample and before the metal foil, and I₂ is the beam current after the foil. The rightmost meter labeled **Piezo** shows the piezo

voltage applied to the piezo crystal used to maximize the beam. The small black knobs to the right of each meter are used to change the reading scale of the meter and will not affect the actual value of current, i.e. they are not amplifiers. On the other hand, the tan boxes at the top of Figure 8 actually change the gain of I0, I1 and I2 respectively. Generally $I_0 = 10^7$, $I_1 = 10^7$ and $I_2 = 10^8$. Offset for all currents = .030 milliamps. These gains should be changed only if the current as detected by the meter is either too large or too small. This normally occurs only when there is no sample. When the currents are too large, the program say so. Once a sample is in place, the currents will drop and the gain will have to be increased again.

NOTE: It is very important that I0, I1 and I2 are all sufficiently large that signal to noise is large. In practice this means that none of them should start a run with values less than 10,000 and I0 should start at least at 100,000. Once the photon shutter is open, the current values of I0, I1 and I2 will register at the top of the menu of Fig. 1. If there is any difference between what the top of the menu says and what the meters read as shown in figure 1, use the menu readings since they will be used to calculate the edge. The necessary conversion factor is 200,000 times mAmps = counts. Therefore, I0 should be ~ 0.5 and I1 & I2 should be ~ .05. In general, I0 will read between 2 and 4 if the beam is relatively intense and the slits encompass the entire beam. Once slit size and the necessary calibrations have been performed it should not be necessary to alter the gain of I0. If I0 drops suddenly during an experiment, check the **troubleshooting** section. Although I1 & I2 should be ~ .05 it is also best to prevent them from becoming much greater than 0.8 because better signal to noise is achieved when the gains applied to these currents are as small as possible. Alter the gains to increase or decrease the values of I0, I1 or I2 as necessary.

Ge detector meters

Ge detector meters are read from a silver-gray box located above the UNIX computers at your right (See Fig. 9).

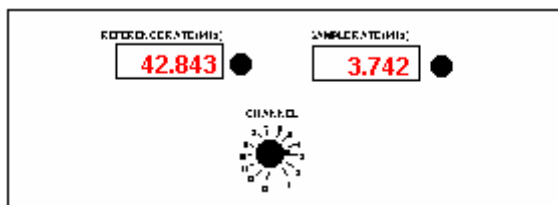


Figure 9

The channel knob chooses the detector whose rates are shown on the two meters. The reference rate meter on the left, labeled Integral Count Rate (ICR) shows the total counts that reaches a particular detector and includes all of the scattered photons and all of the fluorescence photons. This rate should never exceed 200- 250 thousand counts for any detector. In order to keep the response linear, the ICR counts should never exceed 100,000 for any detector. Fluorescence photons are registered at the other meter (Single

Channel Analyzer, SCA). If your sample signals are small, detector number 1 is a good channel to observe because it has a large scattering signal. On the other hand if your sample is very intense such that scattering is generally small with respect to sample fluorescence, then choose the middle detector 2 or detector 7 because they see the largest sample fluorescence. See Fig. 10 for detector positions in 13-element detector array.

Detector Array

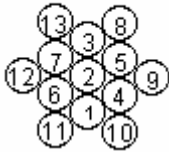


Fig. 10.

Check the range of the detectors by using your most concentrated sample or standard and checking the reference rate for the minimum value of E to be used in any of your scans and observing the rates for the various detectors, particularly detectors 1 and 10. Next observe the rates at $E_0 + 100$ for detectors 2 and 7. If any detector exceeds 200 to 250 kHz for a given sample (or $\sim 80,000$ kHz if linearity is desired), one or both slits (x and z) will have to be decreased to prevent a distortion of the resulting fluorescence energy spectrum.

Note: Do not rotate channel knob during data collection! Leave it on channel 4 or 13.

The fluorescence signals from detectors 8, 9 and 10 are generally small with respect to the other detectors.

How to Change Samples

1. Close photon shutter, open the hutch door and **BACK the detector away** from the sample chamber.
2. Turn off the vacuum. (That is, turn the blue handle on the right hand wall. The vacuum line from the sample chamber clearly goes to this blue handle.) The vacuum can be turned off quickly.
3. As soon as the vacuum is turned off, open the helium flow. This is controlled by a green handle almost directly below the blue vacuum handle. This handle can be opened and closed rapidly but when opening the helium flow, **DO NOT HOLD THE SAMPLE WINDOW** in place but keep your hand nearby in case the helium flow blows the window off.
4. Remove the window and lay it on the table.
5. Remove the old sample and put in a new sample. The sample may be held in place by the middle set screw only. Place a gloved hand under the sample to catch it and loosen the middle set screw. Place the old sample in its storage compartment. Hold the new sample in a gloved hand and place the sample in the slot such that the edge of the sample nearest you is flush with the sample holder and tighten the middle set screw. If desired, tighten the two other screws.
6. Place the window on the two locating pins but don't push it in place.
7. Close the helium valve.

8. Hold the window in place with one hand and **very slowly** open the vacuum valve. Once the window sucks in, the vacuum valve can be opened all the way.
9. Close the hutch and open the shutter.

13 A. Germanium Detector Setup Procedure

Use a sample whose concentration is about 1 mM for the element investigated, and expose it to the beam with the energy set to about 50 eV above the edge. This sample concentration produces relatively intense fluorescence and scatter peaks. The energy must be above the edge to initiate fluorescence and should be relatively close to the edge since this will show the maximum effects of scattering.

1. Turn on the monitor for the Germanium Detector Computer. (This computer must never be turned off!)
2. Once the monitor is on, Figure 11 shown above will appear on the screen.

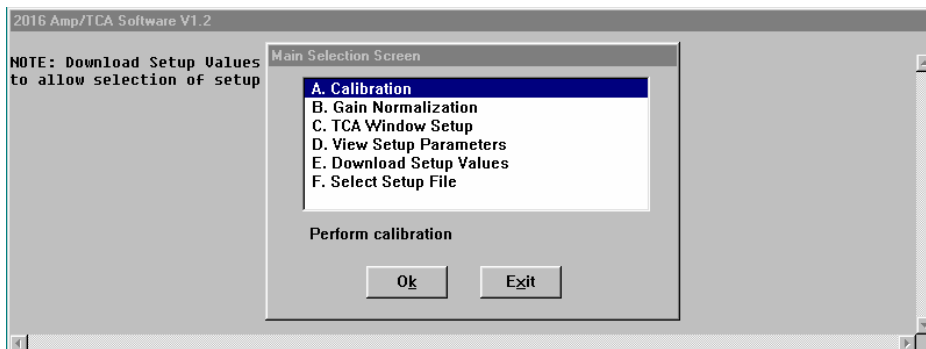


Fig. 11

3. Click C. TCA Window Setup, and then click **Ok**.
4. The screen will be divided in two parts: TCA Window Setup shown in Figure 12 will appear at the top and Gamma - DET01 (Fig. 14) on the bottom.



Figure 12

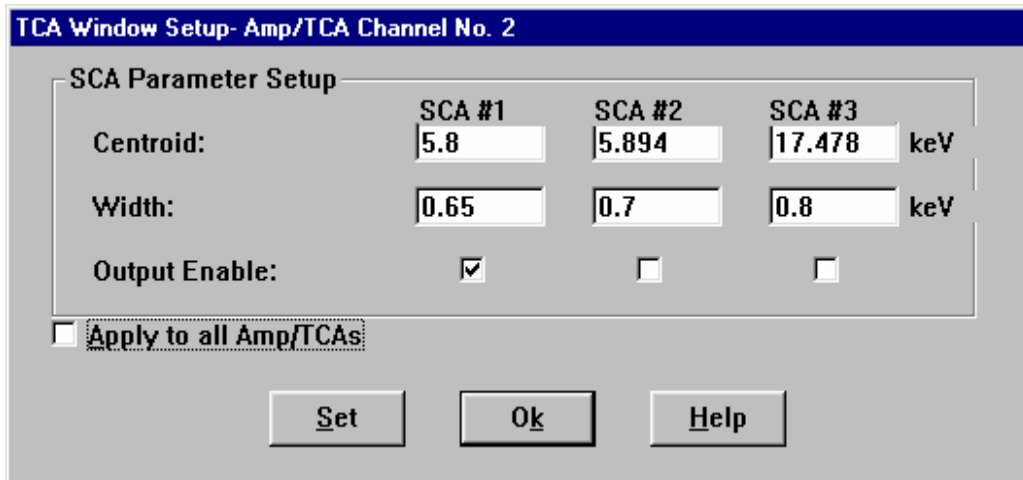


Fig. 13

5. Select the first channel whose sample signal is to be maximized (Channel 1 is a good channel to choose because it has significant scattering.) and then click **Ok**.
6. The new screen (Figure 13) will appear. Only SCA #1 should be enabled by Output Enable check-box.
7. Set the Centroid to the center of the fluorescent peak. Use green cardboard ruler (in the drawer under the beamline computer) to find the fluorescence energy. For instance, for Mn $K\alpha$ line set centroid to 5.9 keV. Set the width initially to about 2 keV. That is type in the changes in centroid and width and then clicks the Set button.
8. Data will be accumulating in the Gamma - DET01 window and should show 2 peaks. The lower-energy peak is the sample fluorescence peak. To be assured that it is the sample peak that is being observed, move the monochromator to E0 - 50 eV and then click on the Clear button. The sample peak will disappear, if it doesn't, you are probably looking at the scattering peak. If a blue region encompasses the fluorescent peak as shown above, do not change it. If it does not, click on Display, and choose ROIs and then click on Clear All. The current blue region or regions will disappear. Move the white brackets to enclose the fluorescent peak and then click on Display, choose ROIs and then click on Add ROI. The region between the white brackets will turn blue.

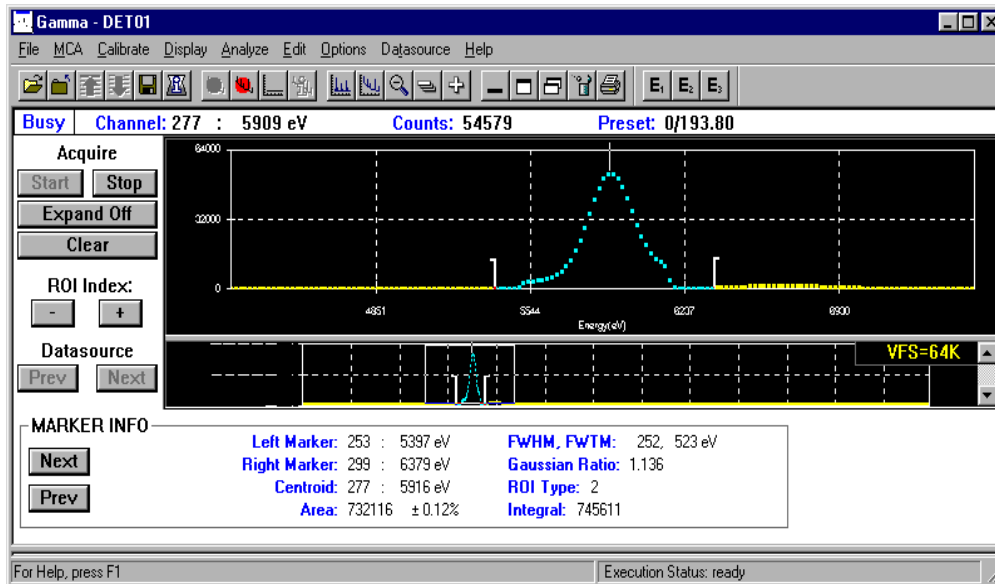


Fig. 14

9. If Gamma - DET01 window looks exactly as shown in Fig. 14, including a region of expansion that includes the fluorescent peak, this region doesn't need to be changed. If no such region exists, press the Expand On button and the expansion region will appear. A white box will appear in the unexpanded portion of the spectra. Use the mouse to drag the box to include the portion of the spectra that is to be expanded. Dragging the edges of the box changes the size of the box and dragging the center of the box changes the location of the box.
 10. Once the fluorescent peak is expanded, go back to the upper window and change both the width and centroid of the window in order to maximize the sample fluorescence while minimizing the scattering. A good starting value for the width is 0.6 to 0.7. Be sure to press the Set button to activate the changes. For weak samples it is best to set the centroid to a value that is slightly lower than the peak value. For example, for Mn setting the centroid between 5.78 and 5.80 reduces the overall scattering relative to sample fluorescence intensity. In general, if the fluorescent peak shows an up turn on the high-energy side, either the width or centroid should be decreased.
- Note:** If the marker info doesn't show, drag the window down to make it larger. The Gaussian Ratio should be close to 1.
11. Once satisfied with the settings for channel 1, check the Apply to all Amp/TCAs box and click Ok. This will make the centroid and width the same for all of the 13 detectors.
 12. After a few minutes (be patient!) the TCA Window Setup will reappear. Set channel to 2 and click Ok.
 13. The TCA Window Setup - Amp/TCA Channel No. 2 will reappear. Uncheck the Apply to all Amp/TCAs check box.
 14. Observe the sample fluorescence and make any changes necessary to maximize the sample fluorescence while minimizing scattering. Once satisfied with the results,

click Ok.

15. Repeat steps 12 - 14 until windows have been set for all 13 channels. After the last channel has been set, return to the TCA Window Setup and click PREVIOUS. The first window (Figure 11) will reappear.
16. Turn off the power to the monitor.

The rate shown by the sample rate meter (SCA) is only the counts inside the limits determined above. These gates are then used to determine which data is sent to the single channel analyzers that collect fluorescence counts from each detector, displays these counts on the screen and saves them to the file.

13 B. How to switch ON and OFF Ge-detector

To switch detector ON

1. Turn ON the ON/OFF button on the back of CANBERRA detector electronic rack
2. Turn ON two ON/OFF button on the front of electron rack
3. There will be an alarm sound, Push LN2 push button on the front of electronic rack [LN2 Monitor]
4. Start the Program
5. Go to MCA and click Adjust
6. Select HVPS
7. Slowly increase the High voltage to 500 V by continuously watching the indicator lights on the front of electronic crate
8. Close the window and start the procedure as mentioned above

To switch detector OFF

1. Go to MCA and click Adjust
2. Select HVPS
3. Slowly decrease the High voltage to 500 V by continuously watching the indicator lights on the front of electronic crate
4. Turn ON two ON/OFF button on the front of electron rack
5. Turn ON the ON/OFF button on the back of CANBERRA detector electronic rack.

14. Starting a EXAFS/XANES Run

Use the NSLS XDACX09Vxx program for acquiring data (xx at the end is program version; use the highest version unless instructed otherwise by beamline personnel).

Top off the liquid N₂ to detector once every other day. Add liquid N₂ until it overflows slightly but don't let it splash into the electronics. Use the black nitrogen level rod, which is hanging on a hook near the dewar, to check the level.

In principle, the operator should have run for you an empty sample holder to make sure that the beam passes pretty much through the center of the holder. After it, the detector may need to move up and down in order for the barrel of the detector to fit into the window in the side of the sample chamber. If you have to [locate the beam](#) as described previously, decrease the gain to I1 and I2 to protect the meters and use the

Kodak burn paper. This should be done before the first sample is run. Simply follow the instructions starting here and continuing through **Print Optic and Motor Positions**. Then follow the instructions below using an actual sample. Start with a standard. You may use the checklist (at the end of this instructions) to ensure that no errors are made.

1. Make sure that someone has checked that the beam will in fact pass through the sample holder.
2. Turn on the helium cryostat and set it to desired temperature. Go to [Turning on the Helium Cryostat](#) for instructions.
3. **Back off the detector** and place a sample in the sample chamber. Refer to [How to change samples](#).

14. 1. The Main Program Screen

Before starting any scan, type in a filename and any comments to be associated with the file. Even the alignment files are saved and so it makes sense to give them a special name so they are not confused with the regular data files. For example, if a sample is called C1, then call its data files C1.001, C1.002, etc. Its x-scan alignment file should be called xC1.001 and identified as an x alignment file. Its z-scan (vertical) alignment file should be called zC1.001 and identified as a z alignment file in the comments section.

Defining Scans

The Scan Parameters section is used to define the type of scan that will be taken. It is faster to run the various types of scans if they are predefined and stored. To do this, press the Defined Scans button under Setup. A new screen ops up (Fig. 15). Depending on the scan type in Main window, you may see the screen exactly as in Fig. 15 for energy scans, or a similar window in which motor type scan are defined, if the scan type in main window is set to 1 motor.

Define, or check that different energy scans are defined. For most EXAFS or XANES scans, the scan is divided in several regions, each with different parameters. Make boundaries and step size (both in eV), and integration time in each of the regions. Give them easily recognizable names.

Once the various energy setups are defined, click Save and Exit. In main window click on the scan type area labeled Energy and choose 1 motor. Define (or check that they are defined) the following scans: Table_z with scan region boundary of 80, 89, region step size of .5 and integration time of 1 sec.; Table_x with scan region boundary of 15, 22, region step size of .2 and integration time of 1 sec. These boundaries are approximate and may need to change according to the sample position. When all of the desired scan region files are defined and the ones that are not needed deleted, press Save and Exit. The main menu will reappear.

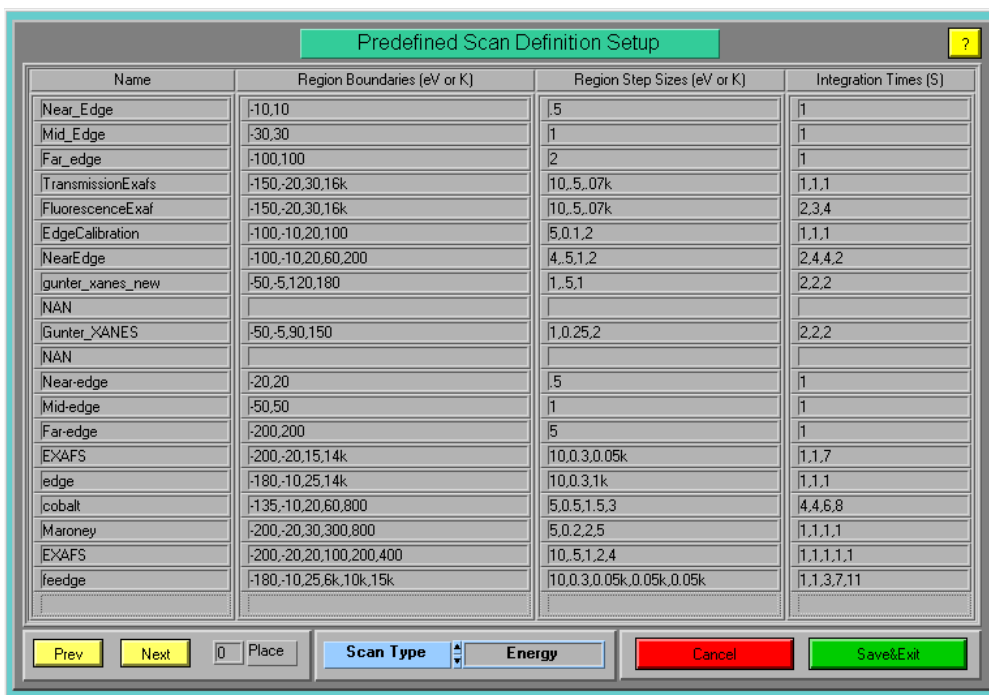


Fig. 15

14.2. X (Horizontal) and Z (Vertical) Alignment

Aligning Sample in X Direction

1. **Make sure the detector is backed off from the sample chamber.**
2. On the main menu, click on **+50** under Monochromator Control. This will move the monochromator so that the energy available during the calibration is 50 eV above the Mn edge. Supposedly, the alignment is independent of energy but it may vary slightly over the entire energy range and it makes sense to have the best alignment around the Mn edge.
3. Check to see that the filename is **xSampleName.001** and x alignment is mentioned in the comments.
4. If the scan type under the scan parameter section in the main menu is Energy, click on Energy and a list of choices will pop up. Choose **1 motor**.
5. Under the label Motor #1, some parameter will appear. Tbl2xSample is the motor to be moved when aligning the beam in the x direction. Tbl2zSample is the motor to be moved when aligning the beam in the z direction. It really doesn't matter which one is aligned first, but let us start with Tbl2xSample. Choose Tbl2xSample from the list.
6. Next, click on **User** next to Predefined Scans, and choose Table_x. Make sure that Number of Scans is set to **1**.
7. Click on Adjust Beamline Optics under Actions. The screen shown in Fig. 2 will appear. Set the horizontal slit size to **.5** mm and set the vertical slit size to **2.5** mm. This is done as follows:
 - i. Click on the box under Optics Select and choose Horizontal_Slit_Size from the list.

- ii. Click on the box under Destination and type in 0.5, and then press the Move Absolute button. The horizontal slit will change to 0.5 mm and register as such in the list.
- iii. For vertical slit of 2.5 mm, click on the box under Optics Select and choose Vertical_Slit_Size from the list.
- iv. Click on the box under Destination and type in 2.5, and then press the Move Absolute button. The vertical slit will change to 2.5 mm and register as such in the list.

Note: To move the slit relative to its current position by a desired distance, type in the desired distance in the destination box, and then press either the > or < buttons in the Move Rel to Current box. To manually move the slit, etc. manually from its current position, press any of the << (fast), < (slow), > (slow), or >> (fast) buttons below the Moving button.

- v. When all of the slits are properly adjusted, press Exit to get back to the main menu.
8. Turn on the beam and press the Start Motor Scan button. The screen shown in Fig. 7 will appear and scanning in the x (horizontal) direction will commence.
 9. In the upper left hand corner of the screen, there is a list of detectors with empty boxes to the left and right of the name of each detector. If the box to the left of I1 and on the box to the right of I0 is not black, then click on the box to the left of I1 and on the box to the right of I0. These boxes will turn black (as shown in the picture) and the plot will be of I1/I0, which is listed just above the detector list. Make sure that no other boxes are black; if they are, click on the black boxes to turn them white. Changing these setting while acquiring spectra will not result in any errors.
 10. To pause spectral accumulation, click the Pause button in the lower left of the menu. This button will begin to flash. To continue, click the Pause button again and the scan will continue with the values being plotted. The Abort button is used to abort the current scan and the Abort group button is used to let the current scan finish and abort all of the remaining scheduled scans.
 11. When the scan is complete, the screen will look like that in Figure 7, and the Continue button will be flashing.
 12. The cursor is shown by a pair of yellow cross-hairs. If only one or neither appear on the screen, move the cursor to the bottom of the screen and click on the little open lock and choose lock to plot. The cursor will suddenly appear, probably in the corner of one of the axis. Use the cursor to drag the crosshairs more to the middle of the trace and then click on the lock again and choose free. With the cursor near the middle of the trace, press Measure Peak Statistics button. The peak statistics will appear as shown in this figure and the cursor will jump to the Centroid. If it does not, press the Center Cursor on Peak button.

Note: The very large peak shown on the right of this trace shows the effect of a bubble in the sample. Unless the concentration of the element in the sample is very high, bubbles such as this should be avoided, provided the overall width of the opening in the sample holder is large enough to accommodate such a move. Use the

mouse to move the crosshairs to where you wish the center of the beam to be located in the x direction and then press Move to Cursor Position button.

13. Once the move is complete, press the Print button to obtain a copy of the scan. On the basis of the location of the center of the beam, determine how wide you can safely make the beam without hitting the sides of the holder or including a major bubble. Record this number in the data sheet.

Press the Continue button. The Main screen will appear (Fig. 1).

Aligning Sample in Z Direction

1. Check to see that the filename is zSamplename.001 and z alignment is mentioned in the comments
2. Click on Tbl2xSample which shows under the label Motor #1 and choose Tbl2zSample from the list that will appear. Next, click on User next to Predefined Scans, and choose Table_z.
3. Click on Adjust Beamline Optics under Actions. Set the horizontal slit to its optimal width as determined above and set the vertical slit to .5 mm. Before closing this screen by clicking on Exit, check the list to make sure that horizontal slit = its optimal width and vertical slit = 0.5 mm. Press Exit to get back to the main menu.
4. Press Start Motor Scan button. The screen as in Fig. 7 will appear and scanning in the z (vertical) direction will commence.
5. Once the scan is complete, use the cursor to drag the crosshairs more to the middle of the trace. With the cursor near the middle of the trace, press Measure Peak Statistics button. The peak statistics will appear as shown in this figure and the cursor will jump to the Centroid. If it does not, press the Center Cursor on Peak button. If the crosshair is not where you want to center the beam, use the mouse to move the crosshairs to where you wish the center of the beam to be located in the z direction and then press Move to Cursor Position button.
6. Once the move is complete, press the Print button to obtain a copy of the scan. Use the cursor to determine the maximum and minimum values of z without the beam hitting the sample holder and write these values on the printout of the scan. These values can be used to help relocate the beam if the sample turns out to have inhomogeneities. (See **Troubleshooting**.)
7. Press the Continue button. The Main screen will appear (Fig. 1). Click on Adjust Beamline Optics under Actions. Set the Vertical_Slit_Size to 2.5 mm. Before closing this screen by clicking on Exit, check the list to make sure that horizontal slit equals its optimal width and vertical slit is 2.5. Record these values on the spreadsheet and then press Exit to get back to the main menu.

14.3. Print Optic and Motor Positions

These are your true optic and motor positions and need to be saved only once unless you had to make major changes in the motor or optic positions. It is used for future reference purposes for you as well as the operator. If the operator makes changes in these

parameters, the changes should also be printed.

1. Click on Adjust Beamline Optics under Actions. The screen in Fig. 2 will appear. Press the Print Optic Positions button followed by Exit.
2. On the main menu, click on the Move Motors button under Actions. The screen shown in Fig. 3 will appear. Press the Print Motor Positions button and then press the Exit button.

14. 4. Recording Spectra

Move the detector forward into position.

Measure dark current (see p.6)

Calibrate Piezo (see p.7)

Scan the Sample

Detector 2 is in the center of the detector array and so will generally receive the most photons. However, during the sample run, the CHANNEL number of the rate meter should be set to 13. The ICR should never exceed 200 - 250,000 counts and should not exceed 80,000 if signal linearity is desired. If you are uncertain as to whether or not the amount of the element in your sample will cause the ICR rate to exceed this value, run a quick scan and observe the meter.

1. On the main menu, type in the **sample's filename**. If this is its first run, the filename should be **name.001**. If previous runs have been already completed, for example 9 runs, than the filename should be **name.010**.
2. Type in any **user comments** that you want to include with the file for identification purposes.
3. Under the scan parameters section click on 1 motor and choose Energy. Make sure that **Element** and **Edge** are correct.
4. Click on User next to Predefined Scans, and choose Your XANES or Your_EXAFS.
5. Change the Number of Scans to the number of scans to be run on this sample.
6. Press the Start Scan button and note the time the scan started on the spreadsheet.
7. The screen shown in Fig.16 will appear and the scan will start.
8. **Observe the values of I0, I1 and I2**. If I0 is small (< 1) but the beam current is large (such as 200 mA), see **Troubleshooting** page 14. If I0 is normal, check I1 and I2. If their values are not between 0.05 and ~ 0.8 , change their gains using the amplifiers shown on page 3, Figure 1 until their values are within this range.

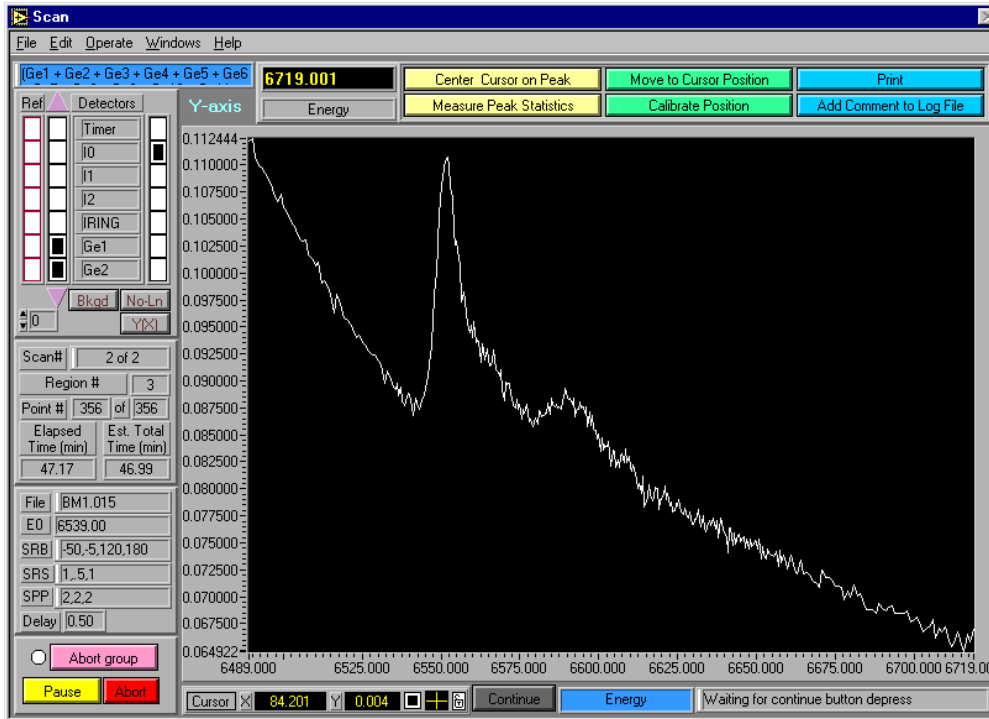


Fig. 16.

9. In the upper left hand corner of the screen, there is a list of detectors with empty boxes to the left and right of the name of each detector.

Note: IRING is a measure of the beam current. Ge1 through Ge13 are the so-called SCA sample rates that correspond to the gated counts for detectors 1 through 13. ICR1 through ICR13 are the total photon counts arriving at detectors 1 through 13.

10. Click the boxes to the left of Ge1 through Ge13 and click on the box to the right of I0. Make sure that no other boxes are black. The display will show a plot of $(\text{Ge1} + \text{Ge2} + \text{Ge3} + \text{Ge4} + \text{Ge5} + \text{Ge6} + \text{Ge7} + \text{Ge8} + \text{Ge9} + \text{Ge10} + \text{Ge11} + \text{Ge12} + \text{Ge13})/\text{I0}$.
11. During the course of a run it is possible by using the procedure described in step 9 to look at any parameter as a function of wavelength. If you click on the Y(X) button, the scan will change to the first derivative of the scan. Press again to get Y(X).
12. If something happens during the course of a run that makes its termination desirable, press the Abort button. If it is desired to abort the rest of the scans, press the Abort group button.
13. After all of the scheduled scans are completed, the Continue button will begin to flash. If desired, press Print to obtain a copy of the spectra.
14. If more scans of the same sample are desired, press the Continue button. In the main menu enter the number of extra scans you desire to run on this sample, and make sure that the filename is ready to increment to the next run number, and then press the Start Scan button.

15. If a new sample is to be started, click on the box to the left of I1 and on the box to the right of I0 to make them black. Make sure that no other boxes are black and then press the Continue button.

For a new sample, check How to change samples (p.14), and repeat the above procedure. Use the spreadsheet, if needed.

Troubleshooting

No signal on I0

Try to locate the problem using the following:

1. Did you open Safety Shutter and Photon Shutter? (see page 1.)
2. Check TV monitor (channel 9) to see if the beam dumped. If so, wait until it comes back again.
3. Locate X3B Interlock and Safety box (black metal box on the crate high above beamline computer). See if any of the red lights are on. If so, you must call Op-co.
4. Did you accidentally turned the Lockout Key (yellow-orange color key) instead of Hutch Door Kirk Key (green) while interlocking the hutch? If so, call Op-co to enable the beamline again.
5. Did the computer hung up and/or you had to reboot it? If so, did you answer Crate or File when prompted for the Monochromator Position? If you answered File, you may need to do Monochromator Calibration.
6. Move the energy to 100 eV above the edge. See if you can read any current on I0. Increase sensitivity (Fig. 8 and p. 7-8). If you can read any current, redo piezo calibration.
7. All motor and optics position have to be re-checked. Locate the printout of all motor positions you have. Compare with the current settings. If you notice a change, try to move the motor (unless listed as restricted motor) to the printout position.

Note: Under no circumstances you should attempt to move monochromator motors or any restricted motor.

8. Try to find the beam position by placing a piece of Kodak Linagraph paper to check whether the beam enters the cryostat (see p. 11).
9. In any other case, call X3B staff (7am to 10 pm), or Op-co for help.

Strange Peaks or Noise in the Scans of Individual Detectors

Samples that are inhomogenous or include ice crystals can scatter x-rays in a peculiar manner resulting in strange peaks or noise in the scans for individual detectors that do not appear in all of the detectors. If these imperfections are not too extreme, it is sometimes possible to move the sample so the beam exposes a different area of the sample. If movement is limited in the x direction because of the dimensions of the sample holder, it is still usually possible to move the sample in the vertical direction. This is done as

follows:

1. If you suspect a sample of being inhomogenous or including ice crystals, view the detector current/I0 for each individual detectors. Imperfects can be quickly seen. Note the detectors that have large glitches and hence should not be used in analyzing the sample. If too many detectors are compromised, the sample must be moved.
2. Go to the Aligning Sample in Z Direction and follow all of the steps through step 6.
3. Do not move the cursor to the position it was previously moved to in the run that resulted in the strange peaks or noise in the scans for individual detectors. Instead look at the copy of the previous Z scan for this sample and determine where the center of the beam can be placed without it hitting either edge of the sample holder and still be in a new location. If necessary, decrease the height of the vertical slits as much as possible without actually decreasing the physical height of the beam.
4. Once these new values are determined, move the cursor to the place you want to center the beam and press Move to Cursor Position button. Exit this menu and change the vertical slit if necessary. Note this new value of the 'center' of the sample on the spreadsheet as well as any changes in vertical slit size.
5. Initiate a new scan and observe detector current/I0 vs. energy for each individual detector. If too many detectors are compromised, move the sample one more time, finding a new z value for the center of the beam. If too many of the resulting scans are still faulty, the sample will probably have to be replaced.

Sample Noise Increases Without a Decrease in I0

If too much time is taken in inserting a new sample or if the sample window is not firmly held in place by the vacuum, it is possible for a fair amount of ice to form on the sample. Such ice can result in more sample noise, and in addition, as the thickness of the layer of ice either increases or decreases, can result in a decrease or increase in the height of the fluorescent peak as well as a distortion of the sample fluorescence spectra. If such changes are observed in your sample spectra, check the sample for ice. In addition, I1 and I2 will both decrease without a concomitant change in I0.

Sample Noise Increases Dramatically Without a Decrease in I0

Sometimes sample noise will steadily increase with time and eventually strange peaks or noise in the scans for individual detectors will appear that do not appear in all of the detectors. In addition, I1 and I2 will not decrease without a concomitant change in I0. In this case it is very likely that water is condensing on the window between the sample and the detector. This will occur when the humidity is high. The solution is to tape 2 small tubes to either side of the detector cap and connect the tubes to the supply of dry nitrogen gas found in the hutch and blow dry nitrogen on the window. You will need an operators help to locate everything so if the day is very humid, ask for this solution to be applied before the problem occurs. This problem is not something you want to occur during a night run.

Sudden Decrease in I0 While Beam Current Remains High

Sometimes there is a sudden decrease in I0, I1 & I2 while the beam current remains approximately constant. This usually occurs immediately after a piezo update whose

screen does not appear during the update. What happens is as follows. A piezo update only adjusts the piezo crystal for the first point and assumes that all of the rest of the points will follow the same curve they took during the full calibration. Sometimes this first point is far off from where it should be located with the result that the updated piezo calibration is incorrect for the entire energy range. Since the beam is not longer located where it should be or properly focused, I0, I1 and I2 will all decrease substantially.

Stop the scan and run a full piezo calibration. If a faulty piezo update is the problem, the calibration screen will include an update that may be as much as 100 volts lower than the full calibration. If this is observed, a faulty update was indeed the problem and can be corrected by running a full calibration.

Note: All full calibrations should have the same general shape and voltage range. If this is not the case, you must manually locate the approximate voltage for the first point. Return to the main menu and move the monochromator by pressing the 0 button in Monochromator Control section. Go to the Piezo Control panel, which is immediately above the Germanium Detector Computer. Change the switch from **Computer** to **Manual**. While watching I0, manually change the voltage applied to the piezo crystal by slowly rotating the rheostat below the **Computer** to **Manual** switch. Change the rheostat until I0 reaches a maximum and then stop.

Be very careful that the applied voltage does not exceed -500 volts. The dial is a 10 turn rheostat and varies the applied voltage from 0 to -1000 volts. The actual reading on the dial will be, for example, 3.65 which is equal to -365 volts. If any operator is around, let him show you how to do this the first time. If no operator is available, call one a home before you do anything and do not attempt this procedure without their permission and initial instruction because applying too much voltage will destroy the crystal, terminate your run and cause considerable down time since there is not a supply of spare piezo crystals.

Once I0 reaches a maximum, change the switch from **Manual** to **Computer** and lock the rheostat in position. Then run another full piezo calibration. While the calibration is running observe the value of I0 and see if it generally reaches a maximum that is close the value obtained above manually for each energy. If the maximum value of I0 obtained by the computer controlled calibration is much lower than what was found manually and the resulting calibration curve is very different from all of the other successful calibration curves, notify an operator of the problem.

If the problem with the update of the piezo calibration is a recurring one, and you have an overnight run coming on, change the frequency of updates to a number of scans that is much larger than the number you plan to run for that particular sample.

The Full Piezo Calibration Is Strange or No Spectra Is Visible During a Sample Scan

If during a full piezo calibration, you obtain a strange scan where the piezo voltage hardly changes at all with energy (unless you are scanning elements with $E_0 > 11$ keV), or during a sample scan no spectra is observed although the program claims to be scanning, then the monochromator may be stuck. If this is the case, look at the value of the current energy. If this value of energy does not change even though the calibration or scan continues, the monochromator motor is probably stuck.

First, look at the beige box that is almost immediately behind the small refrigerator and is to the left of the X3B rotary table. (See Fig.17.) This box has two cables running into it and two red lights on the top. **If the red lights are lit, call for help. This is not something you can fix!**

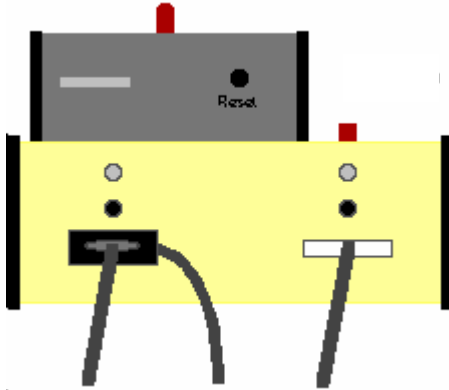


Fig. 17.

The red lights indicate that the safety interlock for the monochromator motor has been engaged and the software problem can't change the energy until the interlock has been disengaged. If you bypass this interlock you can destroy the monochromator crystal which costs about \$80,000 and 6 months to replace because a new single crystal has to be grown.

What they do is as follows:

1. They first check the program and make sure that the monochromator is set to an energy below E0.
2. Next they remove the 2 cables from the box and put the shorting plug on the connector whose wire has the shorting plug is attached to it by a cable.
3. Then they go to the software program and press the **0** button in the Monochromator Control section to bring the motor to a monochromator setting of E0.
4. Once there, the shorting cable is unplugged and the 2 cables are reconnected to the beige box.
5. Next, press the 2 reset buttons of the beige box to make the red lights go out.

If the red lights are out, you may be able to solve the problem yourself. Check the Coarse monochromator calibration section below.

Coarse monochromator calibration

Monochromator calibration is needed if there was an accidental abort of scan, power loss or accidental click on Calibrate Monochromator (in Fig. 5). Read through the following steps and understand them before calling for help. You will need the password for the monochromator calibration (in step 6).

1. Go to the front side of the monochromator assembly, located between X3b and x9a hutches. Read the value of the rotary monochromator position at the scale of

- the monochromator. The scale at the monochromator ring reads degrees and the scale at the left of the monochromator ring displays minutes and seconds.
2. Translate the value in decimal (You can use, for instance, Windows calculator: type in the degrees-minutes-seconds, click on Inv-dms.)
 3. Subtract the fixed offset of **13.1206** from the above value.
 4. Next, read the value (in mm) of the monochromator trolley position at the scale located at the back of the monochromator assembly. You will have to walk behind the X3b hutch and further towards the storage ring, like you did while doing safety checklist. You will find the ruler at the bottom of the back side of the monochromator.
 5. Subtract the fixed offset of **100 mm** from the trolley position you read in step 4.
 6. From the main screen, click on Move Motors. Click on the box under Motor Select, select Rotary from the pull-down menu and read the displayed position. Compare it to the one you obtained in step 3. If the two numbers do not match within $\frac{1}{4}$ of a degree, click on the box under Call This Position, type in the number you found in step 3 and click Calibrate. You will be prompted for the password. Call beamline staff (7am to 10 pm) to obtain the password. Type in the correct position from the monochromator scale, and click Save and Exit.
 7. Repeat step 6 for Trolley position.
 8. Do *Dark current (offset) Measurement* and *piezo calibration* (both on page 8).
 9. Run *Fine Monochromator Calibration* (page 10) using first far-range scan (200 eV around E0, 5 eV steps), and then mid-edge and near-edge scans to further refine the location of the monochromator.

Error on Power Failure and how to restart the system

- On power failure check and write all errors on Motor Protection Box and/or software related error and/or any other error in the Log book
- If Motor protection box says POWER RESTORED, PRESS ENABLE ALL
- Check if the bars near the EDGE of XDAC (software) window are moving and if there are lights flickering on CAMAC crate (On Control Module, check Far Right Module). If they are working that means the computer is communicating with CAMAC and there is NO NEED to restart the software
- In that case, pressing ENABLE ALL on Motor protection box will work
- If the software is not working and/or there is no communication between computer and CAMAC, follow the procedures below
- Press DISABLE ALL (even if they are disabled)
- Restart computer (if needed), press “yes” to “SECOND BACKUP PROGRAM”
- Shut down software
- Start software XDAC version 97 (in the center of monitor screen)
- Click Yes to ERROR indicating that the MOTOR “POSITION DOES NOT MATCH”
- Compare the values of various motor positions on the table Col. 1 and Col. 2
- If the positions are all ZERO, click and select the values FROM FILE

- If the positions are some valid numbers and/or if ONE number in these positions mismatches, click and select the values FROM CRATE.
- Press ENABLE ALL on Motor protection box
- Run hyper terminal