# CHESS Winter 2015

*February 18-24*

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Brendan Pratt, Richard Jones

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## Goals for this run

1. Adjust camera to be able to view 7mm x 7mm diamonds
2. Create a procedure for determining the offsets between the crystal planes and the mount
3. Image newly cut diamonds
4. Determine angular offsets of the reflection plane for each diamond with respect to the surface

## Useful Information

- Phone numbers
    - C1 hutch: 607-255-0256
    - Ken's office: 607-255-0914
    - Ken's home and mobile numbers can be found on a piece of paper hanging from the C1 computer monitor
- To edit an image in a terminal:
    - display <*filename*>.tiff
    - left click to open menu, click enhance -> normalize
    - click view -> resize -> change to 320x270
    - save -> <*filename*>.png
- To take a screenshot in Linux
    - Whatever window you want a screenshot of, make sure it is visible
    - open a terminal and type xwd > <*filename*>.xwd (xwd file type recommended)
    - The cursor should have changed to a plus sign. Click on the window you want a screenshot of.
- To open the ANDOR GUI
    - In the terminal type the command *andorview*
- Image directory and filenames
    - /mnt/andor1/Data/Jones/Feb2015/
    - JonesFeb182015_1_xxx.tiff, where xxx is the image number
- Richard's orientation presentation

## Diamond Inventory for this run

- JD70-1

- JD70-2
- JD70-3
- All of the 4mm diamonds

# FourC Commands

Motor names

- *tth* - two theta
- *th* - theta
- *chi* - chi
- *phi* - phi
- *topsc* - flourescent top screen
- *spinz* - high of the hoop along the phi axis

Commands

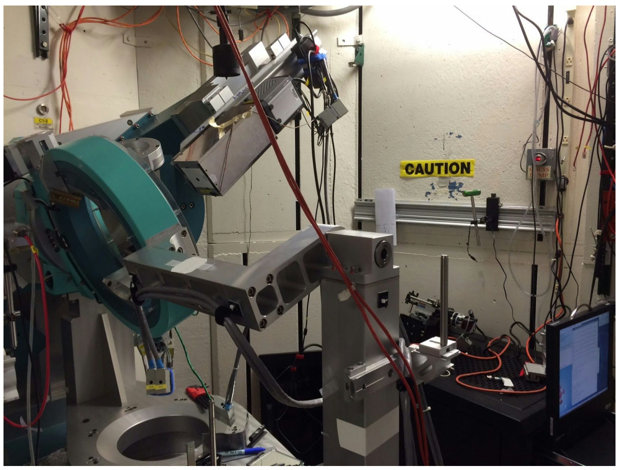
- *ad_lineup_on <time in second>* is the command to turn on the image updater
- *ad_lineup_off* is the command to turn off the image updater
- *tw <motor> <increment>* - tweaks a motor an increment from your current position
- *set <motor> <position>* - declares the current motor position
- *mv <motor> <position>* - moves a motor to a absolute position
- *wh* can be used to check the current values of all settings
- *newfile <filename>* - start a new file for each diamond. The filename should be the diamond name - diamond number - study1 (Ex. JD70-1-study1)
- *ad_on* is the command required before starting a scan
- *ascan <motor> <starting position> <ending position> <number of steps> <exposure time>*
- *shutter_auto 0* - turns off shutter
- *opens* - opens the shutters. This is useful if I2 on the dash above the monitor is low, approximately 100. I2 should be 12,000 or higher when there is beam.
- *closes* - closes the shutters

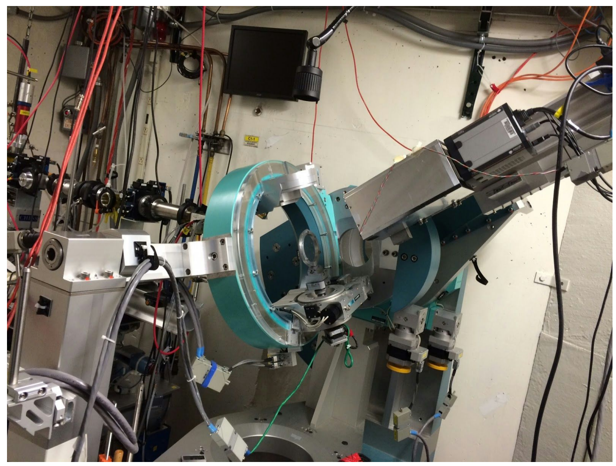
# Conditions for the experiment

- Beam current:
  - start of fill:
  - end of fill:
- Beam dimensions:
  - 
- Camera dimensions:
  - 
- I0 current:
  - start of a fill:
  - end of a fill:
- Time between fills:
- Duration of one fill:

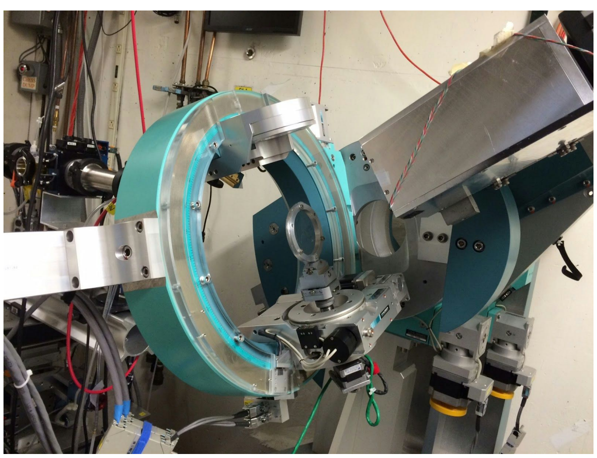
- Inside the ANDOR GUI:
    - Image binning:
    - Image size:
    - PreAmp gain:
    - Encoding:
    - Shutter mode:
    - Readout rate:
    - Overlap:
    - Noise filter:

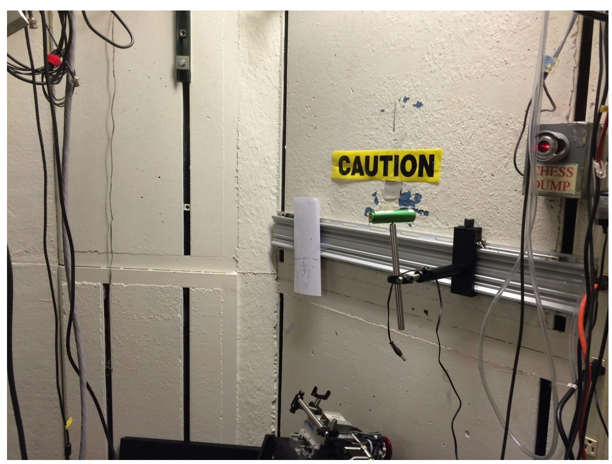
## Photos of the current hutch setup



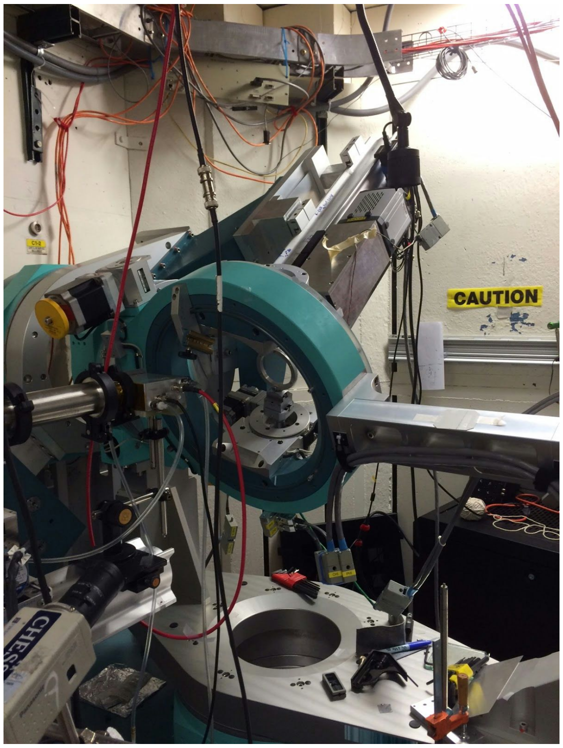



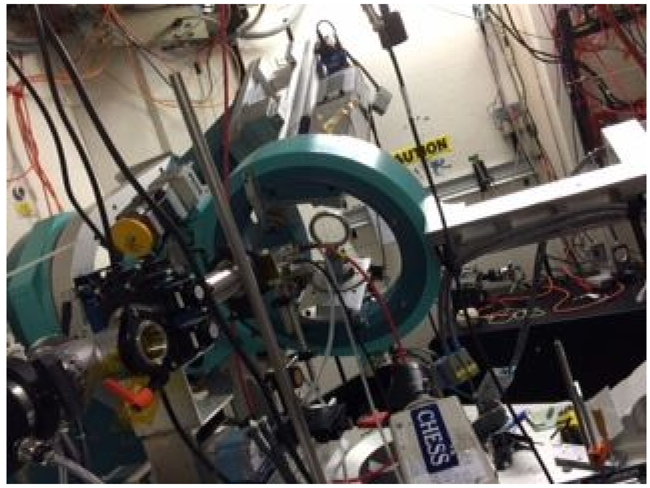
Left: Downstream view. Camera mounted in-line. Laser and paper on back wall.
Right: Upstream view. Hoop and camera mounted



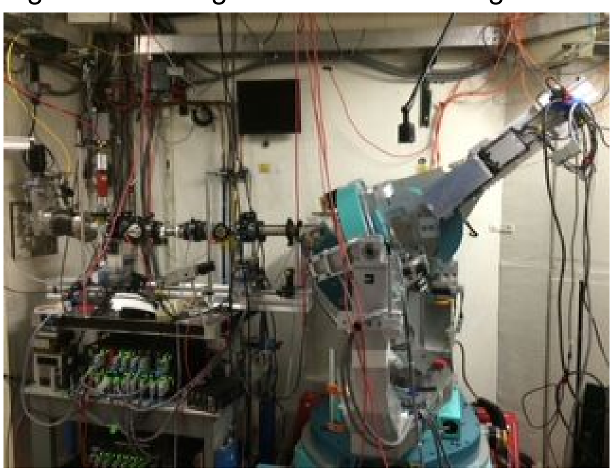
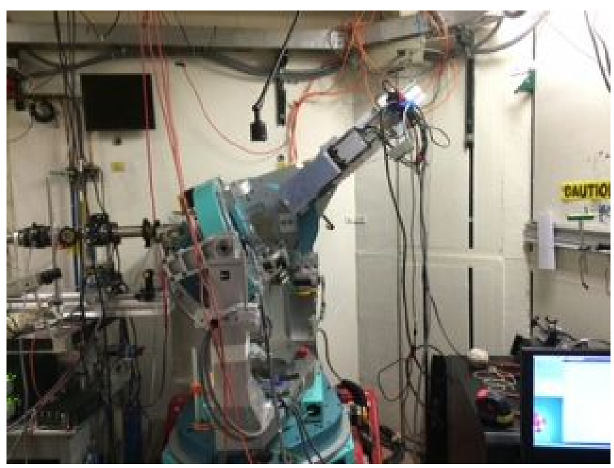



Left: Close up of upstream view.
Right: Close up of laser and paper used for adjusting the surface normal of the diamond

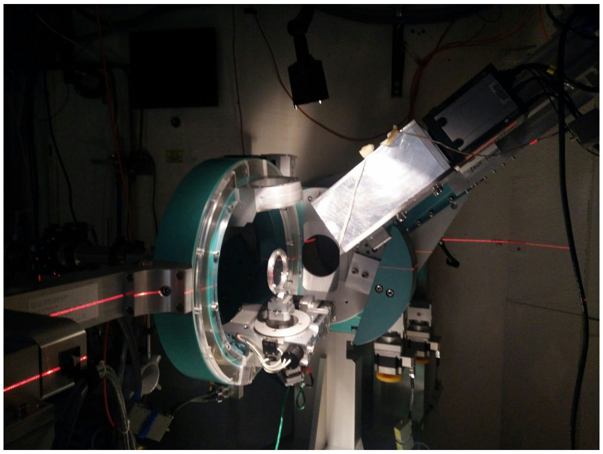


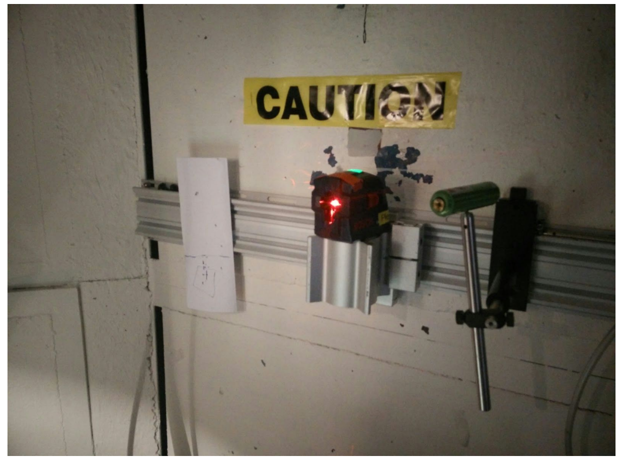



Left: Close up of the downstream view.
Right: View along the beamline looking downstream

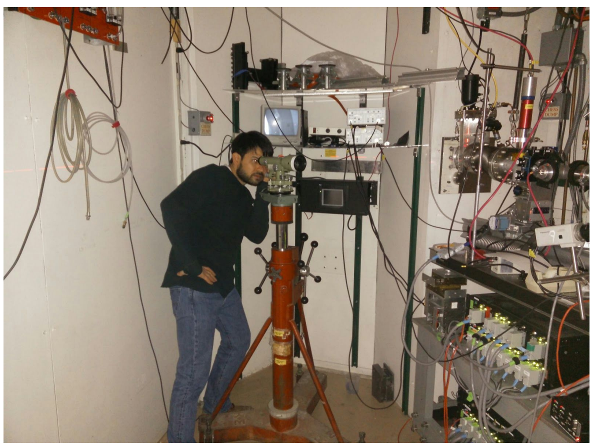



Left: Broad view of the beamline and goniometer
Right: Overall view of the setup including the laser and paper






Left: Setup with laser level. Light above turned on but hutch light turned off
Right: Laser level setup on the downstream wall



Left: Nathan determining if the diamond is level horizontally.

# Initial setup

**2/18/2015 AB, NS, KF**

There have been changes made to the hutch configuration since our last visit. The camera is no longer placed at a 90° angle to the diffracted beam; it is now in-line. For our purposes Ken adjusted the slit openings to increase the amount of beam that will hit the diamond. He also mounted a slit on the rail instead of the camera to make sure the diffractometer arm was in line with the beam.

## Finding the center of rotation and placing a mounted diamond in the beam

**2/18/2015 AB, NS, KF**

The point was placed in the goniometer mount in order to find the center of rotation. Ken did this before we arrived. There is a surveying scope in the corner of the hutch that is used to see where the center of rotation is. A laser was mounted such that it shone on the tip of the point. The point was then removed and replaced with a diamond mounted in a hoop. The spinz motor had to be adjusted to bring the diamond into position in the beam. Using the laser we were able to place the diamond where the point tip previously was. We then unlocked the phi motor and rotated phi to see if the laser reflected off the diamond the same way from both sides. It was important to look at the smearing when the laser grazed the diamond and to make sure it looked the same on both faces. When it's not, adjust the translation stage underneath the hoop until the reflection is symmetric.

### Changing the camera screen to accommodate 7mm diamonds

**2/18/2015 AB, NS, KF**
At the end of our first day here Ken asked the OWL shift worker, Tom, to work on the camera. Tom was the person who originally worked on the camera and was therefore familiar with it. We needed to remove the original screen which was mounted on a piece of aluminum and machine out space for the new screen. Tom did this with Ken's help.

### Adjusting the two-theta rail

**2/19/2015 AB, NS, KF**
We came in today and the new screen had been mounted to the camera. The camera was placed on the rail with fluorescent paper surrounding the opening. Using the beam we looked to see how the image appears in the camera. We discovered that the beam was not hitting the camera where we expected it to. The distance the slit was away from the rail is greater than the distance the camera opening is away from the rail. The diffractometer arm was level, according to a level, whereas the rail had the upstream end pointed down slightly. When the camera is on the rail with fluorescent paper around the opening it can be seen that the beam hits the top right of the opening when looking downstream. We corrected for this by using thin sheets of aluminum as shims for the rail to adjust the vertical alignment. We used the camera to check to see how the position changed.

The horizontal distance adjustment is trickier to deal with. We had to unmount the entire rail in order to add shims to the rail mounting brackets in order to center the camera opening on the beam axis.

## Determining offsets at CHESS

### Aligning the surface normal parallel to the beam with Ken's diamond

**2/18/2015 AB, NS, KF**
Once the mounted diamond was adjusted to be in the center of the beam we moved two-theta to be in the horizontal position. Instead of having the camera attached to the rail we used the slit with a diode attached to the back. There is tape covering the diode to prevent visible light from entering. The current from the diode is sent via BNC cable #2 to the "Pin Diode" value on the dash above the computer monitor. We adjusted the gain to be around 40 when the hutch was open with the lights on. We then closed the hutch and opened the shutters to see the reading on

the diode. It increased to around 6000. The hutch was then reopened and fluorescent paper was placed in front of the diode and the theta and two-theta motors were placed to their approximate locations of 19 and 38, respectively. We then scanned for a peak on the fluorescent paper before removing the paper.

The process now involves measuring theta, two-theta, chi and phi for one 220 peak and then repeating it with phi rotated by 180°. In order to start the process Ken had to make an adjustment in FourC to the counters by typing the following:

> counters
Counter number for monitor? > 3
Counter number for detector? > 4

We then did a scan in theta using dscan th -0.01 0.01 20 1. While this ran a graph appeared with theta on the x-axis and the diode current on the y axis. We used this to find the peak in the signal for theta. The first pass was too coarse and so we repeated it again with dscan th -0.005 0.005 20 1. This provided a good curve so we applied the command, mv th CEN, which moved the theta motor to the center of the peak we found in the plot.

We then repeated this process for the chi motor, dscan chi -0.5 0.5 20 1 followed by mv chi CEN, as well as the two-theta motor, dscan tth -0.5 0.5 20 1.

The diode had interesting behavior where it would sometimes show two peaks in the plot. We tried switching it out for another but the new one also showed the same issue. It was still clear where the peak should be so we continued on.

After repeating the previous scans we found the maximums in the diode current for each motor to be:

tth = 38.4
th = 22.027
chi = 75.868
phi = 90

We rotated phi by 180° and repeated the measurements. It is expected that two-theta will be about the same. When looking at the diamond it is obvious that chi is now off. We took the ideal value for chi, 90, and subtracted the current value of chi, 75.868, to give us about a 14° difference. Chi was compensated by moving it by 28° in the positive direction so that it was off by the same amount on the other side of the ideal value.

Ken typed the command “ca 2 2 0” to give a predicted location of theta for the given energy. We used this value to adjust theta like we did for chi. We took the current value, 22.027, and subtracted the ideal, 19.1, and then adjusted theta by twice this amount in the negative direction. Phi was adjusted to 270.

Fluorescent paper was used to find the new peak and then removed so we could repeat the process from before to find the new motor locations of the peak:

tth = 38.4
th = 16.315
chi = 104.08
phi = 270

We then took the average of theta, 19.171, and chi, 89.974. We moved theta to 19.171 and redefined it at 19.2, and we moved chi to 89.974 and redefined it at 90. Ken noted that the physical chi dial is 270.05. The diffractometer is slightly unlevel.

At this point we have made the diffractometer's chi plane perpendicular to the beam axis.

The next part of the process is to shine a laser on the diamond and monitor the reflection as chi is rotated. When the reflection doesn't change position then the beam is normal to the surface of the diamond at th = 0. It turned out that the laser reflected multiple times so it's important to pay attention to the true reflection and not the secondary reflections.

We placed the laser and a piece of paper on the downstream side of the diamond. This allowed us the ability to walk around and mark the paper where the spot appears. The first adjustment we made was to the horizontal using phi. At chi = 90 we marked the laser spot and then changed phi to 270 and marked the primary reflection again. We now tweaked phi such that the primary reflection moved halfway back to the original position. This process was repeated until a rotation in chi did not show any shift in the horizontal direction. Phi was recorded to be 90.02.

Similarly, we adjusted a theta-like rotation to remove the vertical displacement when rotating chi by 180. This is done by tweaking the stage beneath the hoop mount by a small amount.Sw process is the same as before but now in the vertical direction. We started with the primary reflection for one chi value, marked it, then rotated chi by 180 degrees and marked the spot again. The theta-like angle is tweaked to bring the spot halfway back to the original position and is repeated until a rotation in chi does not change its location. This also means that the peak will no longer be at the ideal 19.171 theta location.

This diamond is now setup and ready for us to find the new peak locations to determine the offsets between the surface and the 220 planes.

## Measuring the angle offsets with Ken's diamond

**2/19/2015 AB, NS, KF**

Now that the diamond has been set up to have its surface normal parallel to the beam, we can now find the location of the peaks and use the information to determine the angle offsets of the 220 plane from the surface.

At this point we will need to record the values for theta, two-theta, chi and phi for two different chi values 90° apart. It is also wise to bring theta back to zero and check that the crystal normal is still parallel to the beam.

We placed a fluorescent paper on the front of the camera to help find the first peak. The values we found for the peak are:

th = 21.2100
tth = 38.4000
chi = 75.6100
phi = 90.0200

Chi has been moved to 165.6100. The fluorescent paper was placed over the camera again to help find the peak. We ran into an issue with finding the second peak because the shutter had closed itself. We had to use the command *opens* to reopen the shutters. Also, if the image stops updating use *ad_lineup_on 1* to update the image. We found the reflection on the fluorescent paper and have removed the paper.

th = 19.686
tth = 38.4
chi = 165.61
phi = 90.02

In order to know the offsets we also need to have another reference point from the crystal. We decided to use the bottom edge of the crystal. The idea is to find the chi value that makes the bottom edge horizontal. To do this we are mounting a laser level on the downstream wall, shining it onto the crystal and looking through the surveying scope to see how much chi needs to change.

We added a laser level to the back wall and turned off the lights in the hutch (see [the setup pictures]). The light connected to the ceiling was turned on to provide enough light to see the diamond in the hoop but dim enough to see the laser when looking through the surveying tube. We adjusted chi until what visually looked like the top of the diamond was level with the laser. In reality it was the bottom of the diamond that was being compared to the laser due to a vertical flip from the surveying telescope. We found the diamond to be level when chi = 77.5°.

**2/20/2015 AB, NS, KF**

After speaking with Ken, the offsets of the planes should be determined by taking theta_measured - theta_bragg as well as chi_measured - chi_horizontal. For this diamond we have:

offset_theta_1 = 21.210° - 19.171° = 2.039°
offset_chi_1 = 75.610° - 77.500° = -1.890°
offset_theta_2 = 19.686° - 19.171° = 0.515°

The second offset for chi was not calculated because we changed diamonds before measuring the horizontal offset when the chi peak is at 165.61. Also, the value theta = 19.171 comes from using a 15keV beam. Ken said we can measure it and it's likely to be off by at most 0.1%.

## Aligning the surface normal parallel to the beam with Ken's second diamond

Nathan and I started working on aligning the second diamond that Ken had mounted. This diamond was mounted while we were busy with the collaboration meeting. Paper was mounted to the downstream wall and the laser pointer was turned on. Nathan looked through the surveyor's scope to see when the laser spot hit the center of the diamond. We then disengaged the chi motor by turning the bolt underneath the ring 3-4 full turns and then tipped the motor gearbox towards the ring. It should make a click noise.

Now that chi was free to rotate we marked a spot on the paper with a pen where the original laser spot was. We then rotated chi by 180 and marked the spot again. The first adjustment we made was to move phi so that the new beam spot moved halfway between the old and new horizontally. Once we moved it we marked the new location and rotated by chi again. The same procedure was followed until we were satisfied with the horizontal alignment.

Once the horizontal alignment was completed we adjusted the theta-like angle on the mount. When chi is set to 90 (the hoop is upright) then this theta-like angle is theta. It is the polar angle of the hoop and is adjusted on the base holding the mount. Like with phi, this was adjusted until the beam spot moved halfway between the initial and 180 rotated position. It was repeated until a rotation in chi yields a difference in beam position within one beam spot distance away.

We re-engaged the chi motor and looked through the surveying scope to find the chi value that makes the bottom edge of the diamond horizontal. This chi value is 87.500.

## Measuring the angle offsets with Ken's second diamond

We placed the fluorescent paper on the camera and looked for the reflection from the diamond. Once we found the peak we removed the paper and looked to adjust the reflection to be centered in the camera. The position of the reflection is

th = 18.375
tth = 38.4
chi = 86.2
phi = 88.46

Now we rotate in chi by 90 and repeat the previous measurement starting with the fluorescent paper. The position of the reflection is

th = 20.0720
tth = 38.4
chi = 176.2
phi = 88.46

The offsets are

offset_theta_1 = 18.375° - 19.171° = -0.796°
offset_chi_1 = 86.2° - 87.5° = -1.3°

offset_theta_2 = 20.072° - 19.171° = 0.901°

### New method of determining the offsets

**2/21/2015 AB, BP, RJ**

At breakfast Richard discussed a different procedure for determining the offsets. The plan uses the hoop as a reference instead of the surface normal of the diamond. This will provide one reference for all of the mounted diamonds instead of a reference for each diamond individually. One way to do this would be to mount a piece of glass to the hoop and use the measurements of the glass as a reference.

## Diamond mounting

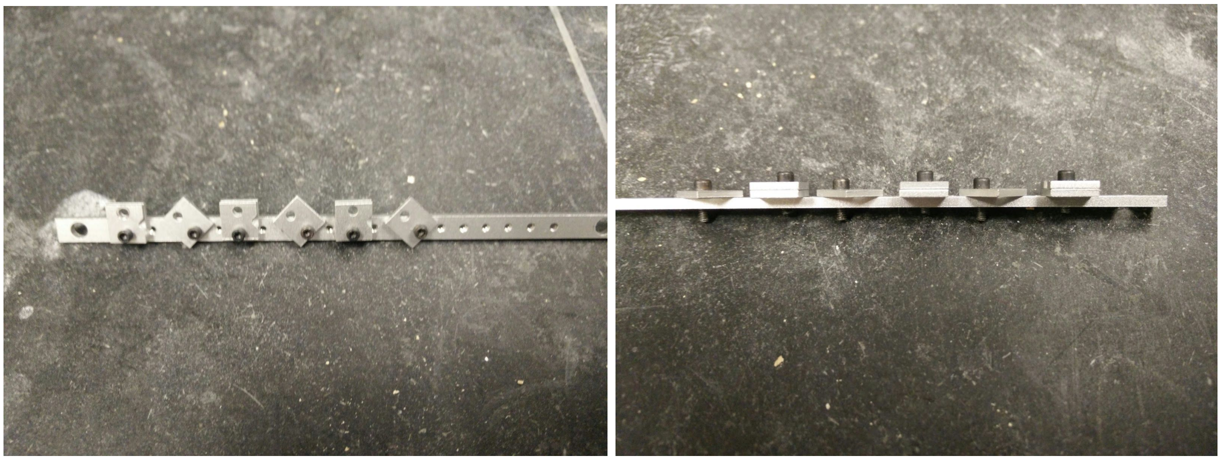
**2/21/2015 AB, NS, BP, RJ**

### Required setup in the CHESS chemical room near the operator

- Microscope with illumination
- Diamonds
- Hoop for the diamond mount
- Epoxy
- Diamond mount
    - support bar (branches)
    - leaves
    - jig

### Pictures of the mounting setup

Mounting area with microscope and hoop



Mounting jig for the diamonds viewed from above and from the side.

### Mounting procedure

1. Clean counter space
2. Clean optics of the microscope if necessary

## Diamond images under the microscope

After setting up the diamond mount Brendan and Alex went to mount the diamonds and discovered JD70-1 had fragmented into 5 pieces. It was brought under a microscope with 32x zoom. Obvious holes were visible and appear to go through the diamond entirely in both the central and frame regions. There is also a visible crack in the frame on the largest of the fragments. Since we don't have a camera mount for the microscope we are using our cell phone to take a picture through the eyepiece.

We suspect that the fracture in the frame was a result from Sinmat and act of traveling with the diamond caused the shatter.

Brendan needs to mark the mount leaves to remember which diamond is where. A single black dot means it's diamond UC30-16. Two dots means JD70-2. Three dots means JD70-3. Four dots means it's J1a50. Five dots means J2a100. Six is Si45-90.

## Determining the offsets using the JLab hoop

### Aligning the JLab mount

**2/22/2015 AB, NS, BP**

We mounted the JLab mount into goniometer using one 1/16th inch shim and securing it with a bolt through a threaded hole, see the pictures below.

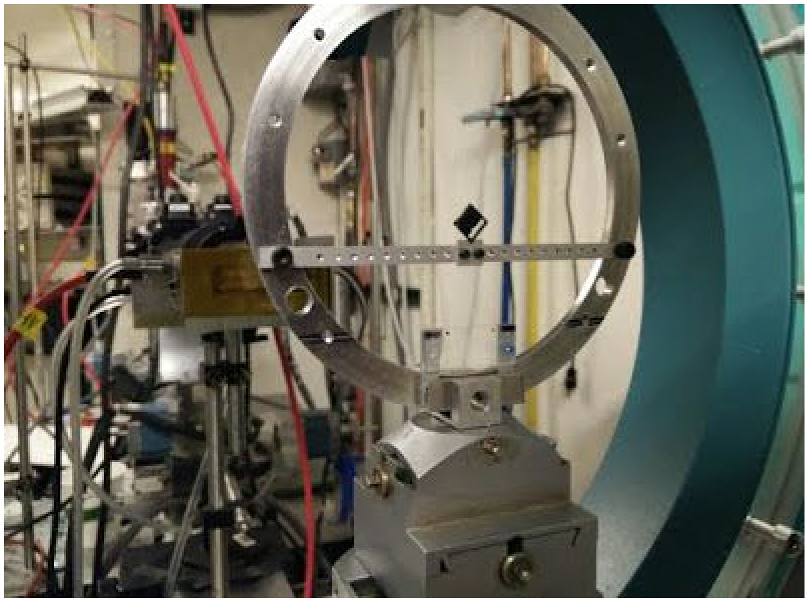
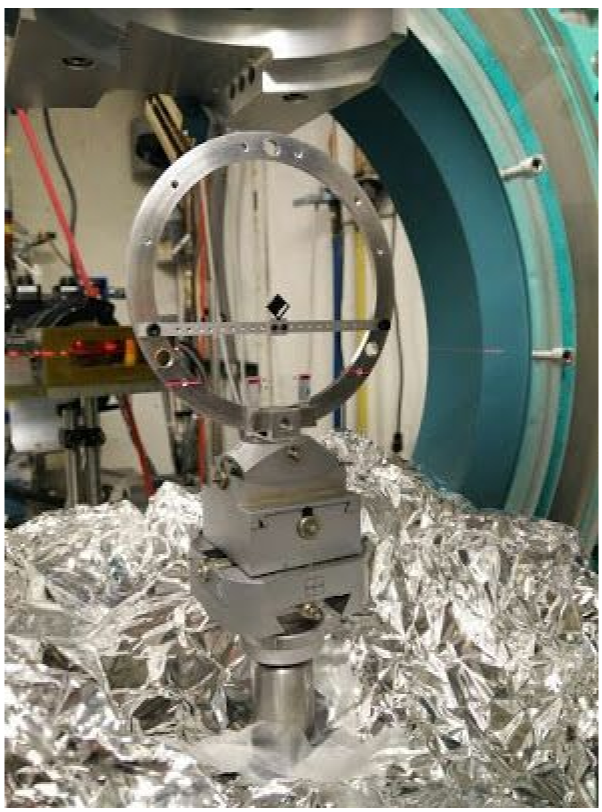


Image of the diamond mount fixed in the holder in the Cline goniometer, with glass slide, holding JD70-3. The photo was taken from downstream of the target, looking upstream toward the source of the X-rays.

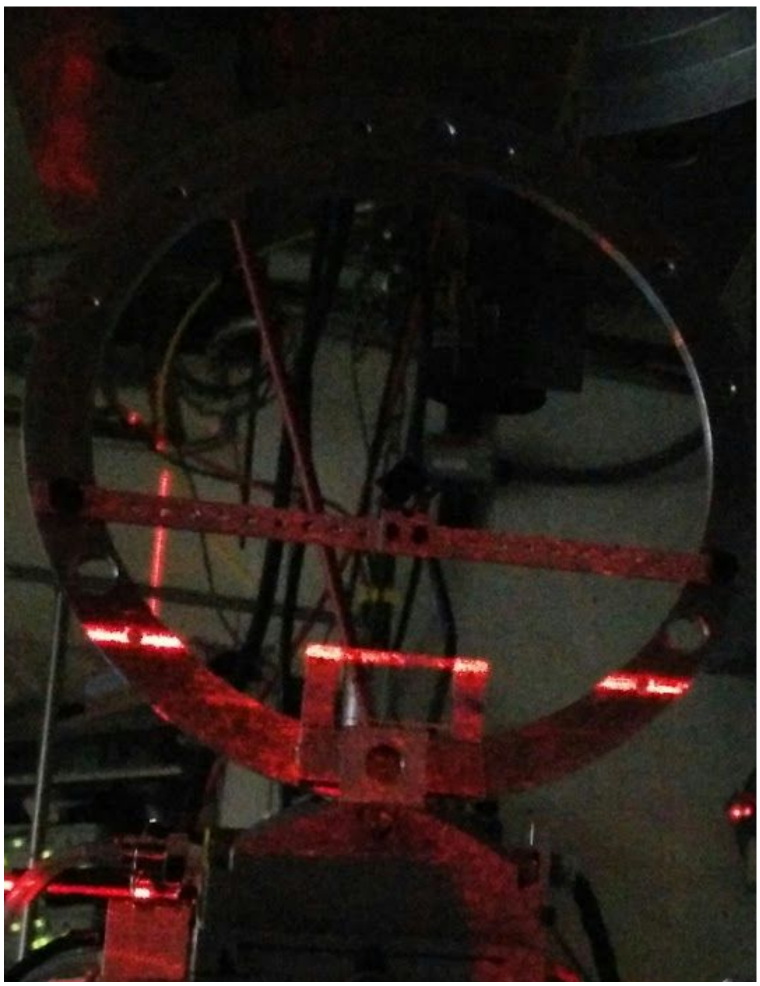


Picture of diamond mount held in goniometer with aluminum foil safety net underneath in the case of a diamond falling off its mount.The leveling laser appears in the photo as a horizontal red line cutting across the upper edge of the glass slide. The glass slide is epoxied to two aluminum legs that screw onto the ring.

The spinz motor needed to be adjusted to bring the glass slide into the center of rotation. The motor was near the maximum limit. Looking through the scope we could see that the glass slide was in the center. As a starting point we adjusted phi so that the hoop was parallel with the goniometer.

We proceeded to align the normal of the glass to be parallel with the beam by reflecting a laser off the glass, marking the spot on a piece of paper, and rotating in chi. The chi motor was disengaged and the chi value was changed by hand. Each rotation of chi we altered only one angle, either phi or the hoop's theta angle by moving the spot halfway to the previous location. Rotating chi by 180 moved the beam spot within 1 spot diameter.

Initially we tried to align the edge of the glass with the laser level but this proved to be difficult since the laser did not show up on the glass as well as it does on mylar. We decided to mark the hoop itself along the laser level, see picture below.



Picture of the horizontal chi reference. Notice the red laser is centered on the two bolt holes. The chi angle when this occurs (with theta = 0) becomes the reference against which crystal chi offsets are measured.

Any future alignment will be done by rotating chi such that the laser goes across these marks on the hoop.

The motor positions for the glass slide reference are:

th = 0.0000
tth = 38.4000
chi = 89.7000
phi = 88.2600
spinz = 24.625

## Attaching and positioning the JD70-3 diamond in the hoop

Now that the hoop's reference has been found we can mount a diamond to the mount. For our purposes at CHESS we decided to mount one diamond at a time at a specific location on the bar. When it comes time to exchange diamonds we will have a new diamond mounted on another bar at the same location and will place it on the hoop in the same position. This will allow us to avoid having to realign the hoop for every diamond.

The first diamond to be mounted is JD70-3. The spinz motor needs to be adjusted to bring the diamond to the center of rotation. We moved the spinz motor to -5.075. Chi now needs to be adjusted to make the side of the diamond horizontal. Chi is now 47.2.

## Finding the reflection for JD70-3

We attached fluorescent paper and adjusted the video camera to see the camera aperture. On the tv monitor we drew a circle around the aperture. We moved theta to 19 as a starting point and tweaked theta in the negative direction to find the reflection on the fluorescent paper. We found theta to be 17.073 for the reflection.

The paper was removed and we scanned again to find the reflection. The reflection appeared on the left of the monitor so we tweaked chi to bring it to the right. This involved moving chi in the negative direction to bring the reflection to the right of the screen.

**2/22/2015 AB, NS, BP, RJ**

Richard arrived and asked about how the diamond was mounted. It turns out we had the mount backwards in phi. To correct this we rotated in phi by 180 degrees to correspond to Richard's orientation plan.

We also changed the name of the directory of where the files are stored. We did this by cd-ing into a directory of our choice.

## Realignment of the glass slide

After discussing with Richard it was decided to redo the alignment process. We talked to Ken about a more exact way of aligning the mount in the center of rotation. For this we added a video camera with a zoom lens aimed perpendicular to the beam axis looking at the glass slide edge on. We then rotated in phi by 180 to see how the glass shifted. We then tweaked the mount translation stage until the rotation did not affect the glass slide's position in the camera.

Once this was finished we re-aligned the normal of the glass to be parallel with the beam axis using the laser setup. We also adjusted the horizontal by lining up the laser level through two bolt holes on the hoop frame. We made sure that the orientation of the diamond agrees with the orientation in Richard's presentation linked above in the Useful Information section, with chi advanced by 90°.

The motor values for the reference slide are:

th = 0.000
tth = 38.852
chi = 78.50
phi = 90.25
spinz = 24.575

## Scanning of JD70-3

We moved spinz to position the diamond. Spinz is -4.50 for the diamond to be in position. We attached fluorescent paper to find the peak and then removed it to find it in the camera. The rocking curve looks wide so we needed to reduce our step size.

ascan th 21.370 21.500 261 10

The whole diamond was not visible. We went back and adjusted spinz and the translation stage to bring the diamond into a better position. We also tried changing the opening of the shutters with *tw dslb .5* and *tw dslt .5*. We are now able to see the frame of the diamond on all sides and ran a scan

ascan th 21.350 21.500 750 10

The motor values need to be recorded after the scan.

| Filename | Orientation | Steps | Step size (degrees) | Exposure (s) |
|---|---|---|---|---|
| JD70-3-study1_scan001* | (2,2,0) | 261 | 0.0005 | 10 |
| JD70-3-study1_scan002 | (2,2,0) | 750 | 0.0002 | 10 |
| ~~JD70-3-study1_scan003~~ | ~~(2,-2,0)~~ | ~~cut~~ | ~~short~~ | |

| | | | | |
|---|---|---|---|---|
| JD70-3-study1_ scan004 | (2,-2,0) | 375 | 0.0004 | 10 |
| ~~JD70-3-study1_ scan005~~ | | | | ~~10~~ |
| JD70-3-study1_ scan006 | (2,2,0) | 250 | 0.0004 | 10 |
| JD70-3-study1_ scan007 | (2,-2,0) | 250 | 0.0004 | 10 |

| **JD70-3** | **Orientation** | **Tth** | **Th** | **Chi*** ** | **Phi** | **Time** |
|---|---|---|---|---|---|---|
| ~~scan001~~ | ~~(2,2,0)~~ | | ~~21.37-21.50~~ | | ~~90.25~~ | |
| scan002 | (2,2,0) | 38.252 | 21.35-21.50 | 17.740 | 90.25 | 19:00 |
| ~~scann003~~ | ~~(2,-2,0)~~ | ~~38.752~~ | ~~17.00-17.15~~ | ~~20.7398~~ | ~~270.25~~ | |
| scan004** | (2,-2,0) | 38.752 | 17.00-17.15 | 20.7398 | 270.25 | 20:07 |
| ~~scan005~~ | ~~(2,2,0)~~ | ~~38.352~~ | ~~16.93-17.03~~ | ~~133.5397~~ | ~~270.25~~ | ~~12:17~~ |
| scan006 | (2,2,0) | 38.352 | 16.93-17.03 | 133.5397 | 270.25 | 12:22 |
| scan007 | (2,-2,0) | 38.352 | 21.29-21.39 | 130.5397 | 90.25 | 1:33 |

*It was discovered after the scan completed that the crystal was not entirely in the beam, we adjusted the translation stage and spinz until the diamond was in the cross hair of the surveyor scope. This positioned the diamond in a position relative to the beam that would include the its entire area.

**This scan had too large of a theta range. The first signs of a reflection appear around 17.032

***The chi motor drive gear was not fully engaged with the stage gear, so these chi values are off by unknown constants. See notation below for when this was discovered and rectified.

## JD70-2

Brendan mounted JD70-2 to the second bar before he left for the night. After finishing with JD70-3 it was removed and walked to the fume hood in the chem room. The paper clips were removed from JD70-2 and placed on the bar holding JD70-3. JD70-2 was then walked down to the hutch and mounted. One person holds the bar from the downstream side and the other tightens the bolts from the upstream side.

The goniometer was not moved in anyway and a peak for JD70-2 was found with some minor tweaking.

It was also determined that the upstream slit was blocking the top of the diamond. The motor is called uslt and was moved by 1 degree.

| Filename | Orientation | Steps | Step size (degrees) | Exposure (s) |
|---|---|---|---|---|
| JD70-2-study1_ scan001 | (2,-2,0) | 100 | 0.0002 | 10 |

| JD70-2 | Orientation | Tth | Th | Chi* | Phi | Time |
|---|---|---|---|---|---|---|
| scan001 | (2,-2,0) | 38.552 | 21.622-21.642 | 126.239 8 | 90.25 | 2:59 |

*The chi motor drive gear was not fully engaged with the stage gear, so these chi values are off by unknown constants. See notation below for when this was discovered and rectified.

After JD70-2-study1 scan 1 was completed, we had trouble finding the second reflection after a 180 degree phi rotation. Several iterations iterations later, we noticed that the chi motor was slipping during an angle change. The way this was discovered was we found a reflection, then moved away in chi and came back, and it was no longer visible. This was eventually traced back to how the drive gear on chi is re-engaged after it has been loosened for free chi rotations. To fully seat the gear in the worm, one must wiggle the chi stage back and forth while pushing sideways on the motor. When the gears are properly aligned, there is a “clunk” sound and the nut tightens several turns further than it would it not fully engaged. It is important to maintain sideways pressure on the motor, after the “clunk”, while tightening the nut to prevent the gear from slipping back out before the nut is fully tight.

**All scans that have been performed up to this point have suffered from unknown chi offsets. To know the offsets of JD70-3 we will need to remount him and repeat coarse scans in the two chi positions.**

## Realignment after chi motor re-engagement

The alignment of the ring normal with the beam has not been called into question by the chi motor slippage, so that alignment did not need to be repeated. To remeasure the chi reference position, we advanced spinz such that the leveling laser just grazed the top edge of the mounting bar.

spinz = -4.8 when JD70 diamond at center position

spinz = +3.2 when leveling laser grazes top edge of bar
chi = 99.0 ± 0.1 degrees with bar horizontal
reference chi = 9.0 ± 0.1 degrees

## JD70-2_study2

| Filename | Orientation | Steps | Step size (degrees) | Exposure (s) |
|---|---|---|---|---|
| JD70-2_study2_scan001 | (2,2,0) | 125 | 0.0002 | 10 |
| JD70-2_study2_scan002 | (2,-2,0) | 100 | 0.0002 | 10 |
| JD70-2_study2_scan003 | (2,2,0) | 150 | 0.0002 | 10 |
| JD70-2_study2_scan004 | (2,-2,0) | 100 | 0.0002 | 10 |

| JD70-2 | Orientation | Tth | Th | Chi | Phi | Time |
|---|---|---|---|---|---|---|
| scan001 | (2,2,0) | 38.552 | 18.211-18.236 | 48.8 | 90.25 | 5:00 |
| scan002 | (2,-2,0) | 38.552 | 16.804-16.824 | 58.8 | 270.25 | 5:50 |
| scan003 | (2,2,0) | 38.552 | 20.176-20.206 | 148.5 | 270.25 | 6:24 |
| scan004 | (2,-2,0) | 38.552 | 21.615-21.635 | 138.5 | 90.25 | 7:13 |

## SI45-S90 mounting and imaging

Spinz had to change to accommodate the smaller diamond. It changed from -4.8 to -3.4.

Due to the cut of this diamond, we were not able to reach the (-2,-2,0) orientation by rotating in phi. Thus, for scan 2 the diamond mounting bar was flipped over in order to obtain this orientation. spinz had to be adjusted to keep beam on diamond; spinz was changed from -3.4 to 8.2. This adjustment of the diamond mounting bar is only needed to reach the (-2,-2,0) orientation. After scan 2 was completed, the diamond mounting bar orientation was changed back, and spinz was put back to its original value of -3.4.

| Filename | Orientation | Steps | Step size (degrees) | Exposure (s) |
|---|---|---|---|---|

| | | | | |
|---|---|---|---|---|
| SI45-S90_study1_scan001 | (2,2,0) | 50 | 0.0002 | 10 |
| SI45-S90_study1_scan002 | (-2,-2,0) | 50 | 0.0002 | 10 |
| SI45-S90_study1_scan003 | (-2,2,0) | 50 | 0.0002 | 10 |
| SI45-S90_study1_scan004* | (2,-2,0) | 50 | 0.0002 | 10 |
| SI45-S90_study1_scan005 | (2,-2,0) | 50 | 0.0002 | 10 |

*ad_on was never initialized before the scan was started and so the data was not saved.

| **SI45-S90_study1*** | **Orientation** | **Tth** | **Th** | **Chi** | **Phi** | **Time** |
|---|---|---|---|---|---|---|
| scan001 | (2,2,0) | 38.552 | 15.840-15.850 | 101.0 | 90.25 | 8:09 |
| scan002 | (-2,-2,0) | 38.552 | 22.767-22.777 | 101.0 | 90.25 | 9:06 |
| scan003 | (-2,2,0) | 38.552 | 20.995-21.005 | 11.0 | 90.25 | 9:44 |
| scan005 | (2,-2,0) | 38.546 | 21.001-21.011 | 5.8 | 270.25 | 10:42 |

*we discovered a missing region of the diamond, we believe it to be a twin crystal

## JD70-3 study 2

Diamond JD70-3 was mounted again and we began a second study, this time with the chi motor properly engaged. The  first scan was finished by the time we got back from lunch, Nathan and I moved chi by 90° from 55.55 to 141.55 and began searching for the peak. We saw a peak as soon as the beam came on, good luck so far.

| **Filename** | **Orientation** | **Steps** | **Step size (degrees)** | **Exposure (s)** |
|---|---|---|---|---|
| JD70-3_study2_scan001 | (2,2,0) | 450 | 0.0004 | 10 |
| JD70-3_study2_scan002 | (2,-2,0) | 450 | 0.0004 | 10 |

| JD70-3 | Orientation | Tth | Th | Chi | Phi | Time |
|---|---|---|---|---|---|---|
| scan001 | (2,2,0) | 38.546 | 21.275-21.455 | 51.55 | 90.25 | noonish |
| scan002 | (2,-2,0) | 39.046 | 21.345-21.525 | 140.85 | 90.25 | 13:24 |

## J2a100_study1

The diamond mounting bar was flipped over as in SI45-S90_study1 for scan2; this time in order to reach the (-2,2,0) orientation. Moved spinz to 8.2 to accommodate this setting for scan 2. After scan2 we moved spinz back to -3.4. Something interesting came up as we were trying to determine the range for the (2,2,0) scan of J2a100_study1. It took a much larger range to cover the entire rocking curve of the diamond, over a factor of 2 larger than the previous scans. We have two hypothesis on this, one is that Brendan adjusted the I2 knob to maximize the intensity seen on the diamond. It could be that this also broadened the range that we can see diffraction over, increasing the total range of the scan. Another is that the hutch light was on during the range setting of the past two scans and that may have affected our sensitivity to the end points. The former seems more convincing to me but, we'll be able to tell when we analyze the data. Most importantly, these diamond have a narrow rocking curve and we more than covered the peak intensity  with the range set by the last two scans, even if the ends were a bit short. We just checked through a few of the end images of scans 1 and 2 of this diamond. The scans do start with some illuminated diamond in the frame, but it does not look too bad.

| Filename | Orientation | Steps | Step size (degrees) | Exposure (s) |
|---|---|---|---|---|
| J2a100_study1_ scan001 | (2,2,0) | 215 | 0.0004 | 10 |
| J2a100_study1_ scan002 | (-2,-2,0) | 215 | 0.0004 | 10 |
| J2a100_study1_ scan003 | (-2,2,0) | 525 | 0.0004 | 10 |
| J2a100_study1_ scan004 | (2,-2,0) | 550 | 0.0004 | 10 |

| J2a100 | Orientation | Tth | Th | Chi | Phi | Time |
|---|---|---|---|---|---|---|

| | | | | | | |
|---|---|---|---|---|---|---|
| scan001 | (2,2,0) | 38.446 | 17.330-17.416 | 101.65 | 90.25 | 15:45 |
| scan002 | (-2,-2,0) | 38.746 | 21.206-21.292 | 101.65 | 90.25 | 16:35 |
| scan003 | (-2,2,0) | 38.396 | 17.665-17.875 | 11.15 | 90.25 | 18:01 |
| scan004 | (2,-2,0) | 38.796 | 17.656-17.876 | 5.55 | 270.25 | 20:01 |

## J1a50_study1

**2/23/2015 AB, RJ**
J2a100 was removed from the hoop and brought to the chem room where it was unmounted and placed with the other diamonds. J1a50 was then brought to the hutch and attached to the hoop, making sure to push the bar towards the glass when tightening the bolts. This diamond needs to be square-shaped when finding the (2,2,0) planes; the bottom of the diamond must be horizontal.

The rocking curve is very sharp for this diamond and will not take long to image.

| Filename | Orientation | Steps | Step size (degrees) | Exposure (s) |
|---|---|---|---|---|
| J1a50_study1 _scan001 | (2,2,0) | 50 | 0.0002 | 10 |
| J1a50_study1 _scan002 | (2,-2,0) | 50 | 0.0002 | 10 |
| J1a50_study1 _scan003 | (-2,2,0) | 50 | 0.0002 | 10 |
| J1a50_study1 _scan004 | (-2,-2,0) | 50 | 0.0002 | 10 |

| J1a50 | Orientation | Tth | Th | Chi | Phi | Time |
|---|---|---|---|---|---|---|
| scan001 | (2,2,0) | 37.996 | 19.196-19.206 | 65.05 | 90.025 | 23:06 |
| scan002 | (2,-2,0) | 38.396 | 18.558-18.568 | 42.05 | 270.25 | 23:39 |
| scan003 | (2,2,0) | 38.396 | 19.215-19.225 | 132.05 | 270.25 | 23:57 |
| scan004 | (2,-2,0) | 38.396 | 19.872-19.882 | 155.05 | 90.25 | 12:28 |

### Consistency check of the glass slide alignment

After imaging all of the diamonds and before the hoop is removed a consistency check of the glass slide alignment was made. This is to see if any changes occurred to the alignment during the imaging process.

The goniometer was brought to theta = 0 and chi = 99, bringing it back to the alignment position. Spinz was moved to 3.2 so that the horizontal bar could be checked with the laser level. At chi = 99.1 the laser looked level which is within our error for the chi reference value. This also means we can remove and replace the horizontal bar well enough that our level is within our uncertainty.

Spinz was brought to 24.575 to put the glass slide back into the alignment position and the laser was turned on. An initial laser spot was marked and then the chi angle was rotated by 180 degrees with the new spot marked. There was a horizontal distance of 7.5mm and a vertical distance of 2.5mm between the dots. This process was repeated with the laser reflecting off of a separate piece of the glass about a half centimeter away in case the glass surface was not flat. The same dimensions were found. We measured the distance from the spot to the glass to be 140mm. The discrepancy in the phi angle corresponds to about 1mrad which is acceptable.

## Testing Yuri’s sample (ANL3x5)

### Mounting

Yuri’s sample was mounted in Ken’s small hoop. It was decided to use this hoop because it fits with the current stage mount. With the goniometer having changed we did not know if our large hoop would fit. The diamond was positioned such that when the mounting stage is vertical the diamond is square shaped, the bottom edge is horizontal. To bring the diamond vertically into the center of rotation, spinz was adjusted to -6.925. The phi motor was disengaged and the x and y translations were adjusted to bring the diamond into the center of rotation. A video camera was used to view the diamond and crosshairs were placed to go through the center of the diamond in one viewing angle. Phi was then rocked by 40 degrees and the view was compared. After multiple iterations of rocking and adjusting the translations the diamond is now in the center of rotation.

### Yuri’s diamond rocking curve (ANL3x5_study1)

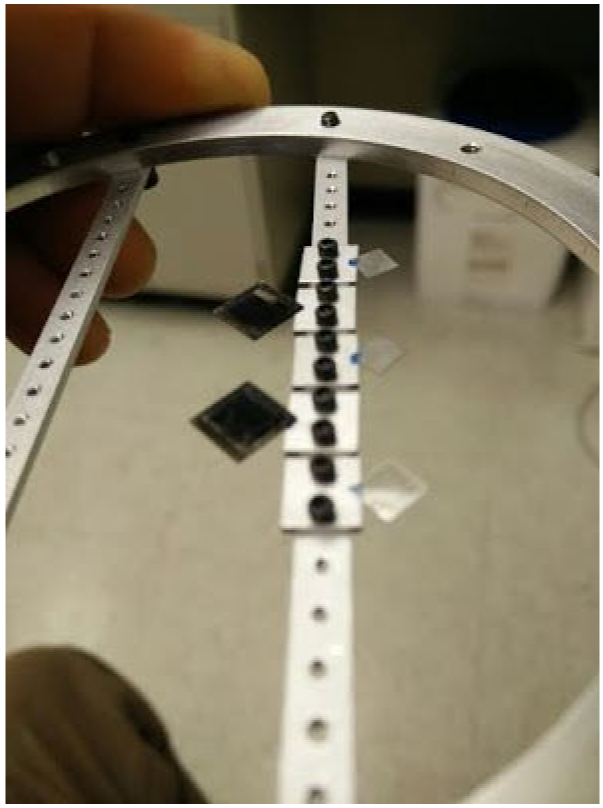
ANL3x5 is oriented like the JD70 diamonds. Because the diamond was mounted such that the phi axis is the same as the (2,2,0) axis we will need to rotated to 4 different chi’s to see all possible reflections.

The chi motor will not move beyond 190. We positioned chi at 99.4 (mount vertical) and released the motor and rotated the diamond into an upside-down position. Chi has been rezeroed.

| Filename | Orientation | Steps | Step size (degrees) | Exposure (s) |
|---|---|---|---|---|
| ANL3x5_study1_scan001 | (2,2,0) | 70 | 0.0002 | 10 |
| ANL3x5_study1_scan002 | (-2,2,0) | 50 | 0.0002 | 10 |
| ANL3x5_study1_scan003 | (2,-2,0) | 50 | 0.0002 | 10 |
| ANL3x5_study1_scan004 | (-2,-2,0) | 50 | 0.0002 | 10 |

| ANL3x5 | Orientation | Tth | Th | Chi | Phi | Time |
|---|---|---|---|---|---|---|
| scan001 | (2,2,0) | 38.396 | 18.694-18.708 | 99.4 | 90.25 | 2:31 |
| scan002 | (-2,2,0) | 38.396 | 19.246-19.256 | 9.4 | 90.25 | 2:54 |
| scan003 | (2,-2,0) | 38.396 | 19.233-19.243 | 189.4 | 90.25 | 3:32 |
| scan004 | (-2,-2,0) | 38.396 | 19.771-19.781 | 279.4 | 90.25 | 3:55 |

After the scans Yuri's diamond was placed in the fume hood in the chemical room near the other diamonds that have tabs. Ken's diamond was placed back into the small mount and returned to it's home in the light gray C1 cabinet, second shelf from the top on the right side.



Diamonds being prepared for transport to JLab. Note the bar is inverted such that the plane of the diamonds lies inside the cylinder subtended by the ring, and the ring is sandwiched between two plates of plexiglass.

## Conversion of rocking curve angles to crystal offsets

*Sunday, February 22, 16:50 [rtj]*
After fixing the chi motor disengagement problem, we aligned the diamond mounting ring to make the ring coordinate system coincide with the X-ray beam coordinate system. The X-ray beam coordinate system has x aligned with the axis of the goniometer theta stage and pointing toward the back wall of the hutch, y in the vertical plane containing the beam and pointing along the [220] direction when at the rocking curve maximum, and zhat = xhat cross yhat. The motor settings that achieve this are:

th = 19.20 ± 0.02 degrees
chi = 9.10 ± 0.10 degrees
phi = 90.25 ± 0.05 degrees

The diamond crystal offsets in the ring coordinate system are $\delta_x$, $\delta_y$, and $\chi_c$. One measured rocking curve peak gives three measured coordinates: θ, φ, and χ, but of these one is fixed by the alignment condition (φ) so it takes two independent measurements to uniquely determine the crystal offsets. These two measurements must be at different χ's because two measurements at the same χ and different φ only provide redundant information about χ. In cases where we took 4 measurements, we can form 4 pairs of orthogonal vectors to independently extract the offsets 4 different ways and look for consistency.

I wrote [a python script]() to accept the goniometer settings at the rocking curve maxima and compute the crystal offsets from these. The results are as follows.

Test of crystal_offsets calculator with JD70-2 from chess-2-2015:
 delta_x, delta_y, chi_c = 0.0141806609029 0.0394494147526 -39.5251537839
 delta_x, delta_y, chi_c = 0.0140324890906 0.0397954694915 -40.3935842105
 delta_x, delta_y, chi_c = 0.0141669508808 0.0389581575168 -39.9760285739
 delta_x, delta_y, chi_c = 0.0140407226328 0.0403175183741 -39.9768264904

Test of crystal_offsets calculator with SI45-S90 from chess-2-2015:
 delta_x, delta_y, chi_c = -0.0924466551452 0.00306242515430 -2.51634472622
 delta_x, delta_y, chi_c = -0.0910412330502 0.00211521917459 -1.81879381444
 delta_x, delta_y, chi_c = -0.0909830241798 0.00211516659456 -1.81484363260
 delta_x, delta_y, chi_c = -0.0925062062008 0.00306263596926 -2.51102611896

Test of crystal_offsets calculator with JD70-3 from chess-2-2015:
 delta_x, delta_y, chi_c = 0.0555090394862 -0.00226379379488 -42.0637956704

Test of crystal_offsets calculator with J2a100 from chess-2-2015:
 delta_x, delta_y, chi_c = -0.0106666579759 -0.00238477061173 -3.05559121911
 delta_x, delta_y, chi_c = -0.0096842675041 -0.00187779806434 -2.30527990118
 delta_x, delta_y, chi_c = -0.0096987704053 -0.00187776487824 -2.30404125307
 delta_x, delta_y, chi_c = -0.0106518082074 -0.00238485627804 -3.05380543156

Test of crystal_offsets calculator with J1a50 from chess-2-2015:
 delta_x, delta_y, chi_c = 0.0102805220675 0.00907463781033 34.0539182057
 delta_x, delta_y, chi_c = 0.0096628447327 0.00869250348022 32.9548229958
 delta_x, delta_y, chi_c = 0.0098503843131 0.00848177043195 33.5035486408
 delta_x, delta_y, chi_c = 0.0101004163280 0.00930188425789 33.5036777525