

BEAMLINE OPERATIONS MANUAL FOR USERS

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Questions and/or suggestions on the manual: Ruslan Sanishvili (Nukri). Any comments are welcome on the hard copy as well.

Other questions and all complaints: your host

Last updated on 6/4/2011

Introduction

Most of the user operations required for diffraction experiments will be carried out with JBluIce. These include

- Robotic sample mounting
- Manual or automated sample centering
- Interactive or automated sample finding and centering with diffraction
- Interactive or automated sample finding and centering with X-ray fluorescence
- Energy change
- Beam flux attenuation
- Beam size adjustment
- Omega axis rotation
- Detector distance and 2θ adjustment
- Changing the Beamstop-to-sample distance
- All data collection parameters
- “Standard” or “vector” data collection
- Automated strategy evaluation for data collection
- Interactive or automated measuring of fluorescence spectrum.
- Interactive or automated recording of absorption edge scans
- CHOOCH calculations to plot f' and f''

Training on some of these features can be found at

<http://www.gmca.anl.gov/video/index.html>

Your host will also provide the training on these and other features.

This manual covers rest of the operations which are not part of JBluIce

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New features

(This is only a list of new features. Your host will explain each feature in detail)

New features in data collection and beamline controls As of June 4, 2011

Raster tab:

- Auto-raster
 - User enters rastering parameters and initial search area
 - JBluIce runs multiple grid searches to find promising sites in two orthogonal planes
 - Sites are individually centered for data collection and crystal size reported
- "Position Sync" is now replaced by "Save Position"
 - Before a raster run starts, the sample position can be saved
 - Once a rastering run starts, user saved positions are cleared

Collect tab:

- Collect tab layout is slightly changed
 - "Single" mode disables vector collection for a given run
 - "Vector" mode enables setting the vector position and then the parameters
 - "Vector" options are moved to a separate sub-tab
- Collect tab field interaction is improved
 - When setting up vector collection, only the "step size" and "number of sites" fields will significantly change the number and spacing of the sites.
 - It's recommended that "step size" and "number of sites" are set first, before any other parameters.
- Strategy
 - In run 0, the user checks "strategy" and enters a second frame angle
 - Clicking "collect" takes two frames and processes them to generate a data collection strategy
 - The results are viewed on the strategy tab
 - Clicking "export" will create a new run tab with the generated strategy
- The collect run 0 start angle field has a dropdown for +/-90 degrees

Screening tab:

- Separate pop-up window with the name and status of the mounted sample

Log tab:

- Beam X,Y and Vector parameters are saved in the log file.
- Log files are now written for images collected from the Screening tab

Other:

- Red Hat EL6 support
- XDS input file is written for a data set containing more than 5 images. User can type xds or xds_par in the sub folder for processing. For now, unit cell parameters should be changed manually.

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New features in data collection and beamline controls

As of 02/06/11

Sample tab:

- High res camera tab has color-coded display of multiple beam sizes
- The size of the displayed full beam can be manipulated by hosts
- Guard Slit size display added to the sample tab

Raster tab:

- Polygon selection in raster tab, including multiple polygons
- Old-style centered rectangle selection
- Display of the beam size which will be used for rastering
- Manual override of automatically selected cell and beam sizes
- If the sample was mounted by the robot, default file, related to the current sample, is put in raster tab
- Color-coded display of the results
- Fluorescence rastering can be done in shuttle mode
- Raster results now list absolute motor positions

Collect tab:

- Selection of the number of sites for data collection
- Sites are now numbered in the vector collection

Screening tab:

- Rinse/Wash buttons added
- Prefix override
- Crystal ID-based default file name is put in the raster tab

Log tab:

- I0 and active beamstop intensity values are plotted as function of the frame number

Other:

- If autocentering fails, it gives a pop-up warning
- adxv can update images during data collection

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New features in data collection and beamline controls As of 10/10/10

General:

- Blu-Ice is now JbluIce
- Error messages are improved when requesting invalid parameters

Hutch tab:

- Resolution predictor takes into account 2θ and displays all resolution circles

Sample tab:

- Wash and rinse added in “Sample Tools”.
- Annealing by CryoJet flow control and by moving the sample are together
- Motions are disabled during diffraction image measurement

Raster tab:

- Rastering cell sizes are determined semi-automatically when the grid size is entered
- Beam size is set automatically depending on the cell size
- “Position sync” is added
- Results are being written in a separate spreadsheet
- Fluorescence rastering results are displayed same way as diffraction rastering

Collect tab:

- Resolution predictor displays resolution rings correctly when 2θ is not 0
- Vector data collection feature is user-selectable

Screening tab:

- Spreadsheets with xlsx format are accepted
- “Update” button is added
- “Autoindex-Strategy” button added
- Right click on the spreadsheet window brings up pull-down menu
- Data collection parameters are independent from the Collect tab
- Mouse-over displays brief explanations
- Motions are disabled during diffraction image measurement

Scan tab:

- Center of the plot can be defined manually

Robot dewar:

- Improved dewars

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Definition of directions

Directions along the beam are dubbed “upstream” (negative) and “downstream” (positive)

Directions perpendicular to the beam, in the horizontal plane, are called “inboard” (negative) and “outboard” (positive). Reference for these directions is stored ring. Vertical directions are “up” and “down”

Using MEDM screens

Motors that cannot be moved by BluIce (for example for beamline alignment), will be moved by Motiff Editor Display Manager (MEDM) screens (Fig. 1).

Instructions below are common for all MEDM screens

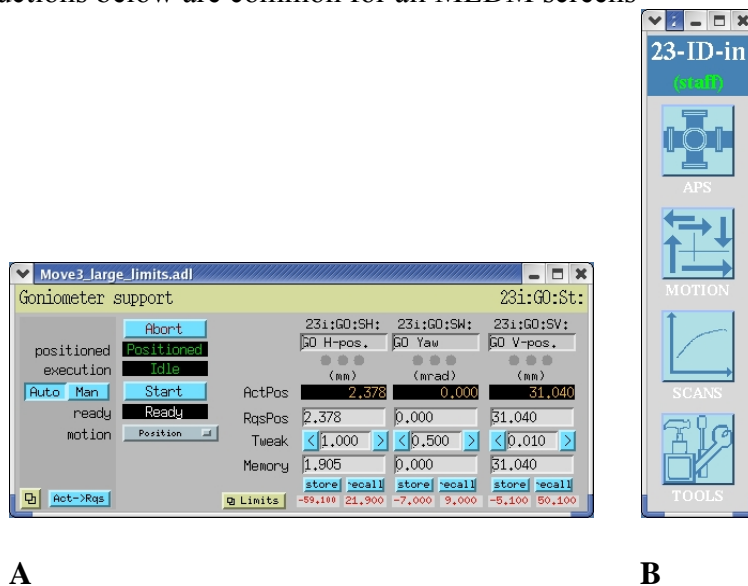


Figure 1 A) Example of MEDM screen. B) Beamline operations toolbar. It can be open by typing in a terminal: 23i on ID-in line, 23o on ID-out and 23b on 23BM.

Actual position of a motor is displayed in a row marked **ActPos** (yellow numbers)

Target value, or requested position is given in the row marked **ReqPos**

MEDM screen can be in manual mode (man) or in automatic (Auto)

To change the number in any MEDM field, you have to type it in and hit ENTER before leaving the field.

To change the motor position you can

- Type the desired value, hit ENTER and then click start if the screen is in manual mode. If it is in auto mode, the motor will drive as soon as you hit ENTER

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- b) Increment or decrement the requested value by clicking on > or < respectively. If the screen is in manual mode, click start after each increment or decrement. If it is in auto mode, motor will drive as soon as you click > or <. You can change the step size itself by editing the number in the field marked Tweak
- c) Restore previously saved value. Some times it is useful to save the position of the motor. For example, position of the goniometer z axis after you've adjusted it to the length of your pins. To save the value, drive the motor to required position and click on store. You can restore this value by clicking on recall.

Energy change

Important: To avoid exposure of the sample (if mounted), first move it up by 200-300 microns.

Currently achievable energy range on IDD and IDB beamlines is 6 to 20 keV. The monochromator energy change and subsequent tuning and alignment are automated. Simply make sure the intensity monitor (Fig. 2) is running before the energy is changed. Once energy change is initiated, wait till the intensity monitor completes tuning (Fig. 2) before resuming other activities.

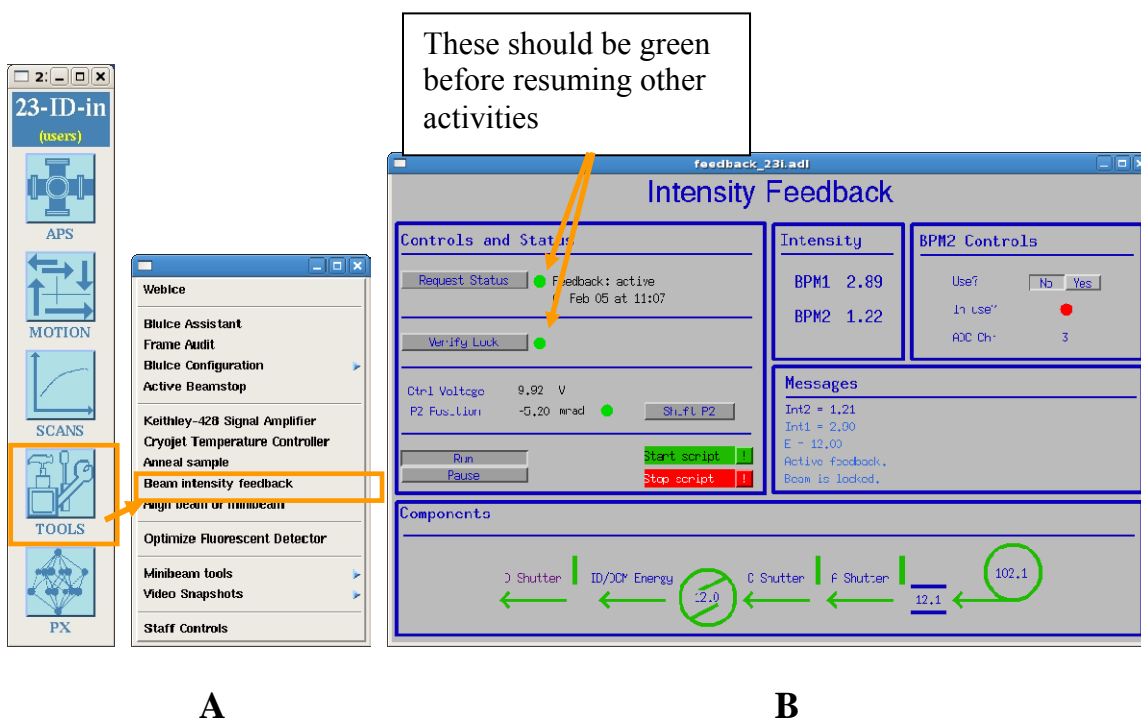


Figure 2 Beam positional and intensity monitor. A) Path to the controls. B) Controls themselves

If energy change is large enough (about 1 keV), beam alignment will be triggered automatically.

If energy change is not large, wait till the intensity feedback is ready (Fig. 2) and then align the beam by clicking "Optimize Beam" on the "Hutch" tab of JBlu-Ice.

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Aligning the beam

Important: To avoid exposure of the sample (if mounted), move it up by 200-300 microns before starting the beam alignment.

Auto-alignment: If the beam position is suspected to have changed (e.g. after energy change, slow drift over long time, fluctuations in the stored ring etc.), make sure the intensity feedback is running (Fig. 2) and align the beam by clicking the “Optimize Beam” in the “Hutch” tab of JBlu-Ice.

Manual alignment: If beam visualization and manual alignment are desired, follow the steps below.

WITH YAG CRYSTAL (SAMPLE NOT MOUNTED)

1. Take the collimator out with “Sample” tab in JBlu-Ice (Fig 3).
2. Move Omega to 0° .

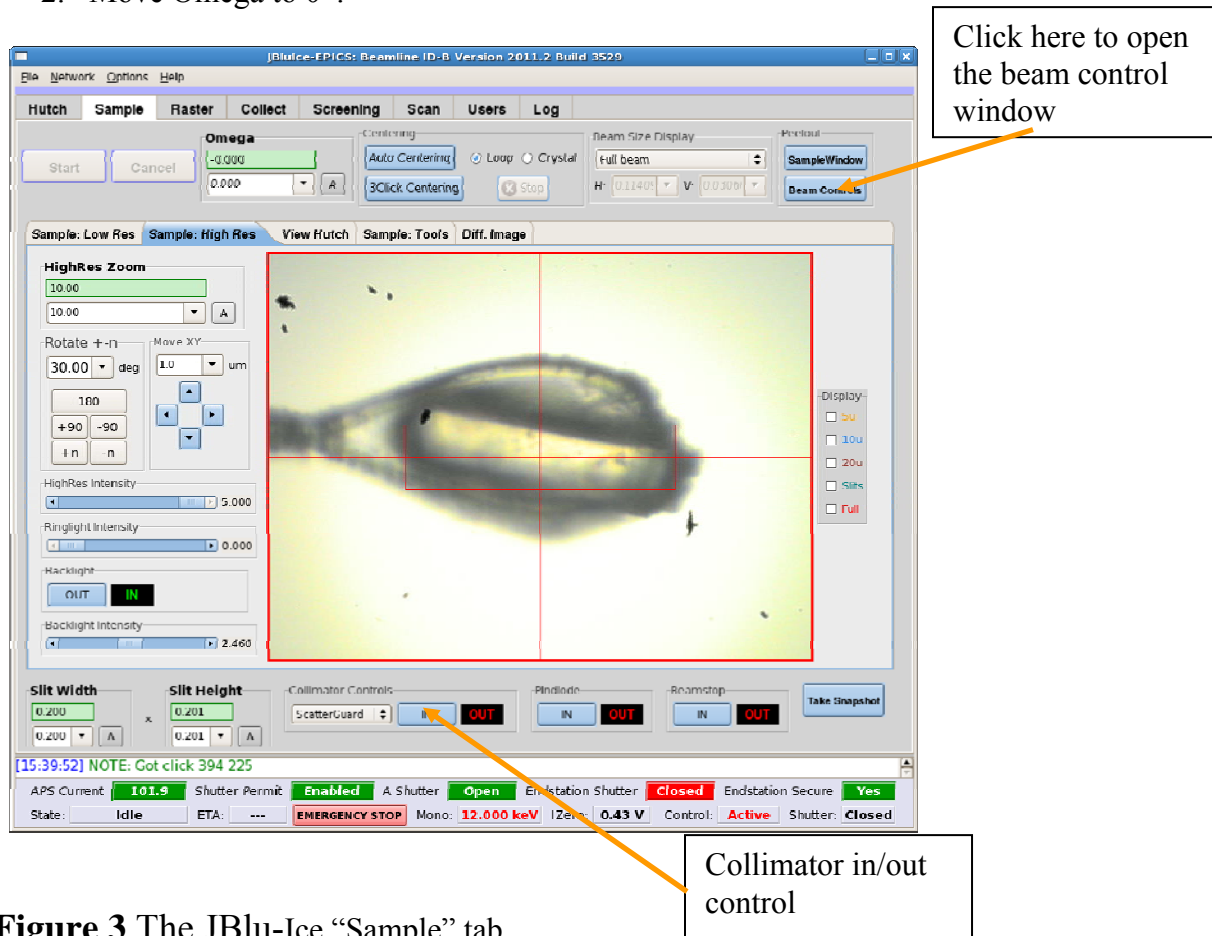


Figure 3 The JBlu-Ice “Sample” tab.

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3. Mount the YAG crystal on the XY stage (YAG plane should be close to vertical) (Fig. 4). Do not move omega after YAG has been mounted.
4. To ensure the YAG crystal is parallel to the camera lens: reduce the zoom value of the low resolution camera to 4.0. Rotate the YAG on the goniometer head manually until “edge on” view is achieved on the low resolution camera.
5. Interlock the hutch and open the “Endstation Shutter at the bottom of JBlu-Ice.
6. Turn off the lights for both cameras and the Ring light from the “Sample” tab of JBlu-Ice.
7. Attenuate the beam about 20 times on IDD station or 10 times on IDB station.
8. Go to the Sample tab of BluIce and select High Res Camera.

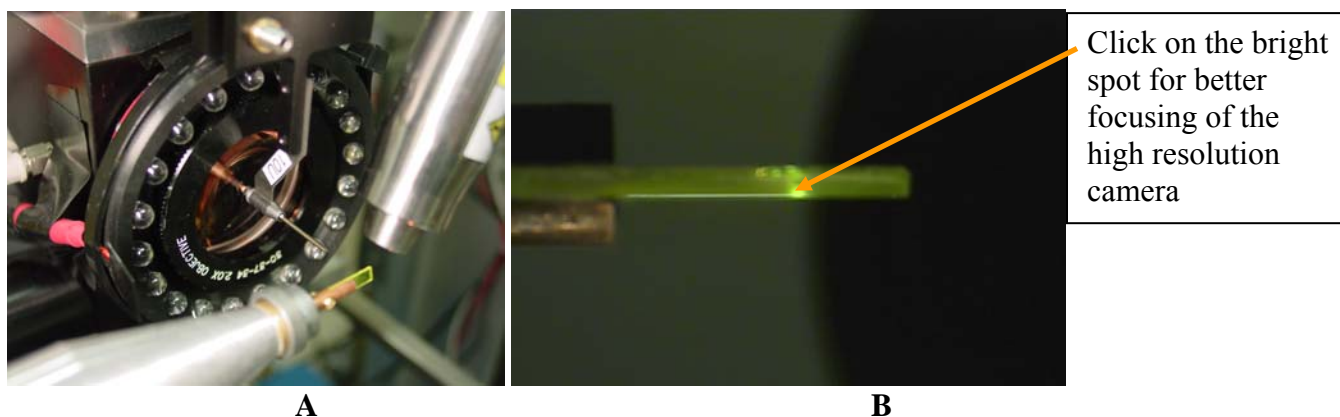


Figure 4. A) YAG crystal, mounted on the goniometer in proper orientation (vertical). B) View from the low resolution camera. To ensure vertical orientation of the YAG, this view must be “edge on”

9. Open Guard Slits to 2 mm × 2 mm (Fig. 5)
10. Open the timing shutter (Fig. 5). You should see the beam, perhaps not centered, on the YAG crystal (Fig. 6). If the beam cannot be seen,
 - A) Check the zoom on high res camera. If the zoom value is low (< 4) and you don't see the beam, it may be centered but obscured by the hole in the lens which is centered at the crosshairs (Fig. 7). Increase the zoom until you see the beam.
 - B) If the zoom value is high (>8) and no beam can be seen on YAG, the beam may be more than 200 microns off the center (less probable case). Reduce the zoom gradually until you see the beam.
11. Beam position can be adjusted by sliding bars in a separate window which can be launched by clicking on “Beam Controls” in the JBlu-Ice “Sample” tab (Fig. 3). **Important!** Sliding bars are very sensitive, so move them carefully. For finer adjustments, click in the fields on either side of the slider.
12. Repeat centering at the high zoom value (14) of the High Resolution camera and make sure the camera is focused on the YAG (next step)
13. To focus the camera, switch the Sample tab of JBlu-Ice to “Sample: Low Res”

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14. Click on the bright spot on the YAG in the JBlu-Ice window (Fig. 4B). It should make the beam image on the monitor sharper. For fine tuning of focus, you may click on the vertical red line just above or below the cross hairs in the “Low Res” view of the JBlu-Ice sample tab.
15. Close the beam width and height back to your working values. Beam should remain visible on the YAG crystal. If not, notify your host that guard slits may need centering.

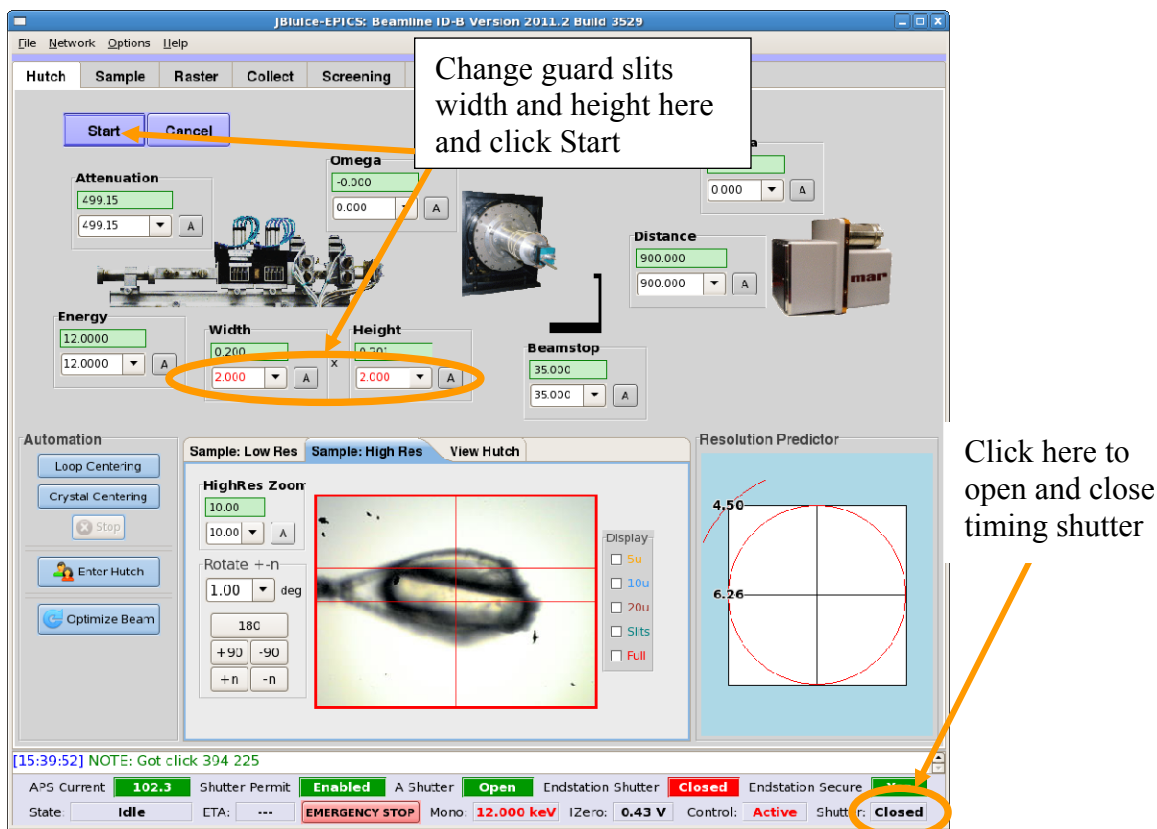


Figure 5 BluIce interface.

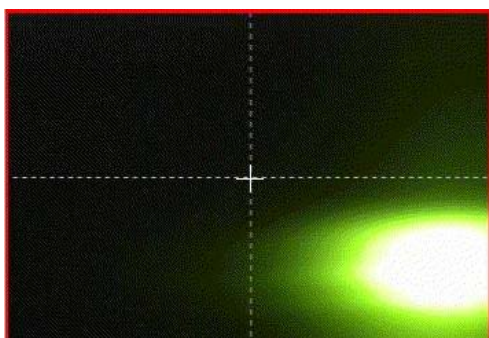


Figure 6 Beam, not centered, as seen on the YAG crystal.

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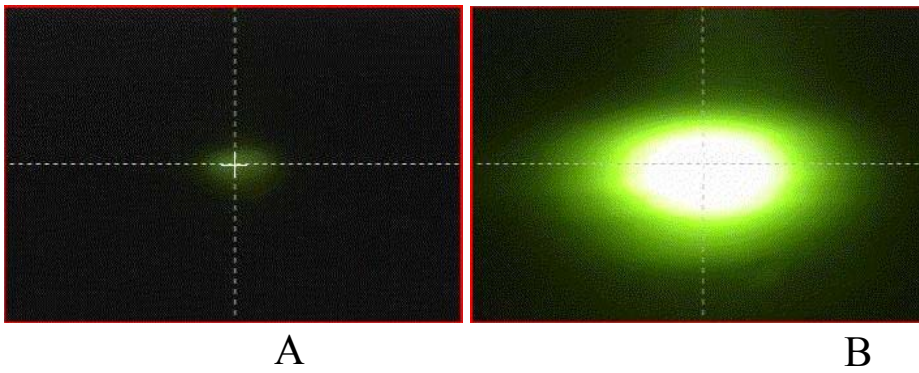


Figure 7. Centered beam on YAG with the zoom value of 2 (A) and zoom 14 (B)

Adjusting the height of the rotation axis

If the rotation axis is not centered on the crosshairs:

1. Mount the alignment pin¹ on XY stage. Alternatively, you can use the tip of your loop, if already mounted. This will be less accurate than using the alignment pin.
2. Looking at the High Resolution camera, center the pin in both X and Y planes the best you can. Remember, if rotation axis needs adjustment, center at this stage will not coincide with crosshairs.
3. Put omega at 0° and center the pin² (Fig. 8A). Note that the pin does not have to be in the center but it must be on the horizontal line.
4. Move omega to 180° . The pin will have moved out of the center (Fig. 8B). Half of the difference is where the rotation center is.
If the pin did not move out of the center, the rotation axis is already centered on the crosshairs.
If it did, continue
5. From the beamline operations toolbar, open *MOTION* -> *goniometer* -> *goniometer support* (Fig. 9A)
Use only vertical motion (Fig. 9B)
6. Move the goniometer support vertical up or down, as needed, to bring the rotation axis back to the center. (In other words, bring the pin half way towards the crosshairs).
7. Re-center the pin by point-and-click (or manual slides).
8. Repeat steps 3-7 at increased zoom values until satisfied.

Note 1: For alignment, you can use your loop instead of the pin, but it will be **less accurate**. For this, center the part of the loop, most extended to right (Fig 10).

Note 2: You can center the rotation axis using the $90^\circ/270^\circ$ pair of angles instead of $0^\circ/180^\circ$.

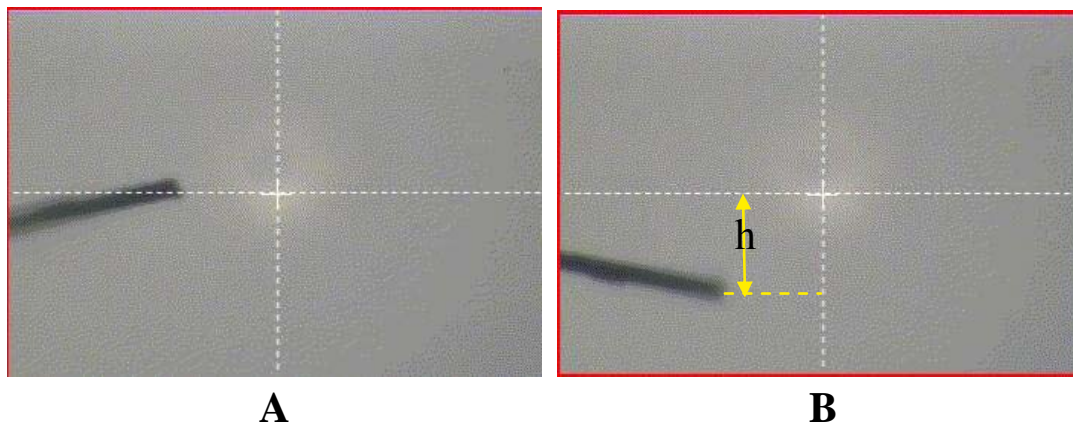


Figure 8. A) Pin “centered” at omega=0. B) Pin position at omega=180. Half of the difference h must be adjusted by the goniometer support vertical motion and another half by point-and-click. Pin is shown off center horizontally to emphasize that horizontal centering is not essential for the adjustment of the goniometer height.

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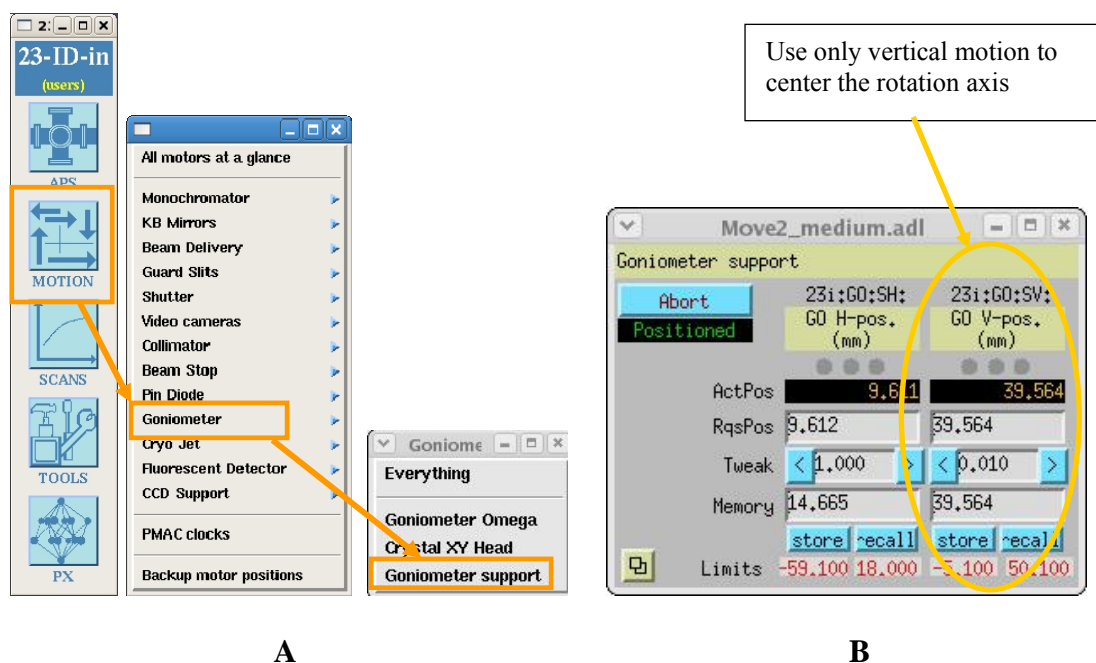


Figure 9. MEDM screen for adjusting the goniometer height (rotation axis)

- A)** path to the goniometer support motors
B) Goniometer support vertical motion

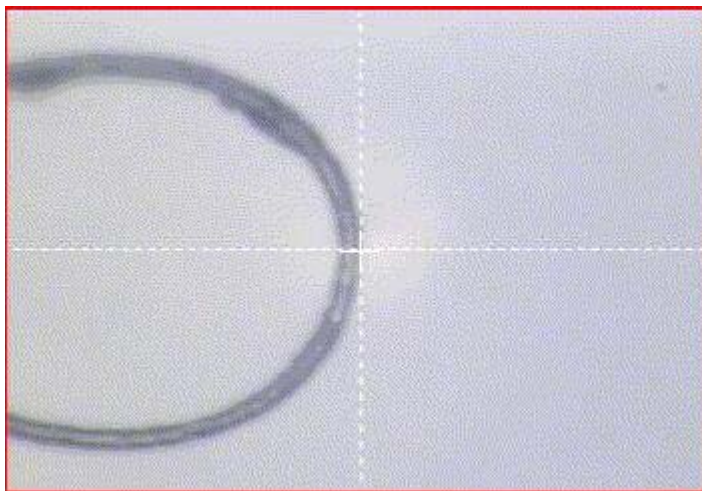


Figure 10 centering with a fiber loop.

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Aligning the cold stream

1. Insert the special alignment pin (Fig 11A) into the nozzle of the CryoJet (Fig. 11B) and tighten the screw. **Note:** make sure the screw is tight enough. Otherwise the alignment pin may fall out and crack the lens of the low resolution camera.
2. Reduce the zoom values on both cameras to midrange with BluIce
3. Using three slides on the CryoJet mount (Fig. 12), center the pin.
4. Increase the zoom on both cameras and repeat the step 3.
5. Take the pin out and put it back in the drawer labeled “sample environment”

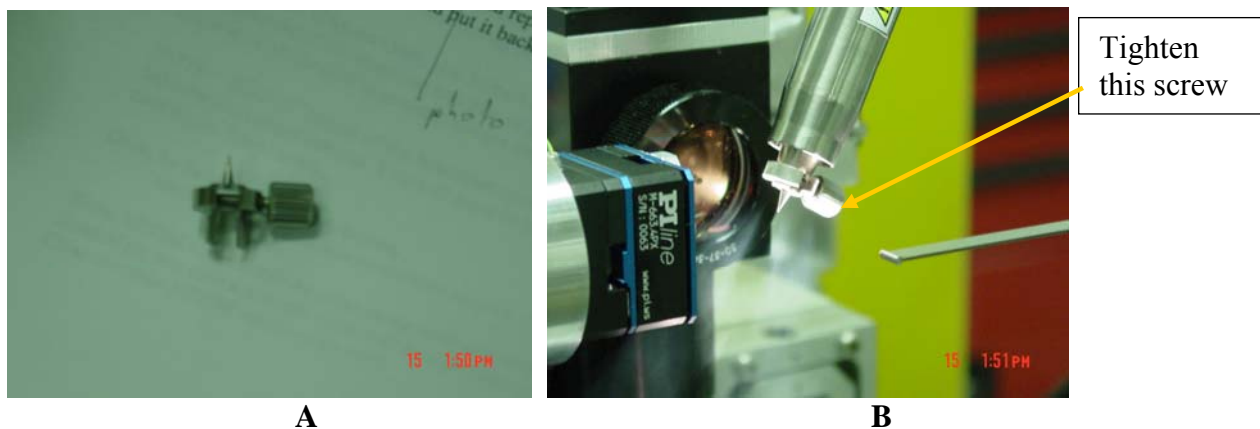


Figure 11. A) Special pin for alignment of CryoJet. B) alignment pin inserted into the cryo nozzle

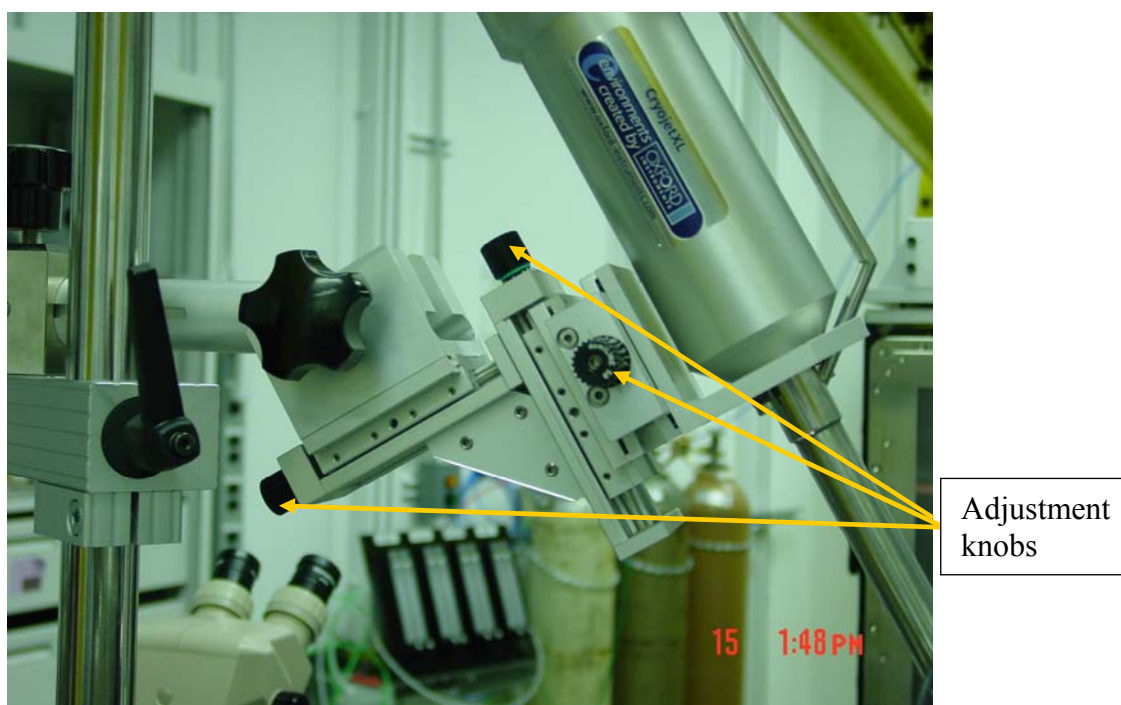


Figure 12. Alignment module of CryoJet. View from upstream.

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Measuring the flux

To measure the flux

1. Open the Keithley control GUI (Fig.13 A)
Tools → *Keithley Signal Amplifier*
2. Put the Keithley gain at 10^7 (Fig. 13 B)
3. Interlock the hutch
4. Close the Endstation (D on ID-D and B on ID-B lines) shutter from the “hutch” tab of JBlu-Ice. Record the reading on Io diode (Fig. 14). This is Offset
5. Open the Endstation (D or B) shutter and the timing (or fast) shutter in BluIce (Fig. 5, page 9) and record the reading on the Io diode. If diode reading is greater than 10, reduce the Keithley gain. If reading is less than 0.5, increase the gain.
6. Calculate the flux with equation:

$$\text{Flux} = (\text{Diode Voltage} - \text{Diode Offset}) \times 10^{6-G} / \text{Conversion Factor}$$

Where G is the gain on Keithley and Conversion Factor is obtained from the calibration plot on (Fig. 15).

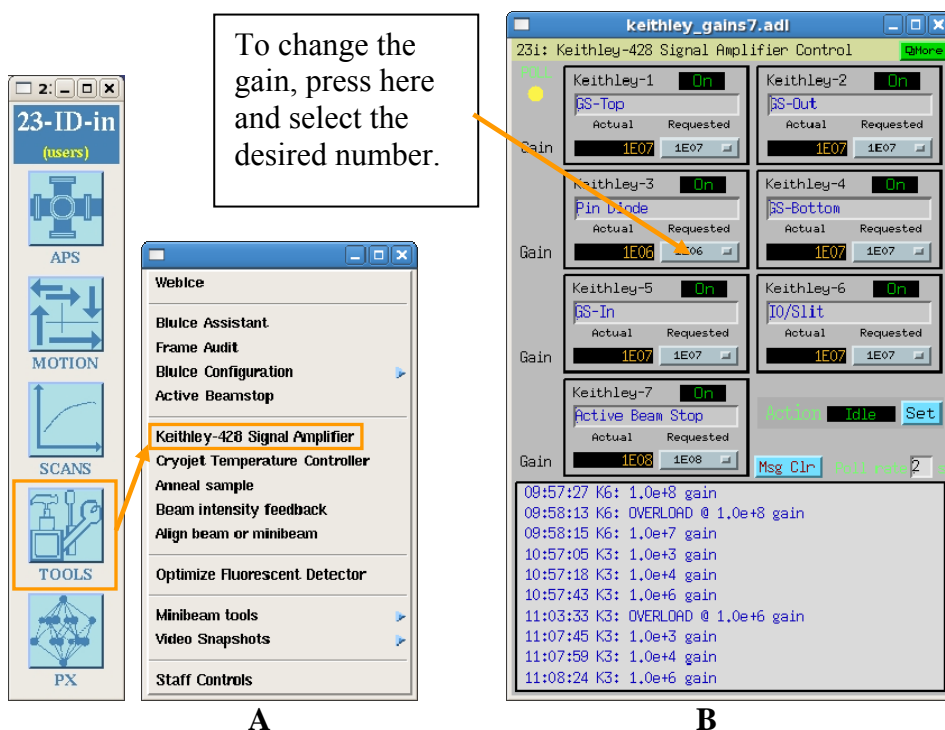


Figure 13. Remote control for Keithley gains. A) path to the control window. B) The control window itself

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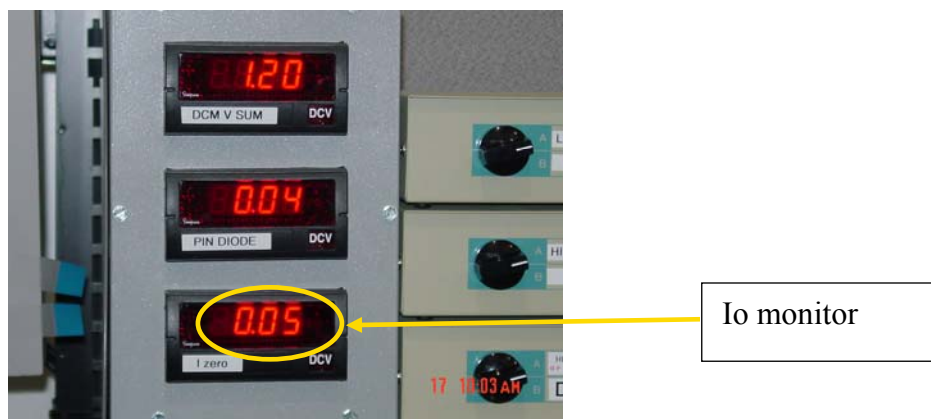


Figure 14. Electronics panel on the shelf above the beamline control computer

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Intensity calibration

I0 Calibration



Figure 15 Intensity calibrations for calculating the flux on 23-IDD line

TROUBLESHOOTING

Whatever the problem, DO NOT reboot the computers. It will likely cost two hours or more to restore all systems and communications.

!! After restarting JBlu-Ice, don't forget to become active!!

Data collection hangs

If JBlu-Ice stopped collecting data with red frame around its GUI and several MEDM screens popped up, just click “Collect” button and it should resume. If not, check if the Omega motor is responding. In the MEDM screen for omega motor (Fig. 16), first click on the field right from RqsPos and hit Enter. If omega did not move and data collection did not resume, click “Abort” and then “Start”. If this did not fix the problem either, it means Omega motor is not responding and it needs homing.

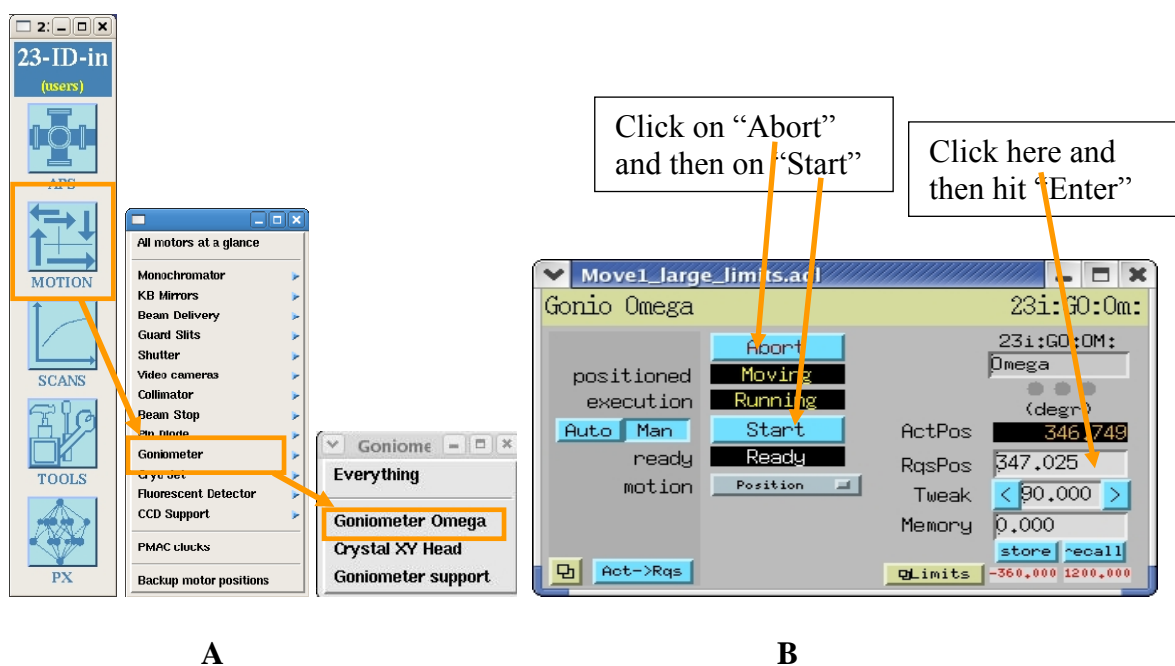


Figure 16 Omega motor MEDM screen

To home Omega motor, open
MOTION → *Goniometer* → *everything* (Figure 17)

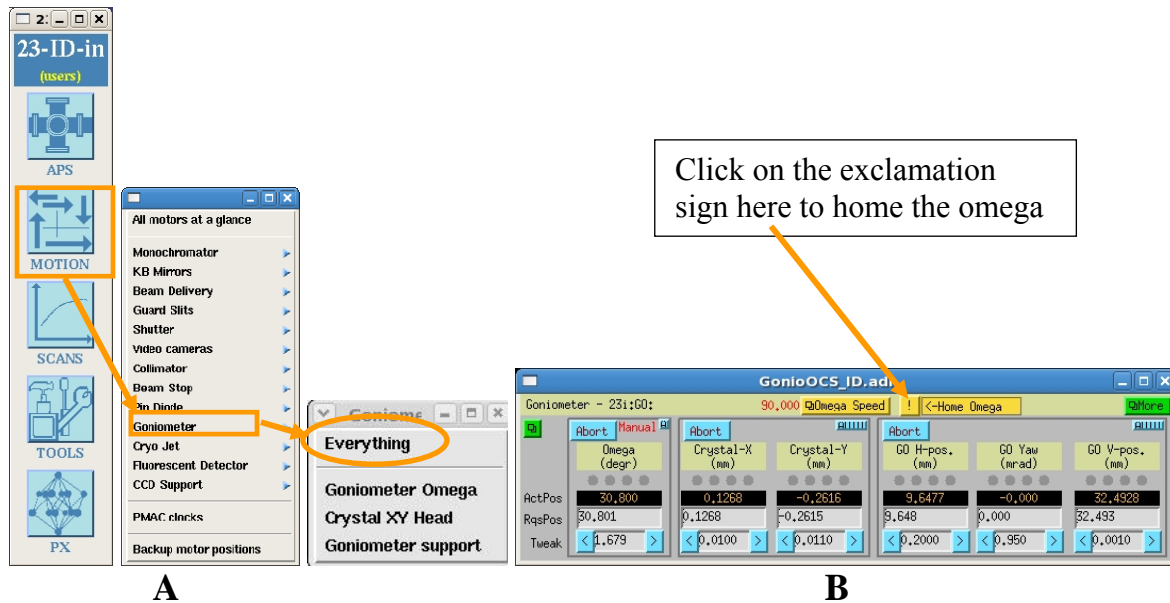


Figure 17. A) Path to home the omega motor. B) Window to home the omega motor

Few MEDM screens will be open along with one terminal window. When homing procedure it finished (in less then a minute) there will be a prompt in the terminal window to exit. At this point you may close the newly opened MEDM screens as well.
After homing Omega, You should restart both MAR and JBlu-Ice software.

!! After restarting JBlu-Ice, don't forget to become active!!

Blu-Ice stuck in “Collecting”

If after completion of data collection, or aborting it, JBlu-Ice is still “thinking” to be collecting data, open

TOOLS → BluIce Interface → Individual Runs → Run-# (Fig. 18A)

Where “#” corresponds to the tab number of the JBlu-Ice which is stuck in the “collecting” mode. In this example it is run/tab 1. In the resulting control window (Fig. 18 B), edit the first line to read “complete”.

Exit JBlu-Ice and restart it. It should solve the problem. **All your data collection parameters will be preserved.**

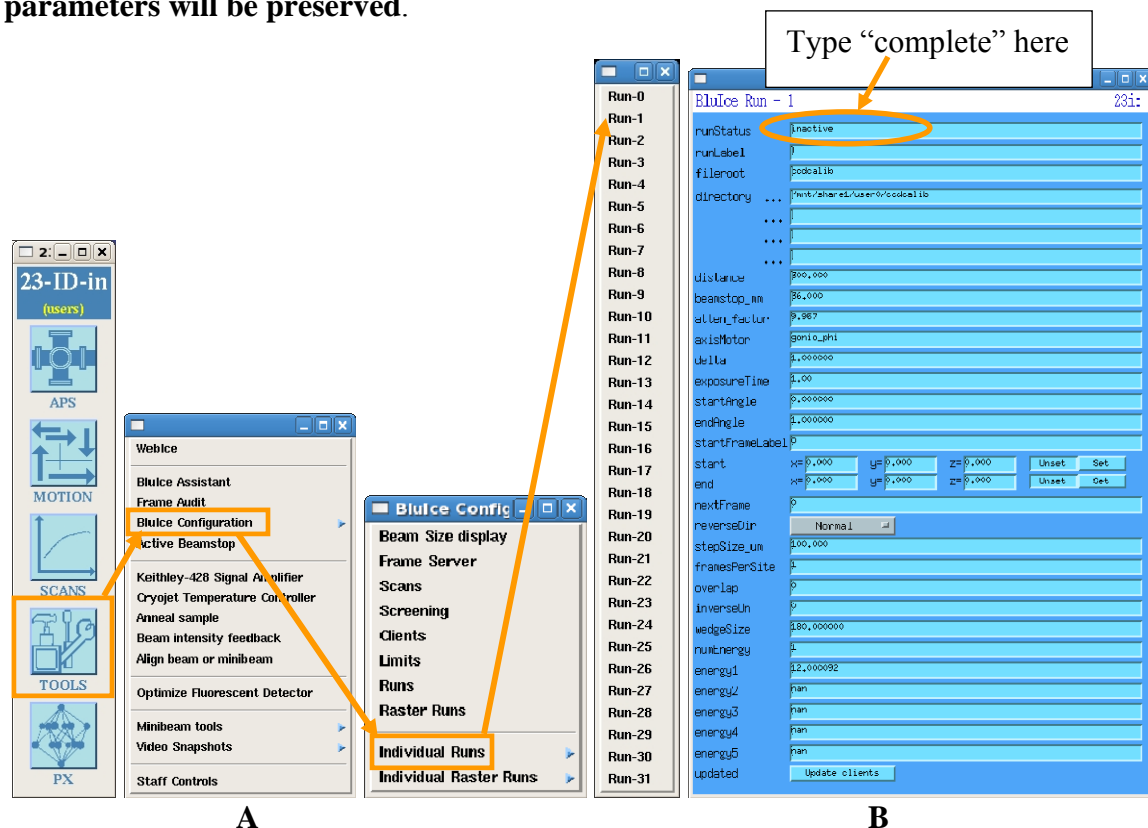


Figure 18 A) Path to open individual run controls of JBlu-Ice. B) individual run controls

If problem persists, open

TOOLS → JBlu-Ice interface → Runs (Fig. 19A)

And in resulting window (Fig. 19B) edit all fields to be 0.

Exit JBlu-Ice and restart it. This will delete all non-0 tabs you may have had open in JBlu-Ice and **you will need to set-up data collection parameters again.**

If JBlu-Ice still doesn't work, exit both JBlu-Ice and MARCCD programs and restart them in proper sequence (MARCCD first followed by JBlu-Ice).

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!! After restarting JBlu-Ice, don't forget to become active!!

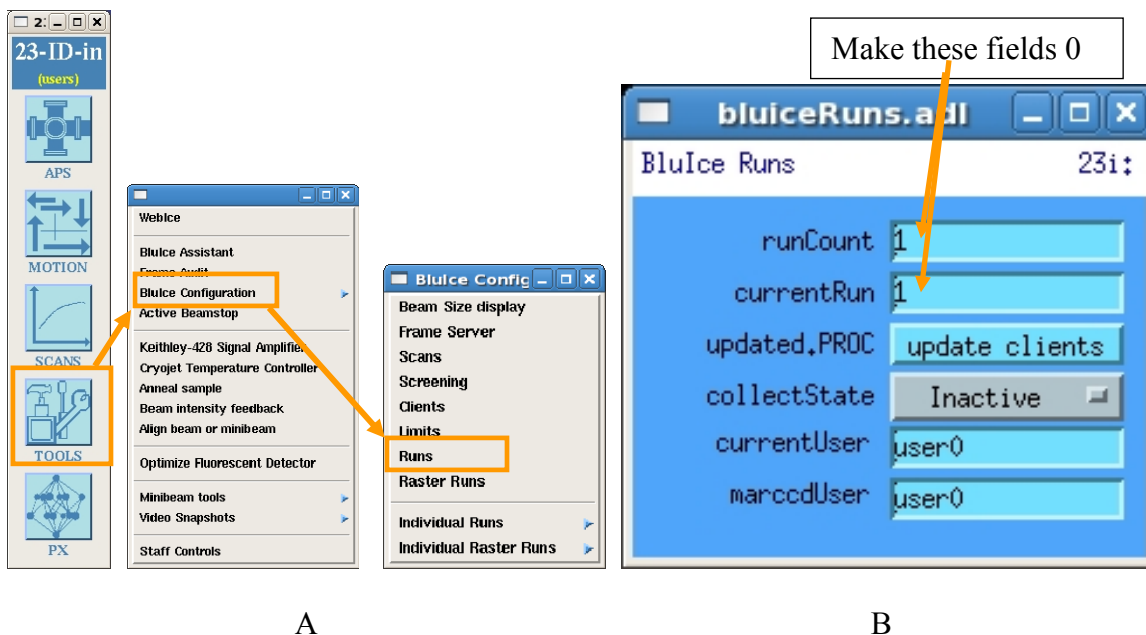
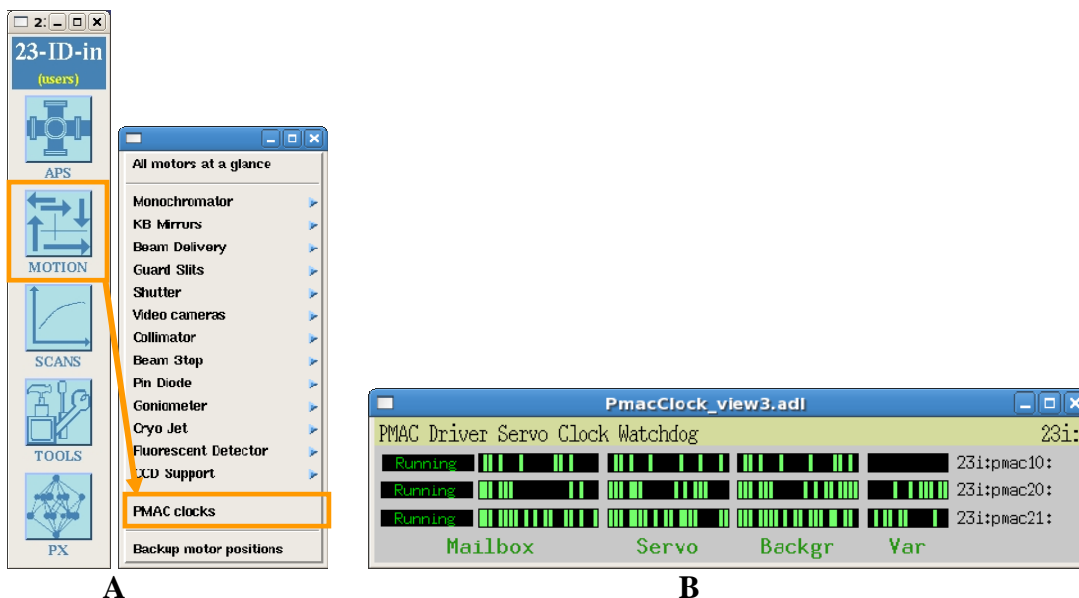


Figure 19 A) Path to the JBlu-Ice runs list. B)

Blu-Ice still won't work

If JBlu-Ice still cannot collect data after trying the above recommendations, check the status of servo clocks: Open *MOTION* → *PMAC clocks* (Fig. 20)



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Figure 20 A) path to opening the servo clock watchdog. B) Servo clock status window

Make sure that yellow bars in all three lines under the “Mailbox” are moving. If any of them is “frozen”, corresponding IOC needs to be rebooted. Contact your host.

NOTE: after rebooting IOC for PMAC20, following motors need to be put in “Auto” mode:

Collimator support
Goniometer Support
Goniometer Crystal XY

Collimator won't move

Check the “Collimator Vertical” field of “BluIce Assistant”. If “manual” is displayed in red, click on “auto” button next to it.

Point and Click is not responding

When Point and Click utility is ready, the video image in JBlu-Ice has a red frame. If the frame does not come back 5-10 seconds after clicking on the screen during the centering procedure, it means that communication has been lost with the XY stage. To restore the communication,

Open the crystal XY head controls in the toolbar

MOTION → *Goniometer* → *Crystal XY Head* (Fig. 21)

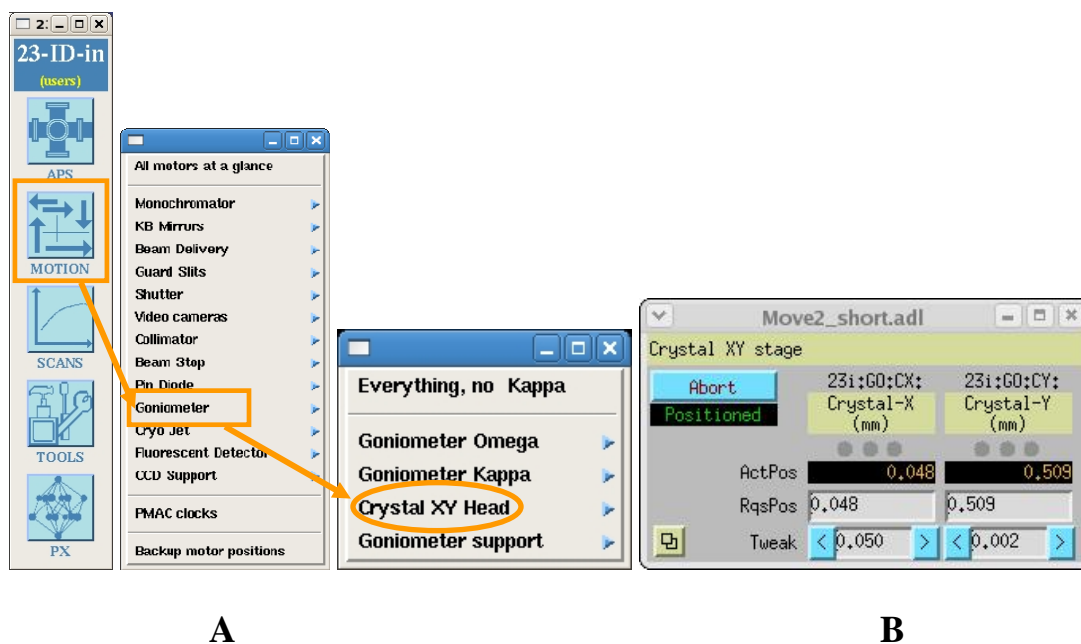


Figure 21. A) Path to the Crystal XY stage controls and B) Control window itself

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Place the cursor in the RqsPos window (for X or Y, does not matter) and hit “Enter”. Do not need to change the position by giving new number in this window. Red frame around the video image should be back and Point and Click is ready to proceed.

If point and click still does not work, make sure the “Crystal XY head” and the “Goniometer Support” motors are in auto mode.

Open *Motions* → *Goniometer* → *Goniometer Support* (Fig. 22 A)

On the resulting MEDM window (Fig. 22 B) click on a button with two small squares and choose “Large”.

On the resulting MEDM window (Fig. 22 C) click on “Auto”.

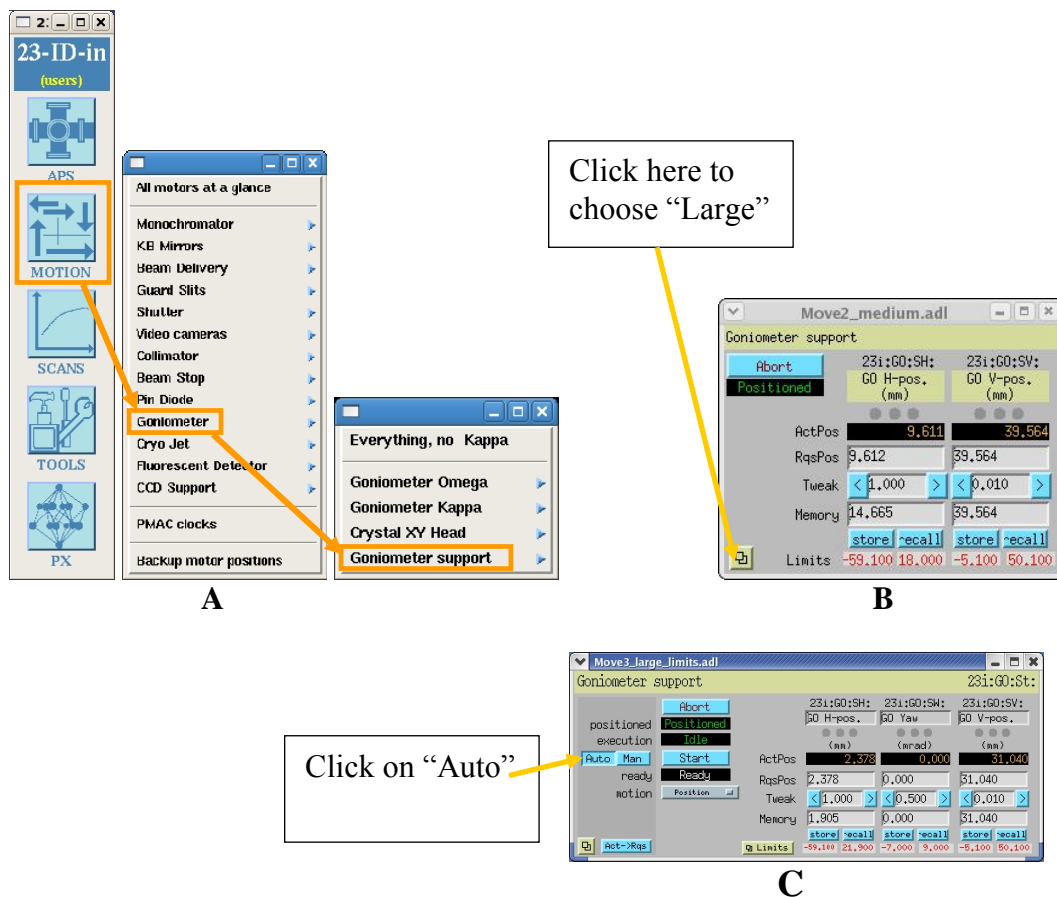


Figure 22

Similarly put the Crystal XY Head (Fig. 21) in auto mode.

Questions and/or suggestions on manual: Ruslan Sanishvili (Nukri).

Other questions and all complaints: your host

Last updated on 6/4/2011

Omega is slow

Speed did not reset from Data Collection Speed.

Open

MOTION → *Goniometer* → *everything* (Figure 21 A)

On the resulting MEDM window (Fig. 23A) click on Gonio Speed (yellow button). It will open another MEDM screen (Fig. 23 B)

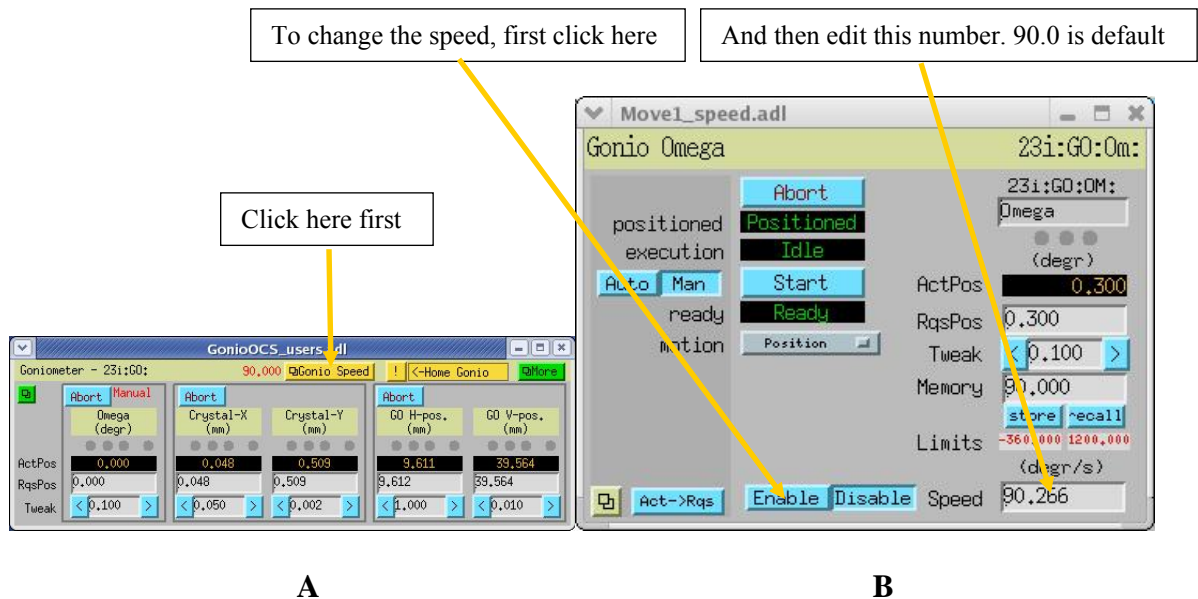


Figure 23 A) Goniometer full controls screen. B) Goniometer omega controls

No windows can be open

If MEDM or terminal windows cannot be open, it means the X-session is dead. Log out from the computer and log back in.

Beam is lost for no apparent reason

If the beam “disappears” for no apparent reason, check the APS stored beam current (Fig. 24). Alternatively, Go to <http://www.aps.anl.gov> and click on "Ring Status" in the list on the left side of the page, or go to <http://www.aps.anl.gov/aod/blops/status/smallHistory.html>).

If there is stored beam but the monitor on Fig. 25 reads 0, make sure all upstream shutters are open (all buttons on the PSS are green). If yes, move the energy with JBlu-Ice about 10 ev up or down and then return the energy to the desired value. Wait few moments to allow for self-tuning and beam should appear in the endstation. You may need to center the beam. See “Aligning the beam on Page 5.

Questions and/or suggestions on manual: Ruslan Sanishvili (Nukri).

Other questions and all complaints: your host

Last updated on 6/4/2011

If the reading on DCM V SUM (Fig. 25) is still 0 while there is stored beam, contact your host.

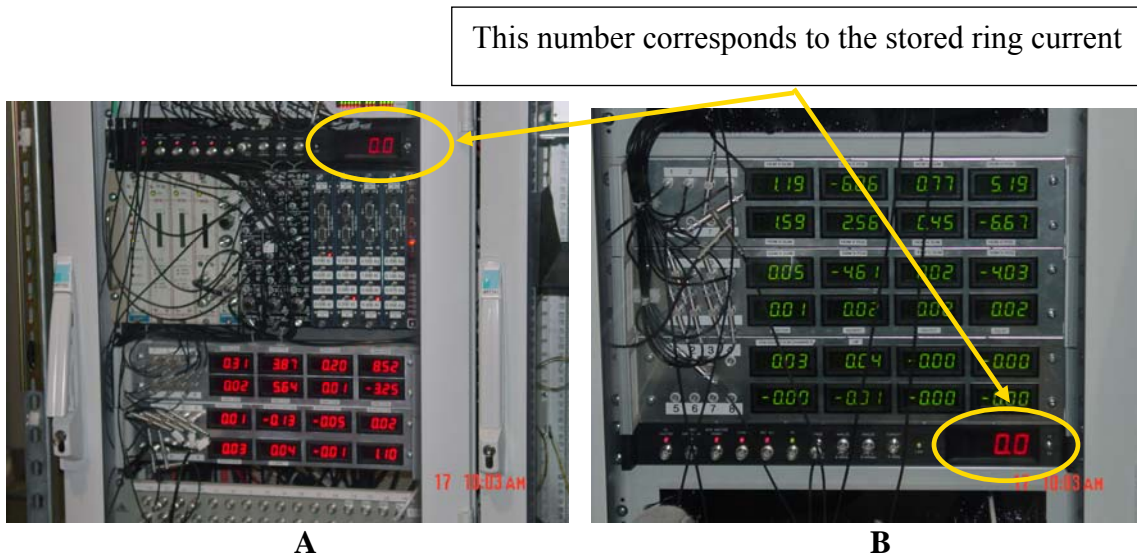


Figure.24 Stored ring current on A) IDin and B) IDout stations

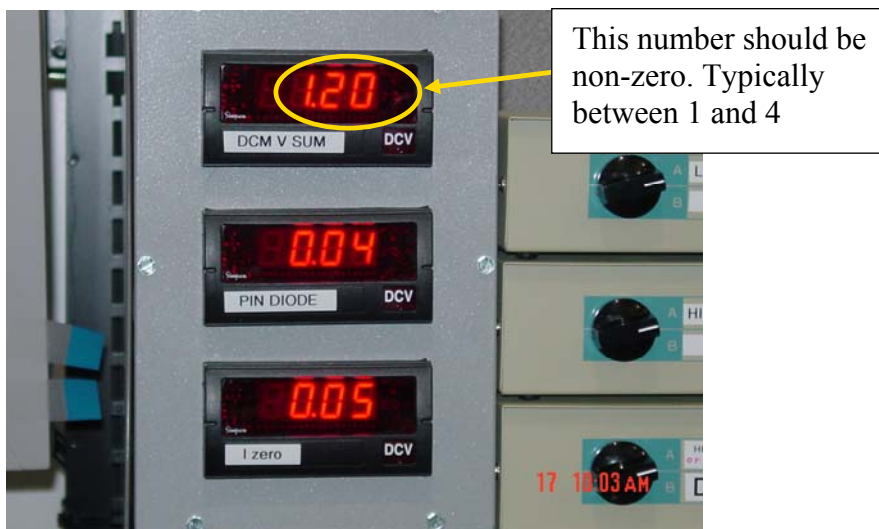


Figure 25 Monochromatic beam monitor.

Lost all icons at the bottom of the screen

- 1) Ctrl+Alt+F1 to switch to a text-mode terminal window.
- 2) Login with a current username and password
- 3) Type: killgnome.pl
- 4) Login back via normal graphical login