Changing Energies on 10-ID with New Monochromator

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Abstract

Several sections will be undergoing changes until the new mono is in the final operational state. Expect changes to happen on a semi-regular basis throughout 2015. It is strongly advised that regular users check back often for updated versions or discuss with beamline scientists if any changes have been made. Relevant information to the ID line can be found at (http://wiki.mr.aps.anl.gov/Index10Id). It is our goal to maintain as much consistency as possible and to diagnose errors as close to their occurrence as possible. If you notice any problems or have suggestions please forward them on to Joshua Wright (jwright@agni.phys.iit.edu).

The new Monochromator contains two crystal pairs. There is a Si (111) crystal that previous users will be somewhat familiar with as far as energy range and resolution is concerned. Additionally, a Si (311) crystal has been added that grants an energy range from 8700 eV to 67000 eV. It is possible to switch between the two crystals with small effort, and we are trying to simplify this procedure. For now please consult with the beamline scientist to do the crystal changes. The Si (311) offers higher resolution as compared to the Si (111) crystal, at the cost of flux.

Throughout setup procedure adjust detector gains as required. Begin with fast purge (maybe 500 sccm) with nitrogen supplied from head gas, not high pressure cylinders, for several minutes if switching between argon and helium. Set up camera with fluorescent screen (between Io and It). Check cabling for detectors (signal and high voltage), cameras, etc. This is intended to be a guide for setting up the beamline and does not cover all potential scenarios. If there are questions, problems, or concerns please consult with the beamline scientist.

1 User specific Information

Select the element that you wish to study: __________
Select relevant edge (K, L-3, etc): __________
Edge Energy (E_e): __________ eV (This value can be found on a chart to the left of the control station)

2 Basic Setup

Gas Mixture:
The general guidelines of 10% and 80% absorption in Io and It, respectively, are not suitable for 10-ID. While those are upper limits, we generally recommend less than 5 μA in detector current. Typically, this can be achieved with 1-2% absorption in Io and 30% absorption in It. One should always maintain a minimum of 20% Nitrogen in the mixture to prevent arcing in the detectors. Additionally, please allow at least 40 minutes for gases to interchange completely, depending on how different the gas mixtures are from the previous setting.

Io He: __________% N2: __________% Ar: __________%
It He: __________% N2: __________% Ar: __________%

Initial Alignment:

We have updated MX to include new pseudo and coordinated motors. These will speed up and simplify alignment of the beam. The motor **bl_angle** will set both the mirror and table angles for your experiments. It is highly recommended that one uses the resources available at henke.lbl.gov and the reflectivity of a thick mirror to determine this angle. Alternatively, you can follow the old method that will take a bit more time. This section is only necessary if the beam has been completely lost and needs to be recovered from a raw calibration standpoint. It is recommended that most users skip this section as it adds no benefit currently.
Move table to zero position.This value is lined up with the fiduciary mark for the beam height. It's not completely necessary, but it should ensure a level starting place and make the beam easier to find.

mabs bl_angle 0

Open the slits to help find the beam in case you are doing a large energy change from the previous user. It is beneficial to move the fluorescent screen between Io and It if not already done.

mabs av_size 10000
mabs ah_size 5000
mabs bh_size 10000
mabs bv_size 5000

When opening the slits there is a chance you will hit the limit switches for ***b_top*** *and/or* ***b_bottom****. You can attempt to fix this with* ***set mo b_(top or bottom) position 0*** *and then keep moving. This however would reset any absolute position used previously. Note that* ***bv_size*** *is a pseudomotor that is made up of two other motors* ***b_top*** *and* ***b_bottom*** *and will change if you adjust either of the component motors.*

Move the mirror out of the way of the beam.

mabs mirror_vert 5000
mabs mirror_tilt 0

3 Feedback

Additional scatterers may be needed, use either Al foil or kapton for high energies not scattered by air. Voltage on the top display should be between .2 and 1.5 V You may have to refer to this section throughout the set up if the beam gets lost.

If the beam is not visible AND beam is detected in the feedback detector, you may also need to adjust motor **b_top**. Be careful to use small steps (500 1000) so as not to exceed the **b_top** limits (note that the direct beam travels near the top of the b slit range).
The new mono has been calibrated to work within the feedback window for all energies. Adjustment of the feedback should be minimal as changes to these parameters can incite a resonance within the mono and introduce noise into the measurements. It is now perfectly normal for the spot on the feedback to wander from left to right during large energy changes. At the lowest energies it should be on the left hand side of the screen and on the right hand side for high energies. If there is concern about finding the beam you may still sweep the offset knob to find the peak, change the gain on the amplifier, or adjust the volt/div knob on the oscilloscope. Further adjustments should only be done by the beamline scientist at this time.
For reference the amplitude should be between 0.02 and 0.04 V and the frequency should not differ much from 165 Hz. These values might change and it is a good idea to ask if there are any concerns or problems associated with the beam if these values are different.

4 Insertion Device Mechanics

Some additional thought must go into the selection of the necessary harmonic for experiments. The Si (311) crystal does not have the same lower range as the Si (111) crystal does, and reciprocally, the Si (111) does not have the same upper range as the Si (311) crystal. More information about the specific ranges can be found on the wiki.

Choose proper harmonic: __________

1st 4.5 - 13.5 keV
2nd 9 - 24 keV (Consult beamline scientist before choosing this harmonic)
3rd 13 - 36 keV
5th 25 - 60 keV

Set this value using

set var und_harmonic __________

If not already open, open the undulator interface using either s10id from a command terminal or selecting the EPICS icon on the lower panel. This provides you with feedback about the undulator as you go through the following steps.

The undulator and the mono should be coupled. We do not yet have quick scans working correctly. For users that wish to experiment with additional scan types there is a motor scan that can potentially take data faster than a step scan with behaviour similar to a quickscan. For more information, consult the section on tapering or ask the beamline scientist.

Ensure that the mono and undulator are coupled.

set var id_ev_enabled 1

Then do an absolute move to the desired energy range.

mabs energy __________
(eV)
Now the undulator must be tuned to be optimized for the scan. Slowly adjust the undulator (**id_ev**) while watching the voltages for feedback and Io. Use small steps of about 10 eV unless you have done a large energy change, then it may be up to 200 eV different.

mrel id_ev 10
Once you have found the peak of the undulator increase the value by another 10-20 eV. This will help ensure beam stability. Be sure to measure the difference between the undulator and the monochromator energies and set the parameters accordingly.

set var id_ev_params 1 ∆E
Until quick scans are implemented you will need to ensure that you have set the correct edge energy. Do this by setting the variable as such:
set var edge_energy __________

5 Table and Mirror Geometry

Move to the proper stripe for your experiment

For energies greater than 18 keV use Pt. For energies below 20 keV use Rh. Check Hephaestus that there are no mirror absorption lines below your intended energies. For very low energies there is a SiO_2 mirror available for use.

mabs mirror_horiz __________
Pt: 60000
Rh: -3000
SiO_2: 31000

We have included a new motor that controls the angle for the table relative to the mirror. Use ($http://henke.lbl.gov/optical\_constants/mirror2.html$) to get an approximate angle for the table. Choose the appropriate mirror stripe for your experiment. Use either Pt, SiO_2, or Rh in the chemical formula. Change the scan parameter on the website from photon energy to grazing angle and go from 0 to 0.5. Then select an energy 1000 eV above the edge energy. A new tab will open with a plot showing the reflectivity curve. Choose the knee of the graph and determine the angle Θ_c.

mabs bl_angle (Θ_c)

Rezero A-slits, if necessary, by opening **av_size** to 10000 and then lowering **a_top** in steps until you just clip the beam, use either a timescan or monitor Io and watch the beam on the fluorescent screen. Next lower **a_bottom** until it also clips the beam. Slowly decrease **av_size** until the beam is completely cut off. Then **set mo a_top position 0** and **set mo a_bottom position 0** and now **av_size** will be zero. Reopen the slits and then close **av_size** slowly to make sure that the beam is cut off uniformly from top and bottom.

mabs av_size 5000

Move mirror halfway into beam. This value depends on the selected energy, but move in steps of 1000 until you get close.

mirror_vert : __________

Move **b_top** to reduce the beam by about 98%. Now close the remainder of the beam using **a_bottom** watching the fluorescent screen.

Scan the mirror angle with a **mirrorscan** and again choose the knee of the graph. This is more correct than the website as this is specific to our beamline and our geometry.

mabs mirror_tilt __________

Mirror tilting should be done at the end of EXAFS range.

The table needs to be adjusted to match the reflected beam.

bl_angle: __________

The mono now operates in fixed exit mode. This means that the height of the beam should not change. Ideally **mirror_vert** will be left at the appropriate height, but the value should be around -500.

6 Tapering

This section is only for a subset of scans called motor scans. These are similar to any of the slit scans or rastering scans that have been done many times, but instead call upon the energy motor. These scans make it possible to do cyclic scans of a sample. Experienced users are encouraged to try them and report any problems or benefits so that we can fine tune their usage.

Decouple ID and Mono

set var id_ev_enabled 0

Begin tapering to achieve as uniform as signal as possible over desired energy range of your scan. Run a scan of energy while recording Io over your scan range +/- 1000 eV. Step sizes should be 5-10 eV and a time of 0.05 seconds. Adjust taper until you get the least variation possible over the range you intend to scan across. Ideally, this would be a flat line with no slope. Taper can range from 0 - 5 mm, it is suggested that this is done in 0.5 mm increments.

taper: __________ mm

Consult the beamline scientist if you wish to perform rapid scans or cyclic scans and are unsure of how to set up the scans. These scans run a motor, like energy, with a fixed step size and measurement time. The benefit is that two scans can be created where the first scan goes from below the edge to the end of the EXAFS range and a second scan that starts at the end of the EXAFS range and goes to below the edge. In this manner you may collect data in both directions as the mono traverses over the energy range and back to start another scan. You will need to leave the mono decoupled from the undulator to run the scans at the highest speeds.

7 B-Slits

Prepare the slits so that you can find the center of the beam.
For Horizontal:
mabs bv_size 5000
mabs bh_size 400

Map out b-slit center (**bh_center**) positions for high and low energies (top and bottom of scan range). Scan once at an energy below the edge (E_0 100 eV) and 2-3 energies above the edge energy to the end of the expected scan range. Ideally all scans will line up, and there will be an area of flat intensity that is the same for all energy ranges. Use gnuplot to overlay the scans and find this region.

Choose **bh_center** that works for range of scan with optimum intensity. Additionally, choose the **bh_size** that is appropriate for your experiment. Find regions of flat intensity throughout the scan range.

mabs bh_size XXXX

Repeat for vertical center.

For Vertical:
Set the vertical size by **mabs bv_size 400** and then run the vertical slit scan in the same way you did for the horizontal center.

8 Scans

There are a few scans that users who are setting up the beamline should be familiar with.
mirrortilt This scan will provide a reflectivity scan as the mirror goes from flat to overtilted.
vslit Scans the b-slits in a caterpillar motion vertically so you can find the center of the beam. If this is stopped while moving your slits will be off.
hslit Scans the b-slits in a caterpillar motion horizontally so you can find the center of the beam.

Perform a scan on a known standard and calibrate energies. Verify that detectors are working correctly and that there is low noise in the system.

An optional final adjustment would be to close the a-slits to reduce radiation in the hutch, which may be required for sensitive samples.