Data Collection at GM/CA using Blu-Ice

Content questions and comments: Ward Smith .
Technical questions and comments: Webmaster.

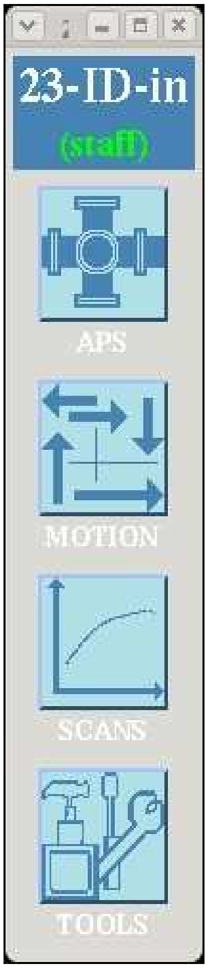
EPICS Control

Starting MEDM

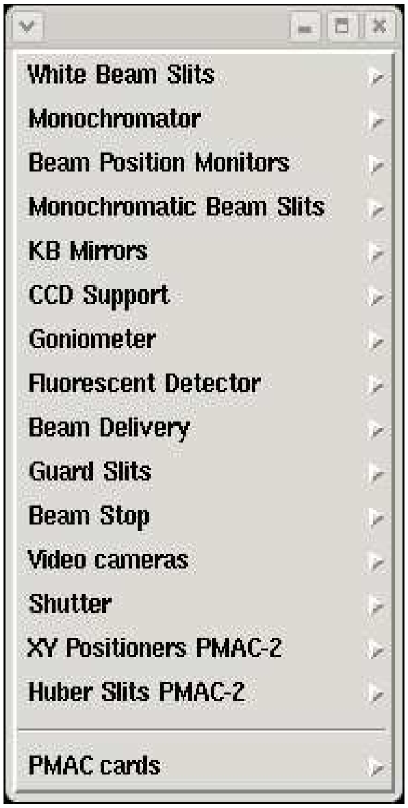
MEDM is the EPICS interface for low-level control of beamline motors

MEDM is started by typing "23id" in a terminal window.

The MEDM interface main menu will appear.



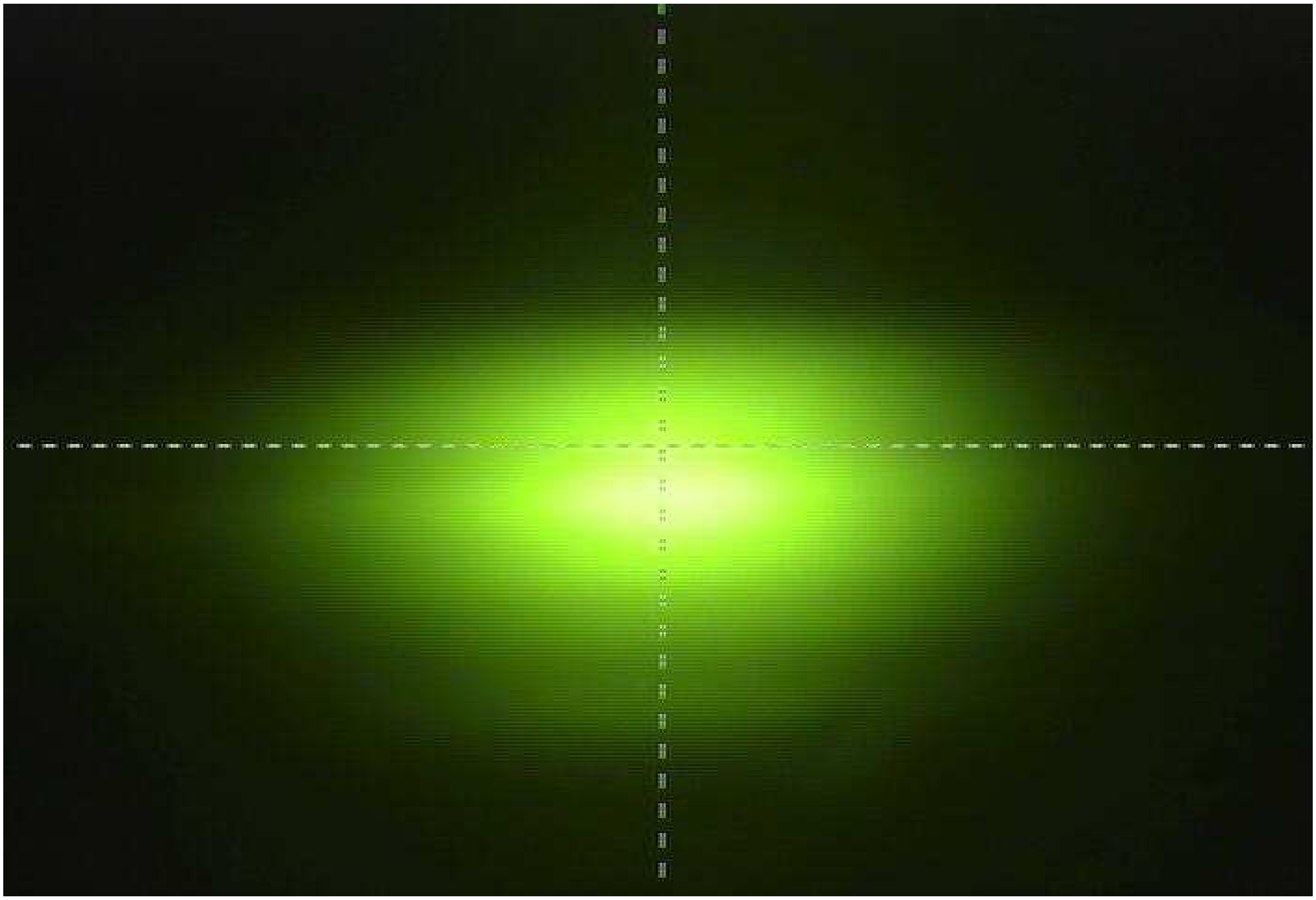
On the MOTION menu item select the desired device



Beam Alignment

- Place the ***YAG*** crystal on the magnetic base vertically and in field of view of the ***High Resolution*** camera.
- Visually align crosshairs in ***High Resolution*** camera by moving Beam delivery Horizontal and Vertical to move the camera support to bring the crosshairs to the image of the beam.

Remember hole in ***High Resolution*** camera will obscure the image at magnifications ~ 1.8 or less!



Goniometer Alignment

- Center the alignment pin to establish rotation axis position ***High Resolution*** camera.
- Move Goniometer Vertical to place the center of rotation at the crosshairs on the ***High Resolution*** camera.
- Scan GS slits in V and H to center slits on beam
- Attenuate and expose 0.1 second w/o sample to find any beam leak around beamstop
- Adjust BS as needed to eliminate leak

Beamstop

40.000 mm

Content questions and comments: [Ward Smith](#).
Technical questions and comments: [Webmaster](#).

Getting Started

Starting Software

The beamline computers are Linux workstations.

The beamline computers control the detector and store the data and analysis files on our strorage array. Login on MAR1 using your user name and password. Remember UNIX is case sensitive.

Start MAR CCD detctor software with the mar detector icon

Start Blue-Ice on BL1WS3.GMCA.APS.ANL.GOV with the Blu-Ice icon

We recommend subdirectories for each data set you collect.

Subdirectories are automatically created in the data collection tab.

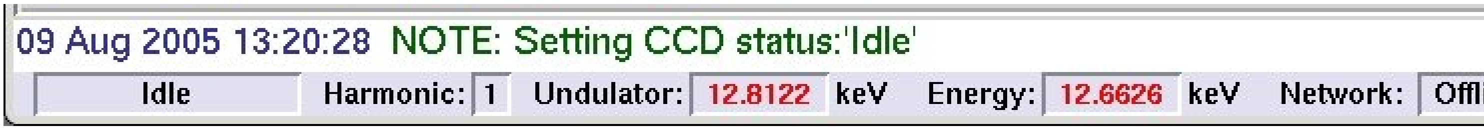
Users should use the additional Linux workstations (BL1WS1 BL1WS2 or BL1WS6) for data processing.

Blu-ice Interface

The BLU-ICE software that controls the beamline is organized into tabs. Different sections of the program can be accessed by clicking on the tabs. Users are allowed to enter the Hutch, Collect , Scan and Users Tab.

Status Bar

The ***status bar*** at the bottom of the screen shows the status of the software and beamline hardware.



09 Aug 2005 13:20:28 NOTE: Setting CCD status:'Idle'

Idle Harmonic: 1 Undulator: 12.8122 keV Energy: 12.6626 keV Network: Offl

The box on the left discribes the status of data collection with the following:

Idle	No data collection taking place
Acquire	Shutter is open and exposing a frame
ReadOut	Getting image from the detector
Correct	Applying correction to image
Writing	Writing image to disk

Also displayed on the task bar:

- Current Undulator Harmonic, Undulator position and Monochromater energy . Different units (KeV, A and eV) can be displayed by clicking on Energy value
- Network status
- Shutter position (open or closed)
- Current Time

Network status

Beamline software may be used by multiple computers simultaneously. However, only one user at a time can implement movements. In order to control the beamline software the network status must become ***Active***. To become active click on the word ***Passive*** written in black at the bottom of the screen next to network. It should now say 'Active ' in red letters.

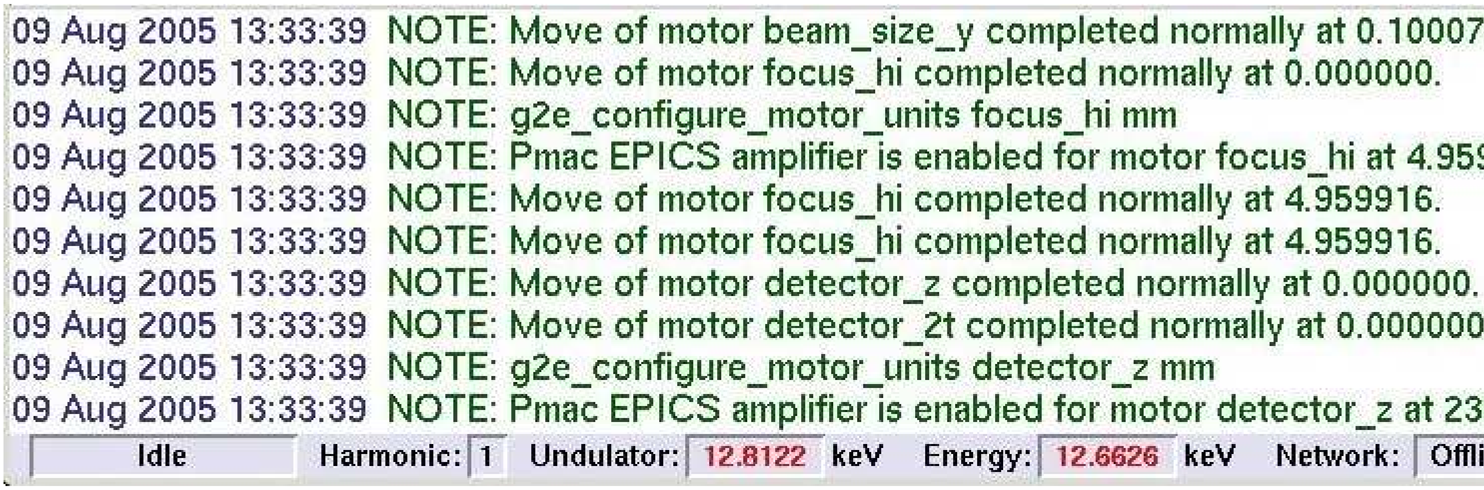
Network: Active

Network: Passive

If the status reads ***Offline***, you do not have privilege to run BLU__ICE, contact staff to set the appropriate privileges.

Message Window

The ***message window*** just above the ***status bar*** displays important information regarding data collection. This area can be enlarged by dragging the upper egde of this window with a mouse.



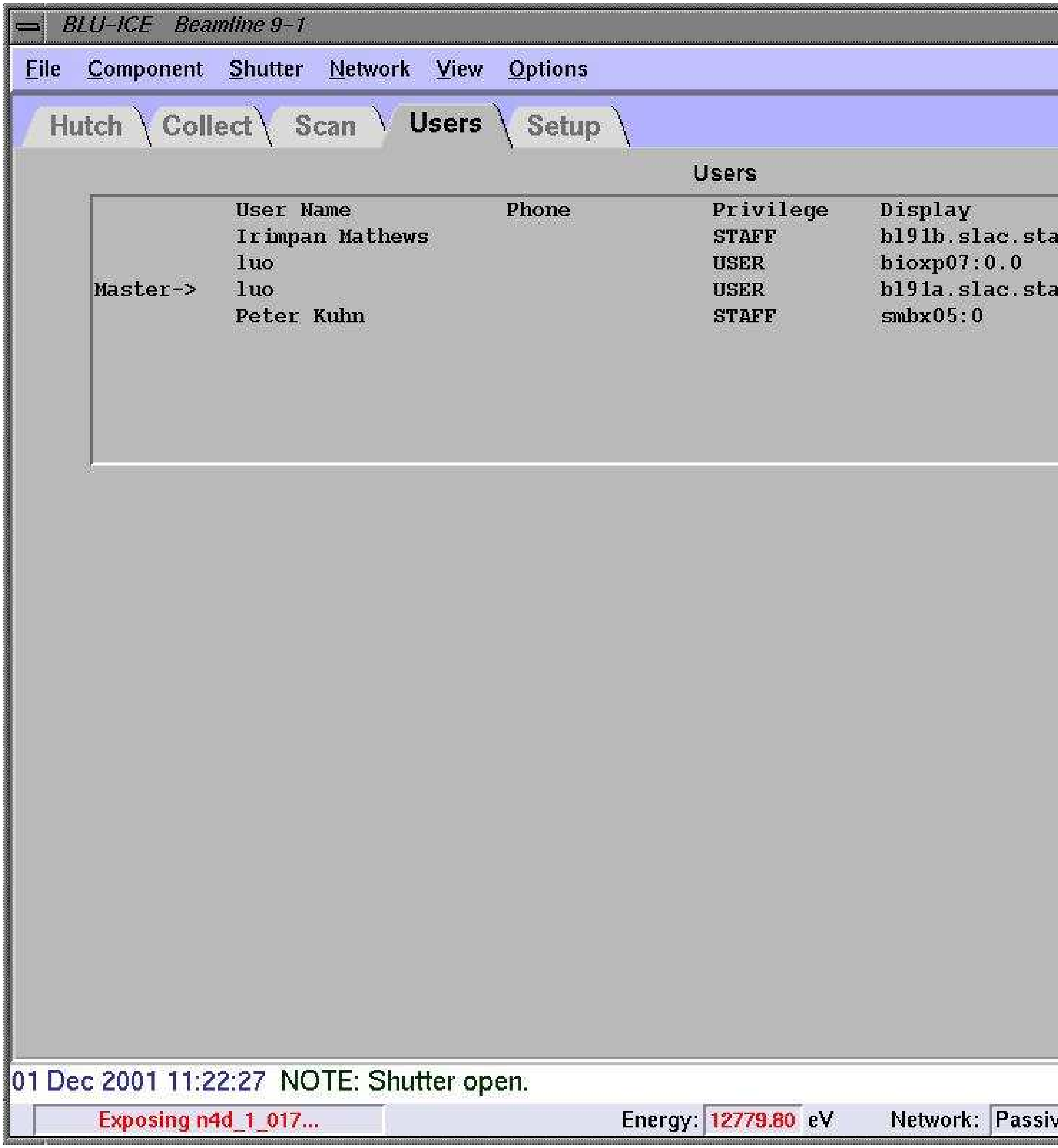
```
09 Aug 2005 13:33:39  NOTE: Move of motor beam_size_y completed normally at 0.10007
09 Aug 2005 13:33:39  NOTE: Move of motor focus_hi completed normally at 0.000000.
09 Aug 2005 13:33:39  NOTE: g2e_configure_motor_units focus_hi mm
09 Aug 2005 13:33:39  NOTE: Pmac EPICS amplifier is enabled for motor focus_hi at 4.95
09 Aug 2005 13:33:39  NOTE: Move of motor focus_hi completed normally at 4.959916.
09 Aug 2005 13:33:39  NOTE: Move of motor focus_hi completed normally at 4.959916.
09 Aug 2005 13:33:39  NOTE: Move of motor detector_z completed normally at 0.000000.
09 Aug 2005 13:33:39  NOTE: Move of motor detector_2t completed normally at 0.000000
09 Aug 2005 13:33:39  NOTE: g2e_configure_motor_units detector_z mm
09 Aug 2005 13:33:39  NOTE: Pmac EPICS amplifier is enabled for motor detector_z at 23
```

Idle Harmonic: 1 Undulator: 12.8122 keV Energy: 12.6626 keV Network: Offli

Users and Setup Tabs

- The ***Users Tab*** lists the names and status of all the people monitoring data collection.

- It also lists versions of software the user is running.



```
          User Name          Phone        Privilege    Display
          Irimpan Mathews                 STAFF        bl91b.slac.sta
          luo                             USER         bioxp07:0.0
Master->  luo                             USER         bl91a.slac.sta
          Peter Kuhn                      STAFF        smbx05:0
```

01 Dec 2001 11:22:27 NOTE: Shutter open.

Exposing n4d_1_017...

Energy: 12779.80 eV

Network: Passiv

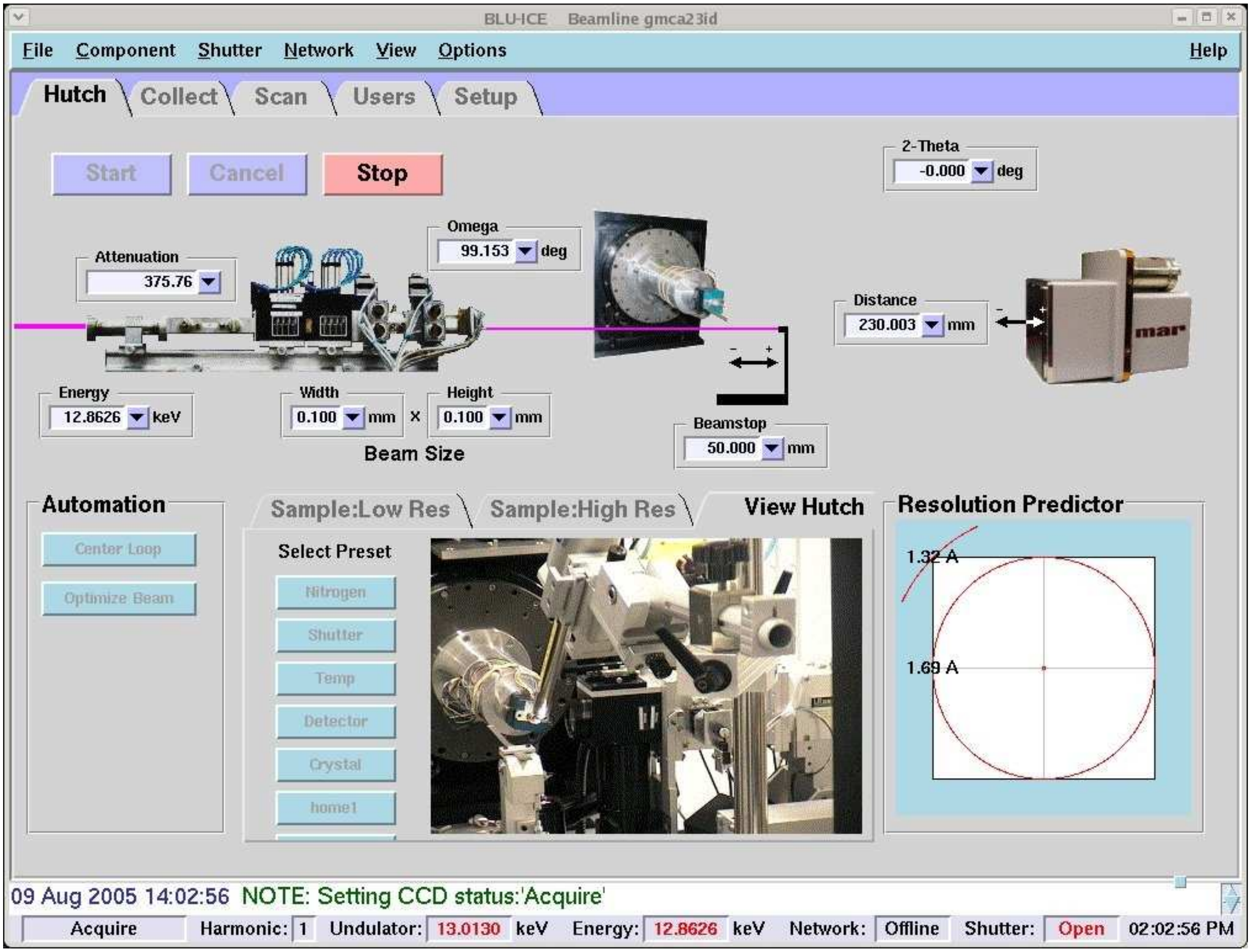
- Users can not generally access the ***Setup Tab***; staff uses this for beamline alignment.

Content questions and comments: Ward Smith.
Technical questions and comments: Webmaster.

Hutch Tab

- Mounting the Crystal
- Crystal Centering
- Hutch View
- Adjusting Beam Size and Energy
- Adjusting Detector Position
- Determining Resolution Limits
- Pull-Down Menus
- Main Index

The ***Hutch Tab*** allows the users to adjust various parameters for data collection. The changes are shown in red, however, nothing actually changes until the ***Start*** button is pressed. The network mode must be ***Active*** to execute changes. The ***Cancel*** button resets the parameters to their original values.



2-Theta: -0.000 deg
Omega: 99.153 deg
Attenuation: 375.76
Distance: 230.003 mm
Energy: 12.8626 keV
Beam Size: Width 0.100 mm × Height 0.100 mm
Beamstop: 50.000 mm
1.32 A
1.69 A

09 Aug 2005 14:02:56 NOTE: Setting CCD status:'Acquire'

Acquire Harmonic: 1 Undulator: 13.0130 keV Energy: 12.8626 keV Network: Offline Shutter: Open 02:02:56 PM

Mounting the Crystal

- Move the detector away from the sample for easy mounting.

- Practice mounting a pin with a blank loop on the goniometer to make sure your system is compatible with our cold stream. Contact staff if you run in to any problems.
- Mount your sample on the goniometer head.

Crystal Centering

1. Select the zoom level to find the sample on the Low Resolution camera.
2. Click on the sample to center it.
3. Rotate phi 90 degrees.
4. Click on the sample once again to center.
5. Select higher zoom level.
6. Repeat steps 2 to 4 to align the crystal accurately.
7. Once you see the sample on the High Resolution camera, repeat.
8. Click on the sample to center it.
9. Rotate phi 90 degrees.
10. Click on the sample once again to center.
11. Select higher zoom level.
12. Repeat these steps to align the crystal accurately.

If you want to hit the crystal in a very precise spot and you cannot click on it accurately enough with the mouse you can use the "Move Sample" buttons. The double arrow buttons translate the sample horizontally by half a screen. The single arrow buttons translate vertical or horizontally by one pixel.

View Hutch

Five preset views of the hutch (can be selected from the ***View Hutch*** window.

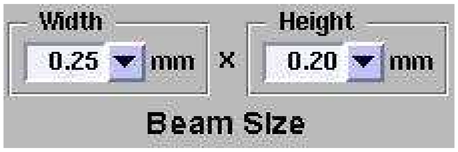
- ***Detector*** shows part of the goniometer, including the detector, sample, cryostream and rotation axis.
- ***Crystal*** shows a closeup view of the sample.
- ***Temp*** shows the end LN2 level and temp of the CryoJet.

Users can also access a web browser displaying the beamline Video System.

Adjusting Beam Size and Energy

Beam Size

- To adjust the beam size click on the windows labeled ***Width*** and ***Height*** under the beam size label. The white box on the Hutch monitor is 100x100 microns with the camera zoom level at 'high'. The beam should be optimized after changing the beam size using the ***Optimize Beam*** button.
- Ideally the beam size should be the same as the crystal size.


Width
0.25 mm
x
Height
0.20 mm
Beam Size

Energy

- To change the energy use the box labeled ***Energy*** in the ***Hutch Tab***. Energy has to be entered in KeV and click

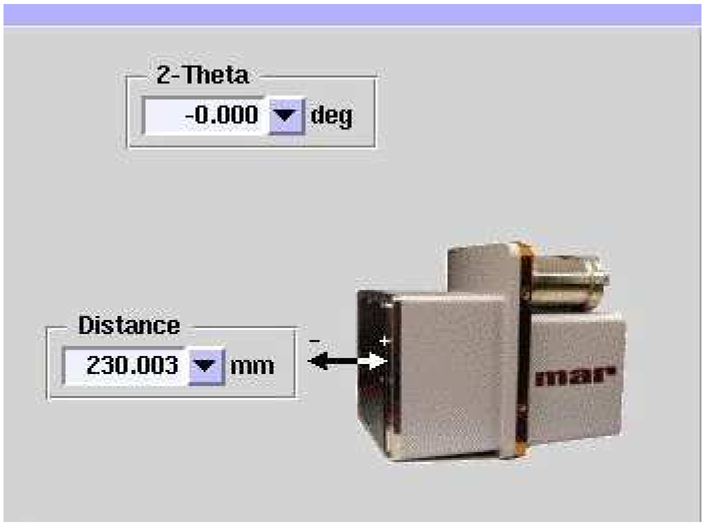
Start to effect the change.

- Beam should be optimized after a large energy change.

Energy

14468.30 eV

Adjusting Detector Position



2-Theta

-0.000 deg

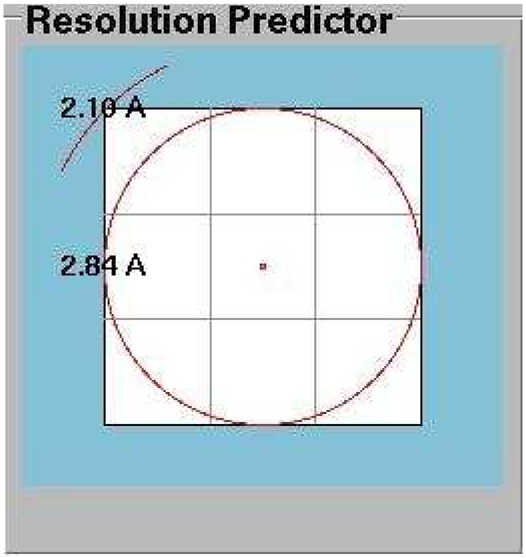
Distance

230.003 mm

- To adjust the detector distance or 2-theta, change the values in the corresponding box. Press ***Start***.

Determining Resolution Limits

- The resolution limit at a particular detector position may be determined using the box labeled ***Resolution Predictor***. When a change in the detector position or energy is made, the view in this box is updated. Press ***Start*** to move to this new position, ***Cancel*** will return 'red' values to the original values.
- The label on the largest circle that fits in the box shows the resolution that can be fully recorded. Partial circles represent resolutions that can be reached partially.



Pull-Down Menus

- The pull-down menus such as those for detector position control (i.e., distance, 2-theta) show the minimum and maximum values for the particular control with a range of values in between.
- Any values can be input ***within*** this range.

Content questions and comments: *Ward Smith* .
Technical questions and comments: *Webmaster*.

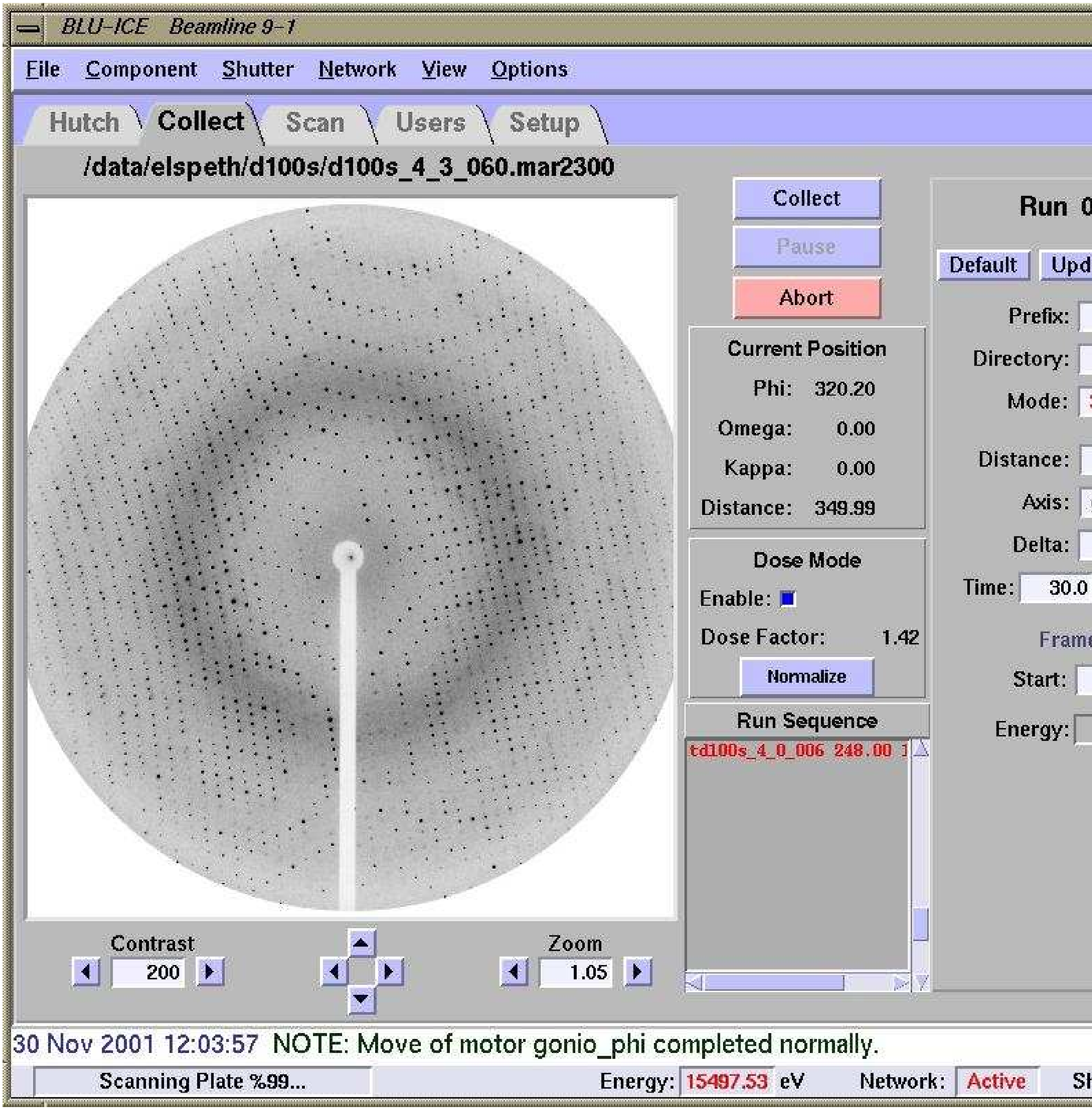
Last modified: Thursday, 28-Jul-2005 10:43:29 PDT.

Collect Tab

The ***Collect Tab*** is dedicated for data collection.

Collecting a Snapshot

Initial screening of crystals can be done with ***Run 0***. This is useful for taking snapshots in order to test crystals and plan strategies for data collection.



/data/elspeth/d100s/d100s_4_3_060.mar2300

Current Position

Phi: 320.20

Omega: 0.00

Kappa: 0.00

Distance: 349.99

Dose Mode

Enable: ■

Dose Factor: 1.42

td100s_4_0_006 248.00

Time: 30.0

Contrast 200

Zoom 1.05

30 Nov 2001 12:03:57 NOTE: Move of motor gonio_phi completed normally.

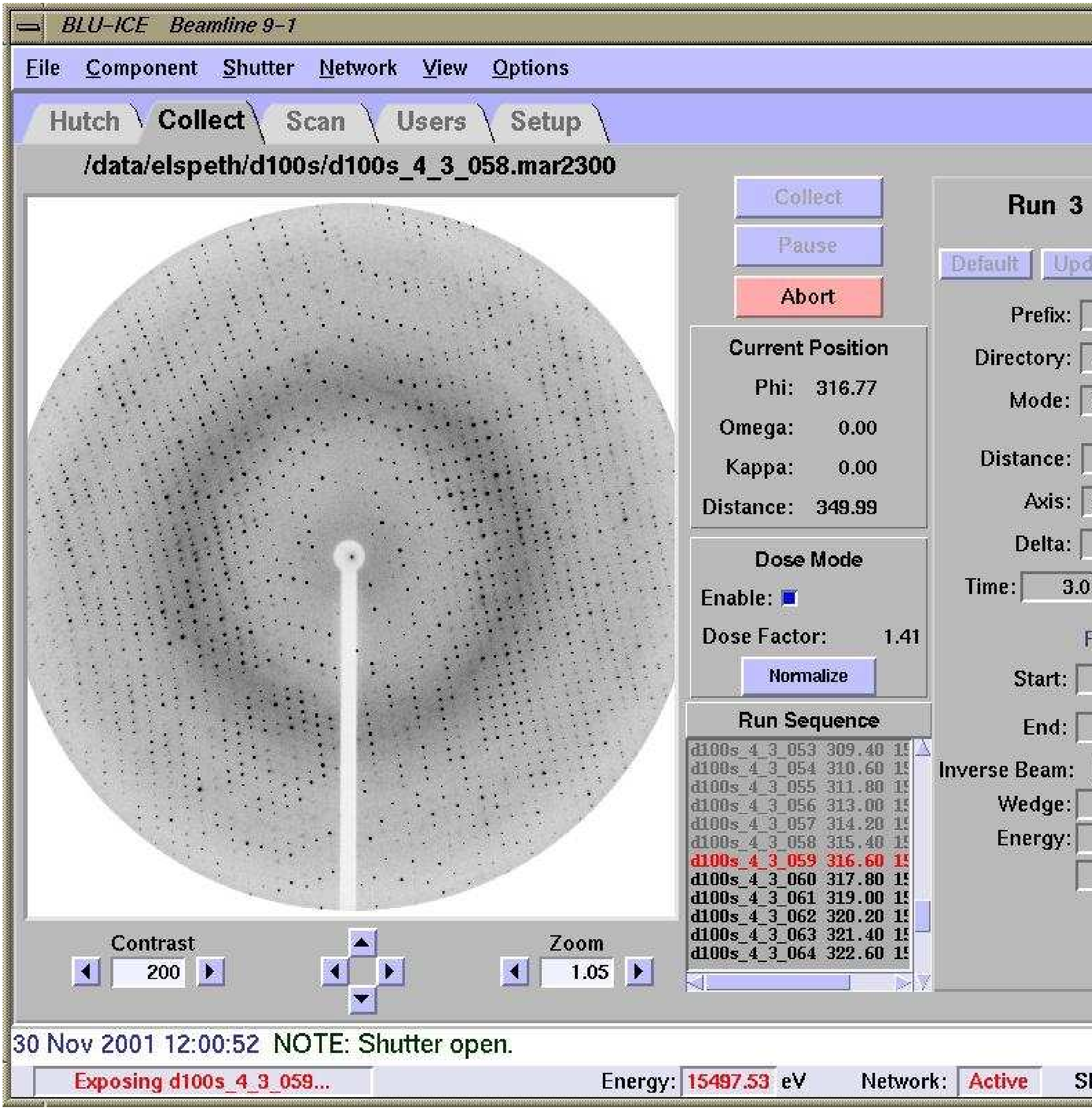
Scanning Plate %99...

Energy: 15497.53 eV

Network: Active

Data Collection Run

- All other ***Runs*** (Run 1, Run 2, etc.) can be used for data collection.
- Each ***Run*** is independent and this allows the users to schedule an extensive data collection with changes in detector distance, exposure time, etc. (eg. high resolution and low resolution passes).
- To create a new ***Run***, click on the '*' tab below the '0' tab.
- After setting all parameters to your desired values click ***Start*** to collect an image.
- A red value indicates that just before data collection starts, a change will be executed.
- Each run is automatically executed, consecutively .
- You must be the in the network ***Active*** mode to collect an image.



/data/elspeth/d100s/d100s_4_3_058.mar2300

Current Position

Phi: 316.77
Omega: 0.00
Kappa: 0.00
Distance: 349.99

Dose Mode

Enable: ■
Dose Factor: 1.41

Time: 3.0

Run Sequence

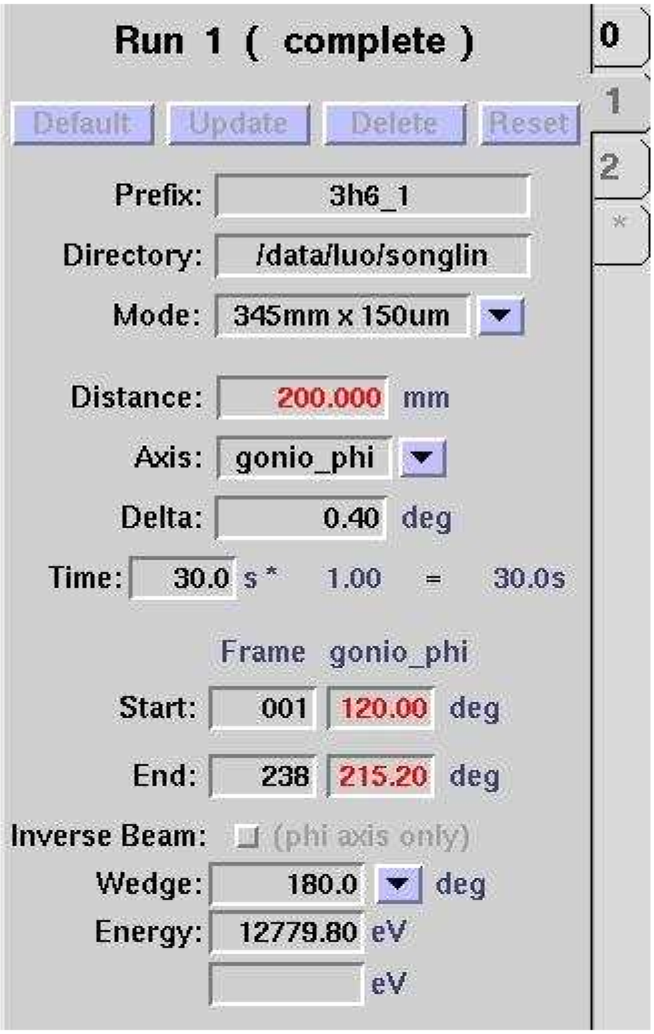
```
d100s_4_3_053 309.40 15
d100s_4_3_054 310.60 15
d100s_4_3_055 311.80 15
d100s_4_3_056 313.00 15
d100s_4_3_057 314.20 15
d100s_4_3_058 315.40 15
d100s_4_3_059 316.60 15
d100s_4_3_060 317.80 15
d100s_4_3_061 319.00 15
d100s_4_3_062 320.20 15
d100s_4_3_063 321.40 15
d100s_4_3_064 322.60 15
```

Contrast: 200
Zoom: 1.05

```
30 Nov 2001 12:00:52 NOTE: Shutter open.
```

Exposing d100s_4_3_059...
Energy: 15497.53 eV
Network: Active

Commands and Parameters

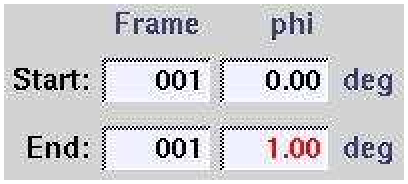
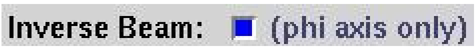
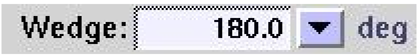
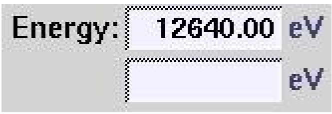


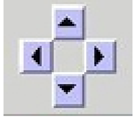
Run 1 (complete)

Prefix: 3h6_1
Directory: /data/luo/songlin
Mode: 345mm x 150um
Distance: 200.000 mm
Axis: gonio_phi
Delta: 0.40 deg
Time: 30.0 s * 1.00 = 30.0s
Start: 001 120.00 deg
End: 238 215.20 deg
Inverse Beam: ☐
Wedge: 180.0 deg
Energy: 12779.80 eV

- Red indicates that the value set for data collection is not the same as the current position, and that a move will be made before data collection starts.
- If you click on ***Default***, the current values of distance, axis and energy will be selected and displayed. In addition, prefix will be set to 'xtal', directory to '/data/username', CCD mode to 'slow', delta to 1.00 deg, time to 1.0 sec, and frame start to 001. The default mode for Q315 is 'unbinned'.
- Click on ***Update*** to update only the fields for the current motor positions.
- Although not advised, you can use the ***Delete*** command to delete the run that you are in. You cannot delete Run 0. Be careful when using this command. Once a run is deleted, you cannot return to it.
- Although not recommended, you can use the ***Reset*** command to re-use a run.

 Prefix: test	Filename root or file prefix
 Directory: /data/awu	Shows the directory in which the image will be saved (new directories are created automatically).
	***MARMosaic CCD*** detector collection - no options.
 Distance: 200.000 mm	- Distance between detector and sample in millimeters. Mimimum distance varies between beamlines. Contact the support staff for data collections requiring shorter distances. - When moving the detector in, be careful not to hit the

	beamstop.
 Axis: phi - ***Phi*** - ***Omega***	- ***Phi*** is used. - ***Kappa*** is not available.
 Delta: 1.00 deg	Degree of rotation during exposure (commonly 0.5 to 1 deg, depending on spot overlapping). Values for the delta phi can be determined using the TESTGEN option of Mosflm.
 Time: 40.0 s * 0.67 = 26.8s	Length of exposure time in seconds.
Phi	Indicates the phi value for the current image
 Frame phi Start: 001 0.00 deg End: 001 1.00 deg	***Start:*** The number and phi value assigned to the first image. ***End:*** The number and phi value assigned to the final image.
 Inverse Beam: ■ (phi axis only)	- Rotates the crystal by 180 deg to collect the friedel pairs for the input phi range. - If wedges are used, the inverse beam is collected before changing the energy.
 Wedge: 180.0 deg	The phi rotation range that is collected successively at each energy.
 Energy: 12640.00 eV eV	As you enter an additional energy, an empty box appears for further energy entries.
Collect New Dark	When checked, forces a new dark image. This image is used to correct for background noise. It is not normally necessary to collect new dark because the software does it automatically when required.
 Contrast 200	The default image Contrast value is set to 200. Change this value for a desirable contrast between spots and background.
 Zoom 1.0	Click on this to zoom in or out of the diffraction image.

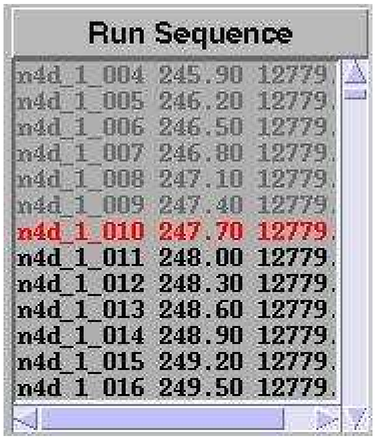
Move Arrows 	Moves the image within the display box.
	Double click on the diffraction image to analyze it with ADXV

Data File Naming

- Each data file in your selected run sequence is named in the following way: 'file prefix'__'run number'__'energy number'__'image number'.img.
- For example, a file could be named data__2__E2__010.img. This image is in the 10th frame collected in Run 2 at the 2nd energy.
- If two images happen to have same name after resetting the run, older images will be written to the OVERWRITTEN subdirectory. However, if the users start to collect the same image again and again, the file will be overwritten in the OVERWRITTEN subdirectory.
- With only one energy level selected, the file would be name 'file prefix'__'run number'__'image number'.img.

Run Sequence

The ***Run Sequence*** for a simple data collection shows image prefix, run number and the frame number. The progress of the data collection is indicated by changes in the color of the file names. The current active frame is shown in red. Completed frames and the pending frames are in grey and black, respectively.



Run Sequence

```
n4d_1_004 245.90 12779.
n4d_1_005 246.20 12779.
n4d_1_006 246.50 12779.
n4d_1_007 246.80 12779.
n4d_1_008 247.10 12779.
n4d_1_009 247.40 12779.
n4d_1_010 247.70 12779.
n4d_1_011 248.00 12779.
n4d_1_012 248.30 12779.
n4d_1_013 248.60 12779.
n4d_1_014 248.90 12779.
n4d_1_015 249.20 12779.
n4d_1_016 249.50 12779.
```

Your run sequence might become complicated depending on the values for phi, wedge, energy or inverse beam that you choose. However, all files that will be produced will be displayed in the 'run sequence' window. Here are examples of some possible run sequences.

One Energy, Inverse Beam Off, Phi < Wedge Size (simplest case) example: test__1__001 test__1__002 test__1__003 (phi: 0-3 deg, wedge: 5 deg, 1 energy, inverse beam off)	Two or more Energies, Inverse Beam Off, Phi < Wedge Size example: test__1__E1__001 test__1__E1__002 test__1__E1__003 test__1__E2__001 test__1__E2__002

	test__1__E2__003 (phi: 0-3 deg, wedge: 5 deg, 2 energies, inverse beam off)
Two or more Energies, Inverse Beam On, Phi < Wedge Size example: test__1__E1__001 test__1__E1__002 test__1__E1__003 test__1__E1__181 test__1__E1__182 test__1__E1__183 test__1__E2__001 test__1__E2__002 test__1__E2__003 test__1__E2__181 test__1__E2__182 test__1__E2__183 (phi: 0-3 deg, wedge: 5 deg, 2 energies, inverse beam on)	Two or more Energies, Inverse Beam On, Phi > Wedge Size example: test__1__E1__001 test__1__E1__002 test__1__E1__181 test__1__E1__182 test__1__E2__001 test__1__E2__002 test__1__E2__181 test__1__E2__182 test__1__E1__003 test__1__E1__183 test__1__E2__003 test__1__E2__183 (phi: 0 - 3 deg, wedge: 2 deg, 2 energies, inverse beam on)

Useful Tips

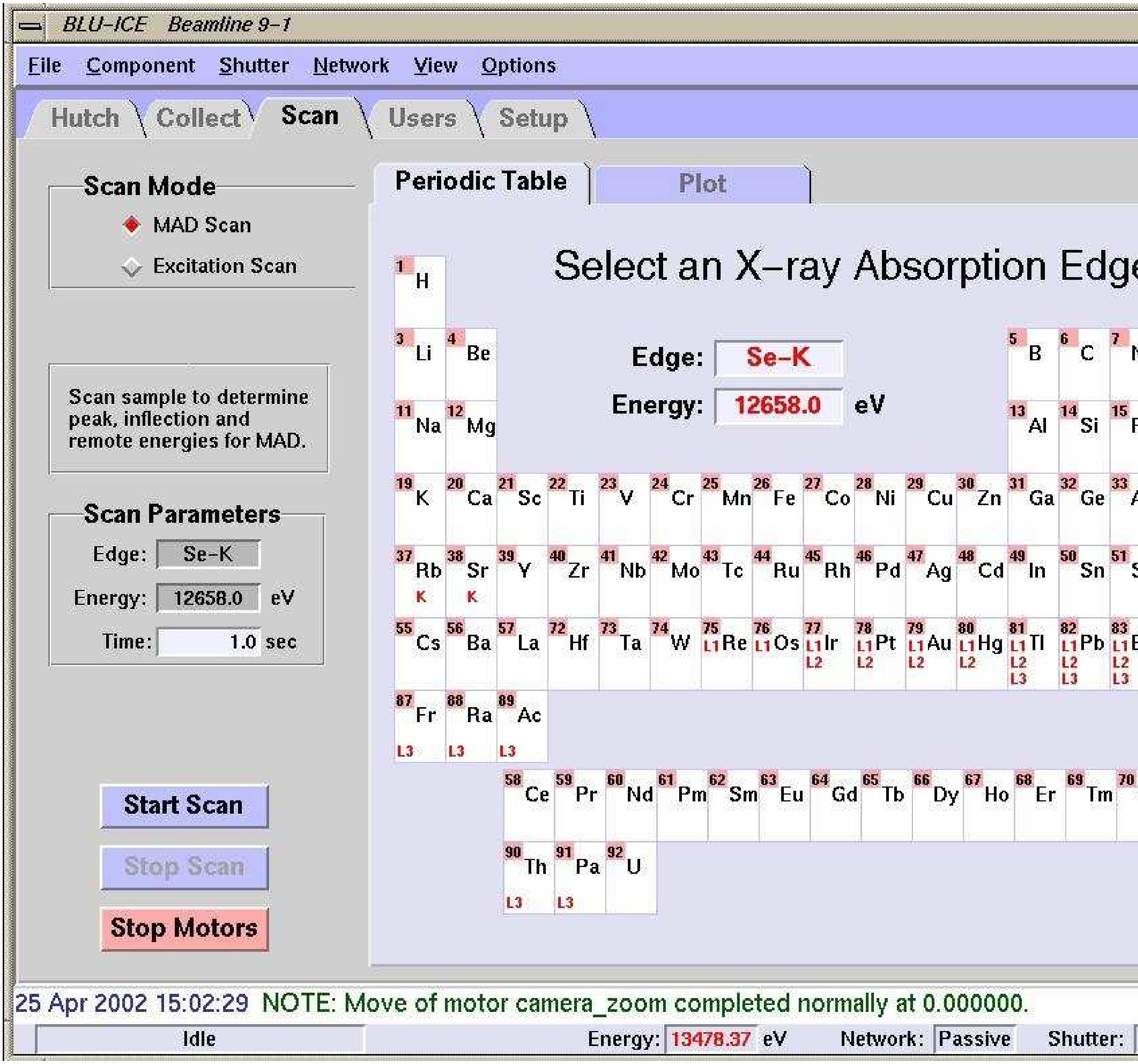
- You can jump to a different image by double clicking on the desired image name. You will notice that the color of the selected image turns to red.
- Both ***Pause*** and ***Abort*** stops data collection after completion of the current image.

Content questions and comments: Ward Smith.
Technical questions and comments: Webmaster.

Scan Tab

- MAD Scan
    - Selecting an X-ray Absorption Edge
    - Analyzing the Fluorescence Scan
- Excitation Scan
- Main Index

The ***Scan Tab*** is used for energy scans.



Scan sample to determine peak, inflection and remote energies for MAD.

Edge: Se-K

Energy: 12658.0 eV

Edge: Se-K

Energy: 12658.0 eV

Time: 1.0 sec

25 Apr 2002 15:02:29 NOTE: Move of motor camera_zoom completed normally at 0.000000.

Energy: 13478.37 eV

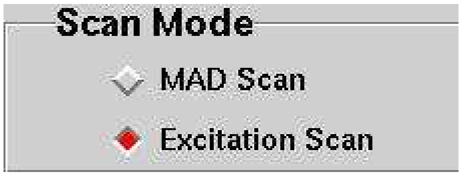
Network: Passive

Shutter:

The two available scan modes are ***Excitation Scan*** and ***MAD Scan***.

Excitation Scan

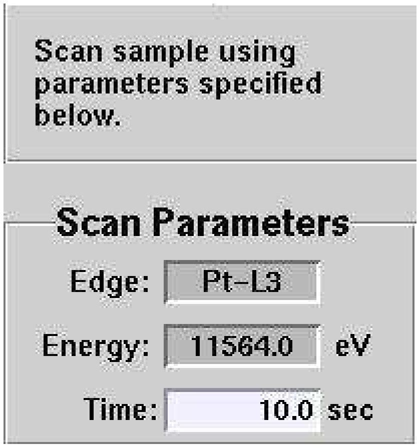
- Excitation scans are required to set up the Fluorescence detector and can be very useful for the identification of heavy elements in a crystal. They take less time than the MAD scans and are a faster way to determine the presence of a heavy atom derivative/ligand in the sample.
- Move to an energy above the absorption edge of the element on interest using ***Energy*** box in the hutch tab and click the ***Start Scan*** button. While MAD scan measures the fluorescence counts from a single element by changing the energy, the excitation scan measures the fluorescence from different elements without changing the energy. All the heavy elements with absorption edges below the incident energy will show peaks in the spectrum at their characteristic emission energies.



Scan Mode

○ MAD Scan

● Excitation Scan



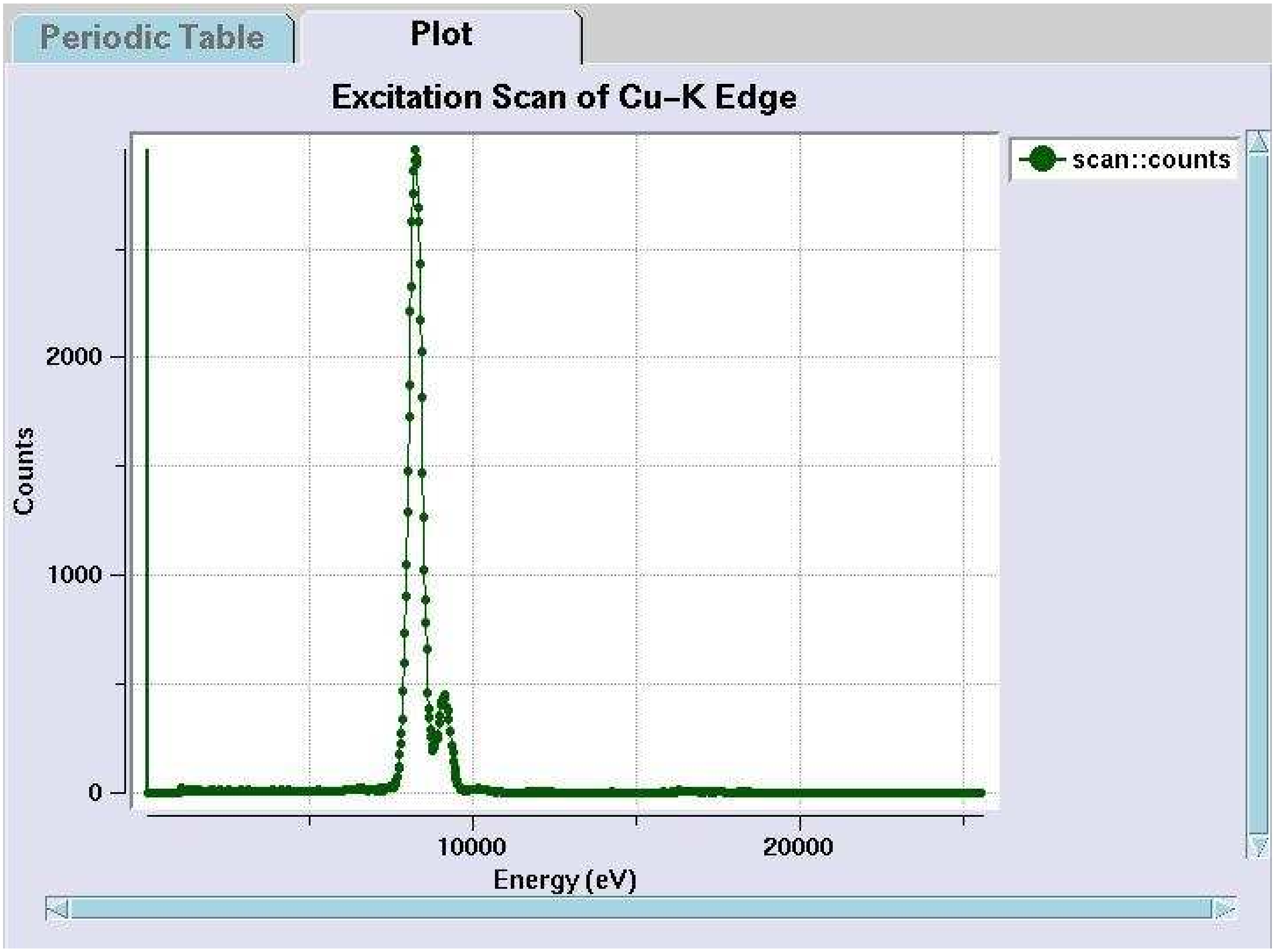
Scan sample using parameters specified below.

Scan Parameters

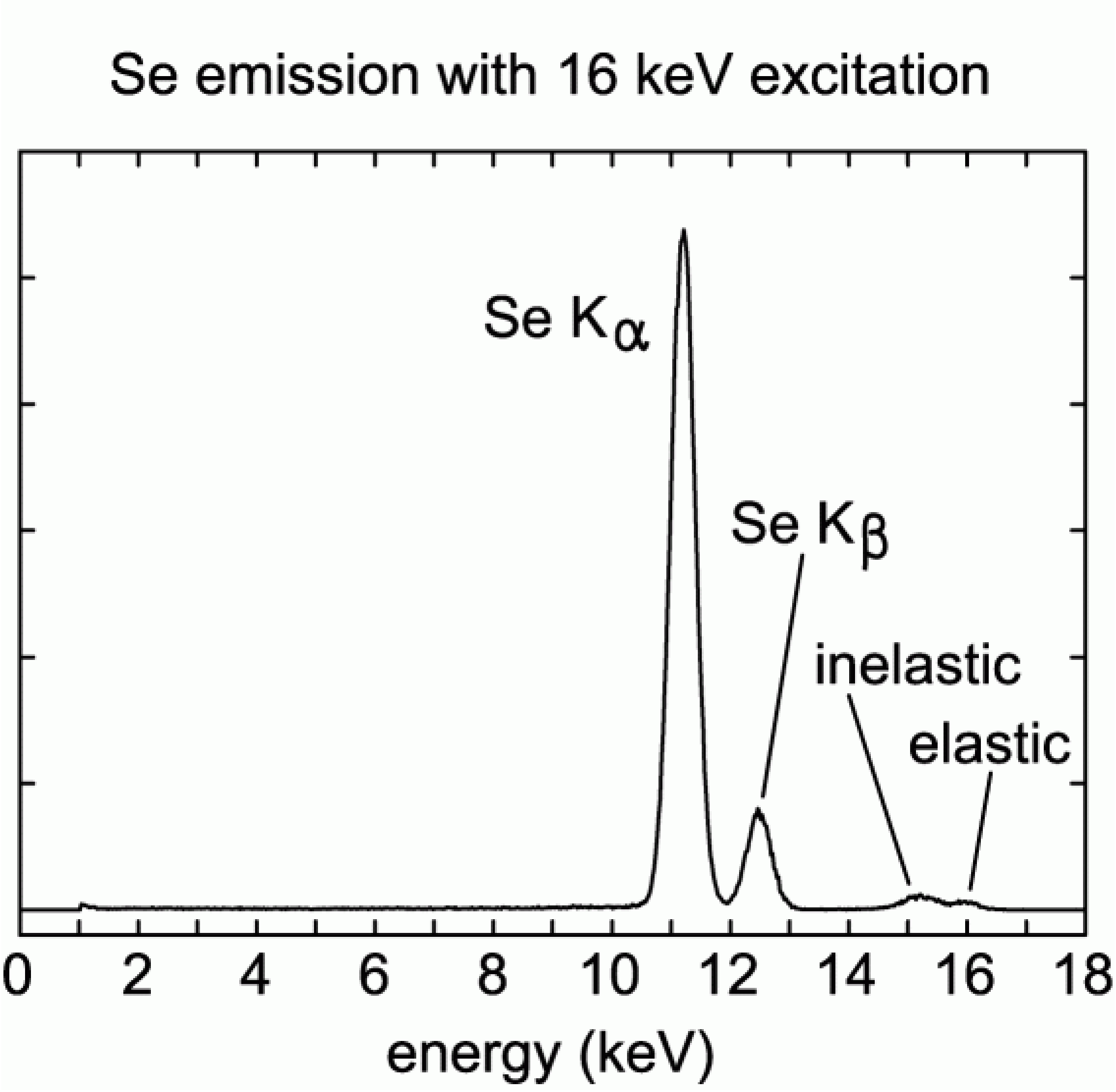
Edge: Pt-L3

Energy: 11564.0 eV

Time: 10.0 sec



Sample plot of Copper excitation scan

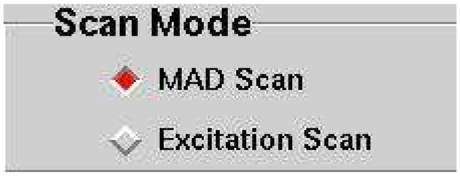


Sample plot of Se excitation scan describing various peaks

MAD Scan

- This mode performs the scan of a sample to determine the peak, inflection and remote energies for MAD data collection. The desired absorption edge can be selected from the ***Periodic Table*** tab. Select an edge by clicking on a particular edge in red (K, L1, L2, L3) and press the ***Start Scan***. Click on the ***Plot*** tab next to the ***Periodic Table*** tab to display the plot of the spectra.

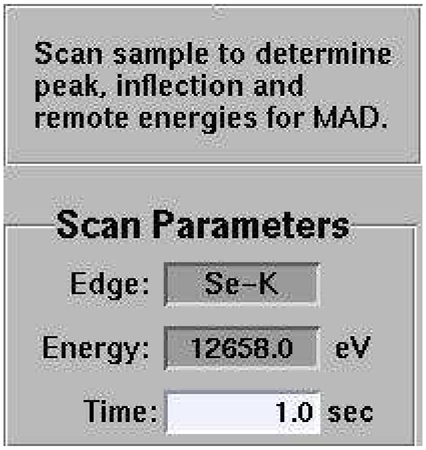
- If the initial scan looks noisy (large oscillations in the fluorescene counts from points to points), the scan time can be increased to improve the signal.



Scan Mode

● MAD Scan

○ Excitation Scan



Scan sample to determine peak, inflection and remote energies for MAD.

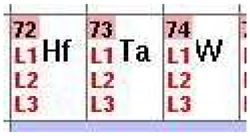
Scan Parameters

Edge: Se-K

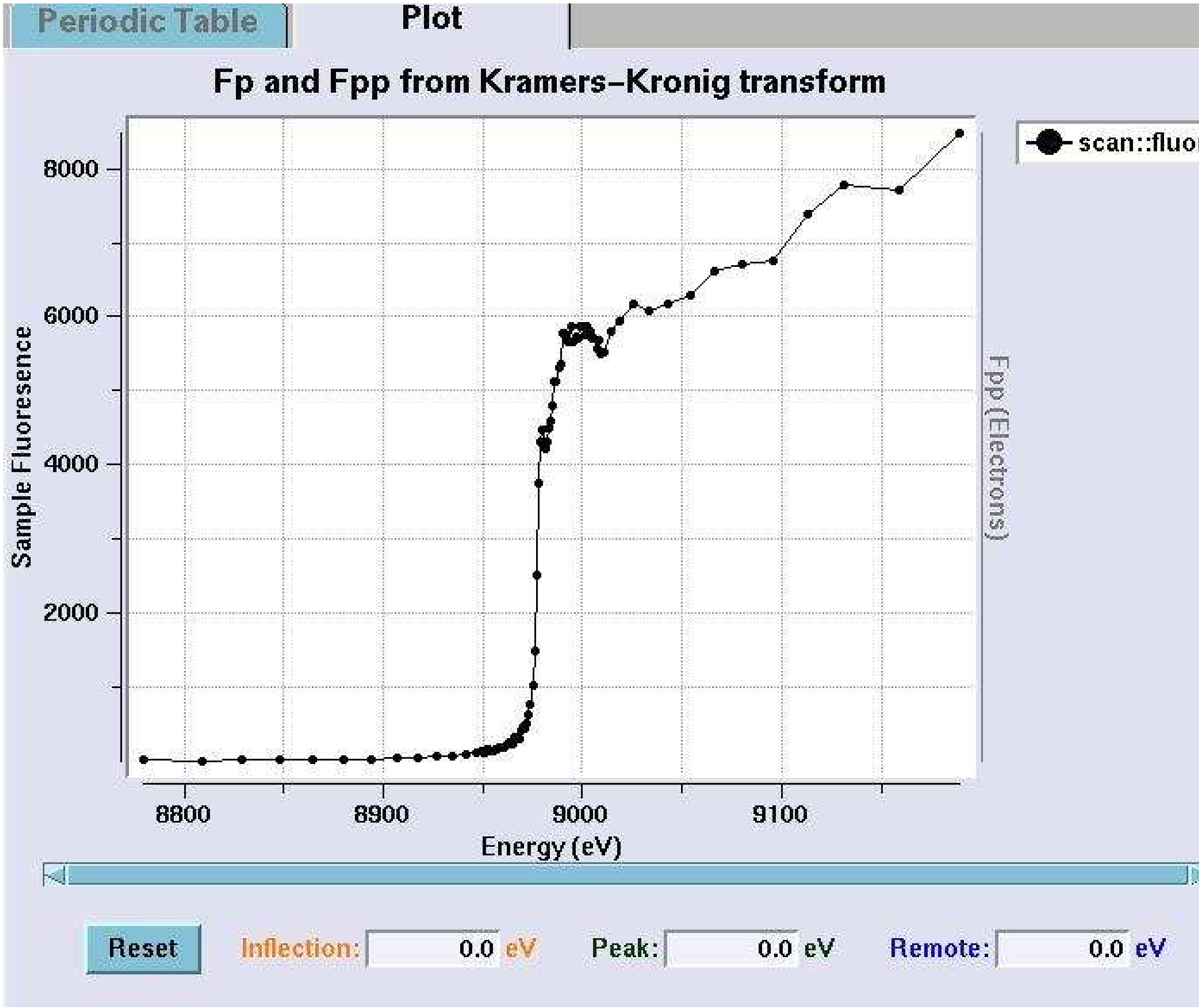
Energy: 12658.0 eV

Time: 1.0 sec

Selecting an X-ray Absorption Edge



- For some elements, such as those shown to the left, you can select different edges-- ***L1, L2, L3***. As a rule of thumb always select ***L3*** for heavy elements (exceptions are Sm or Eu). Consult the support staff for help with selecting the edge. Additional information regarding MAD data collection is available at the MAD data collection at SSRL site

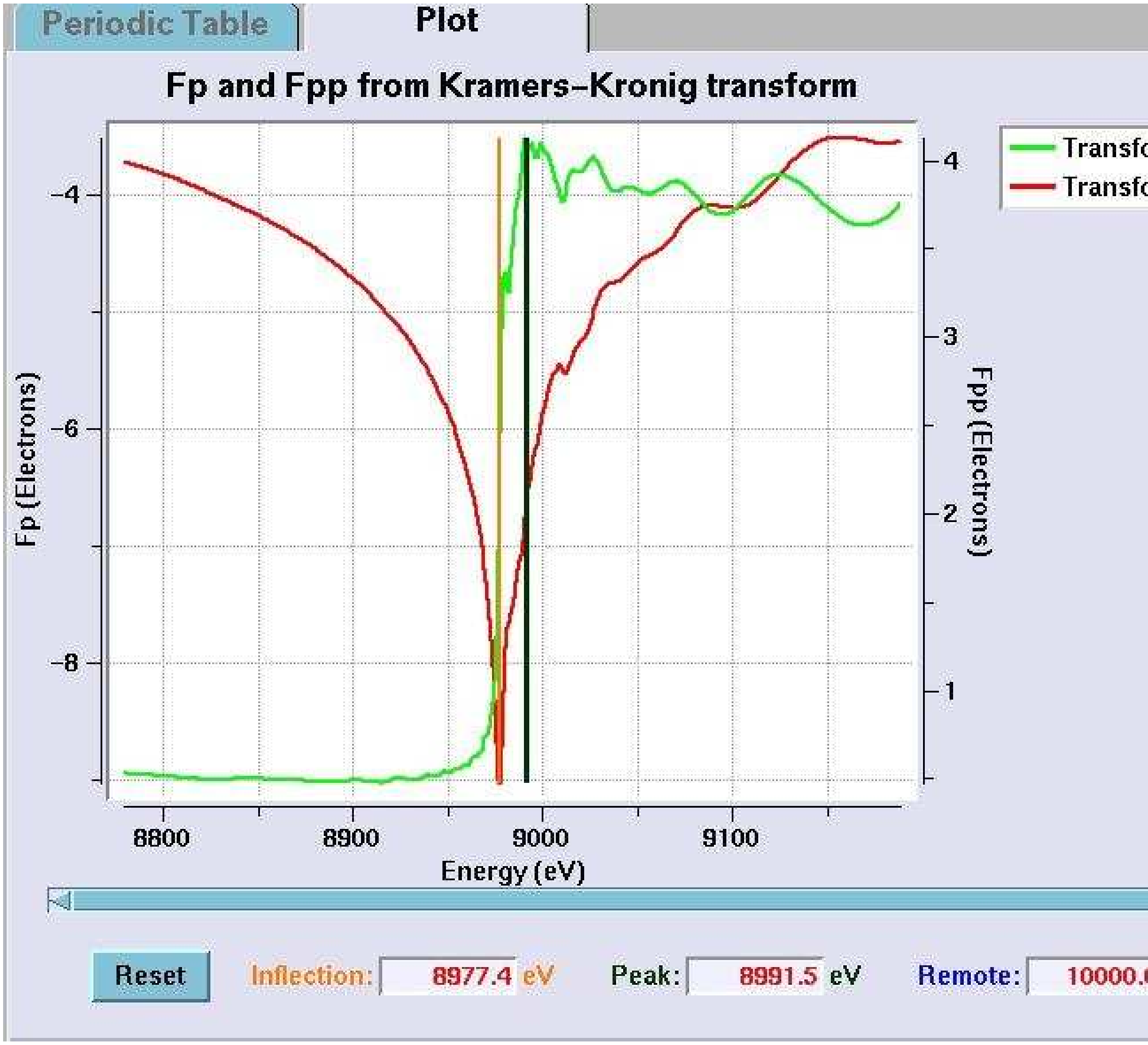


Inflection: 0.0 eV Peak: 0.0 eV Remote: 0.0 eV

Sample plot of Copper edge scan

Analyzing the Fluorescence Scan

- Chooch, a program for calculating Anomalous Scattering Factors from X-ray fluorescence data, runs automatically and displays the peak, inflection and selected remote values.



Inflection: 8977.4 eV Peak: 8991.5 eV Remote: 10000.

- The values can be adjusted by moving the cursors.
- Pressing the ***Update*** button in the ***Collect Tab*** imports the above selected values into the ***Collect Tab*** for MAD data collection.
- The units of Y-axis of the spectra can be changed by clicking the right mouse button on the left side of the spectra. Clicking the right mouse button inside the graph offers additional features: "Zoom", "Save", "Print", etc.
- By clicking the right mouse button on the signal plot or the transform or any other lines in the spectra, users can change various parameters like line color, line thickness, symbol shape and color, etc.
- Placing the mouse over the plot line will display the x and y coordinates for each point. This is useful to find out the f' and f" values for the selected energies.
- To zoom in the plot, left click on a point of the plot window, hold down the mouse key and drag the mouse to define the zoom rectangle. The zoom level changes after releasing the mouse button.

Content questions and comments: Ward Smith.
Technical questions and comments: Webmaster.