

13BMC X-ray diffraction user manual (Nov 28, 2018)

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DAC alignment

1. Put the DAC on the sample holder. **Make sure the gasket is as close to the center of the holder as possible.** Then put the DAC on the experimental stage
2. Rotate kphi to 120 degs, and search for sample chamber with the microscope

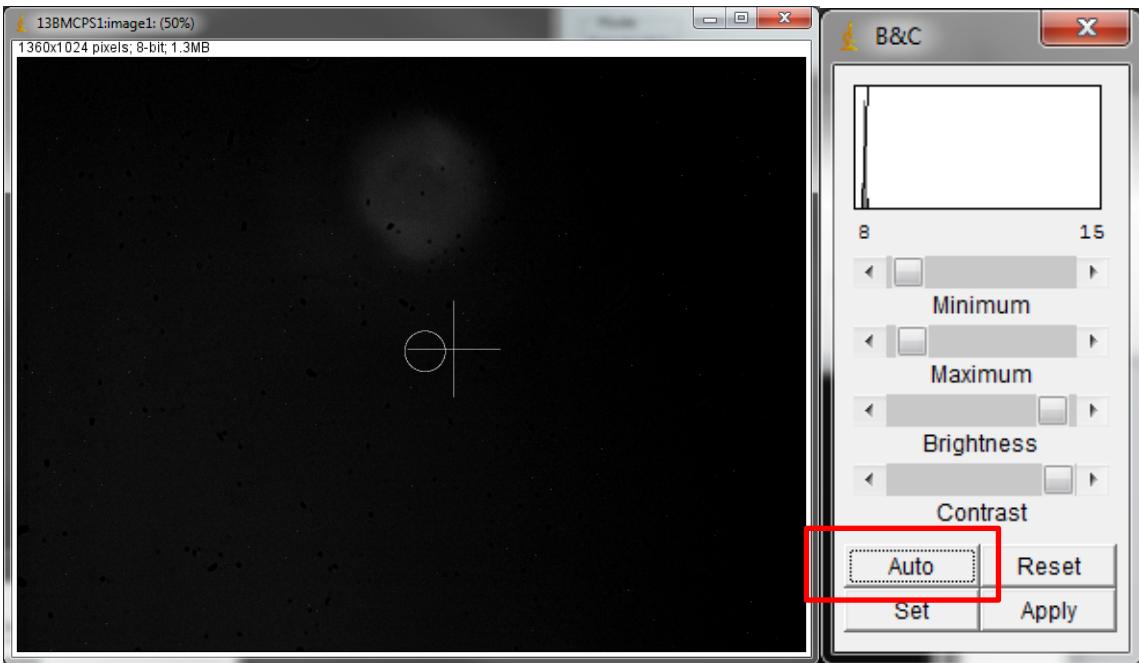


NAV-ZOOM controls the zoom of the microscope. For DAC experiments, in the end we will put the NAV-ZOOM to 0.1 (max magnification, smallest field of view). To coarsely locate the sample, put the NAV-ZOOM to 0.6 or 1.1 (larger field of view).

In the current geometry ($k\phi = 120$), sample Y brings sample up and down in the camera, sample Z moves sample horizontally, and sample X changes the focus of the image. DAC_View_Z also changes the focus, but should be fixed to 1 (if you use Boehler-Almax diamond, ~ 1.6 mm thick) or 1.5 (standard diamond, > 2 mm thick) at this stage. Change sample X = 0, Y=0, Z=-4 (rough initial estimate), move around y and Z with zero 0.1 step size to find the gasket hole. Move sample x to have the most clear image. The crosshair on the screen shows the X-ray position for the previous user (should be close to your DAC), and the circle shows the 532 nm laser position for Ruby pressure determination.

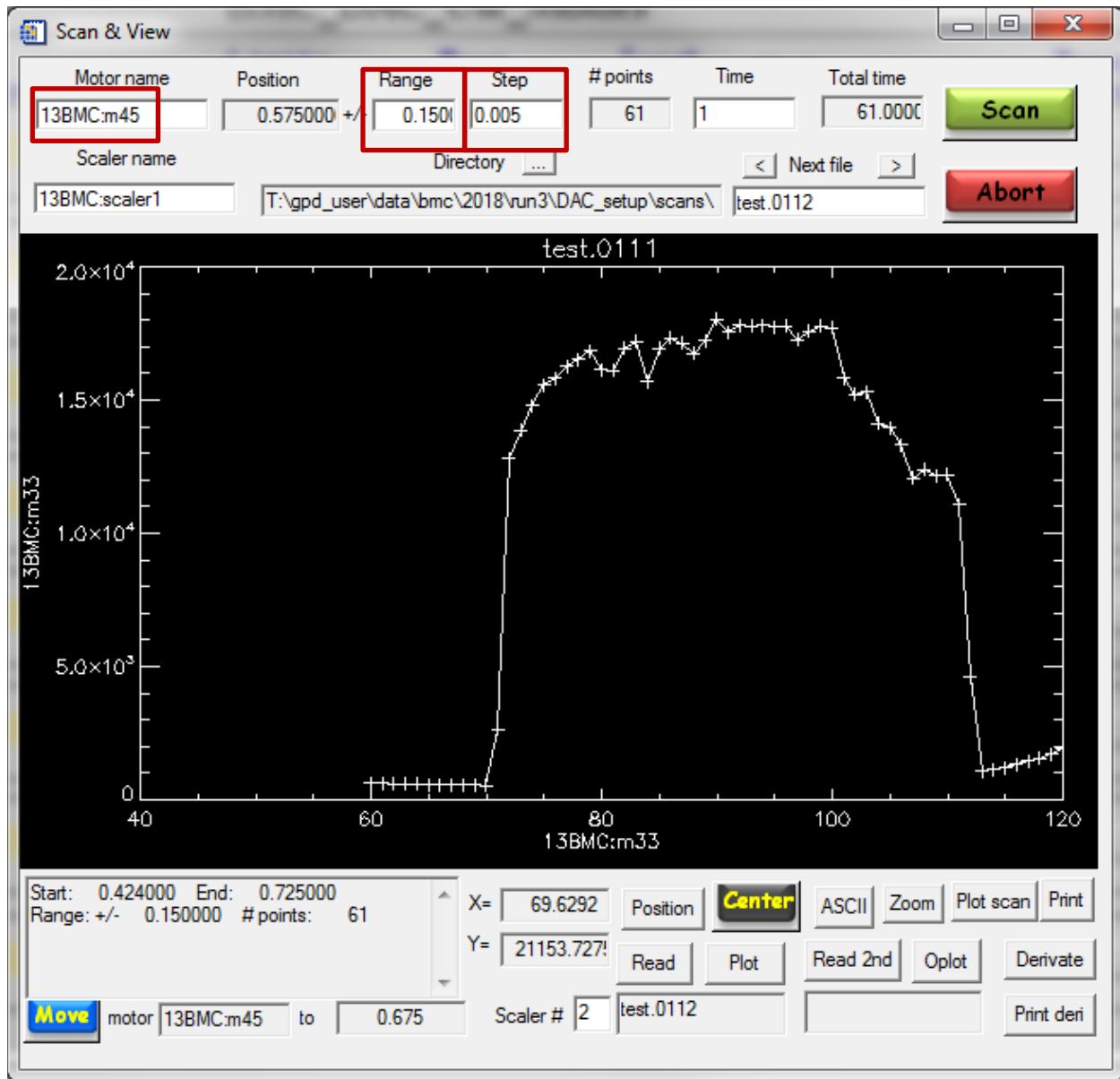


Zoom in: put NAV_ZOOM to 0.1, then use "auto" to change contrast. You can press "auto" multiple times. If the "B&C" window is gone, click on the image, then press Ctrl+Shift+C.



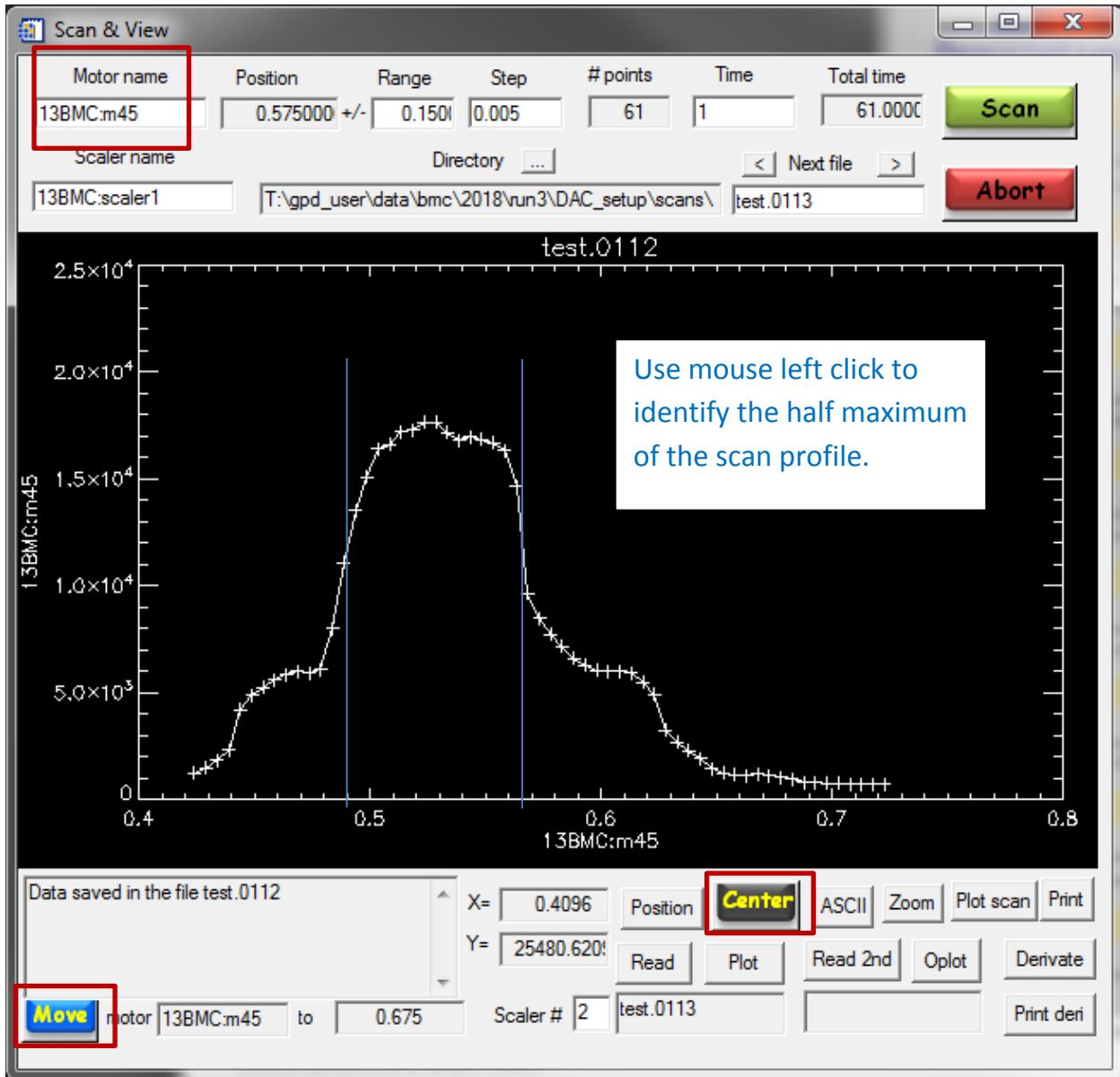
Use sample Y & Z to align the gasket hole center to the crosshair, use sample X to get the gasket focus. Search the station C, then close the hutch gate.

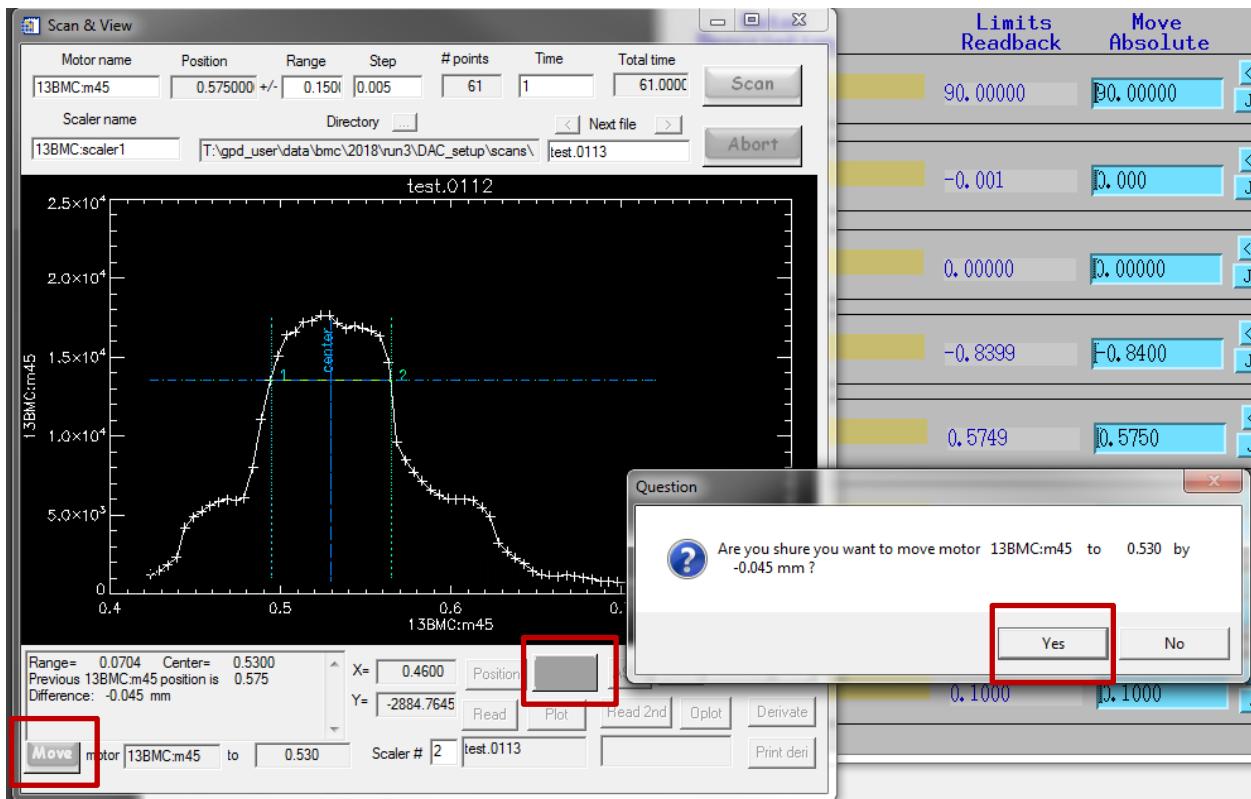
3. Scan sample chamber to refine the sample position. In this step there will be 5 scans. First scan sample Y to align sample Y (Scan #1), then scan sample Z to align sample Z (Scan #2), then scan sample Y 3 times at different angles to align sample X (Scan #3, #4, #5).



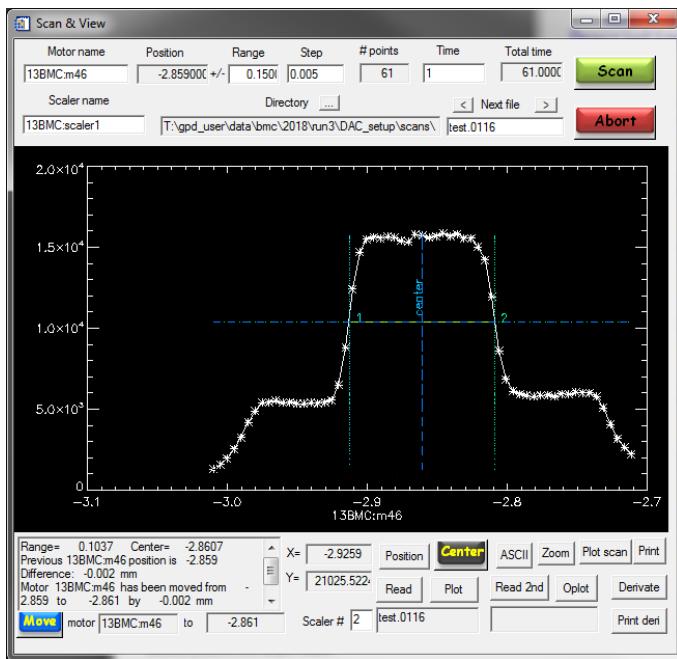
For the ScanW program, you need to press "enter" at the Motor name to update the values. One common issue is, when the "range" is out of the soft limit of the motor, the ScanW software will throw out error. This happens often when you switch from rotation angle scan (m33) to translation scan (m45/46), and click OK acknowledge the error messages. Change step size first (or you cannot change the scan range, usually for DAC use step size of 0.005), then set scan range to gasket diameter (0.1 ~ 0.2).

Scan #1: Put kphi to 90, scan m45. Find the center of gasket by clicking “center” first, left click on the half maximum position on both sides of the scan profile, then press “move”. Acknowledge the move by clicking “Yes” on the message that jumps out.

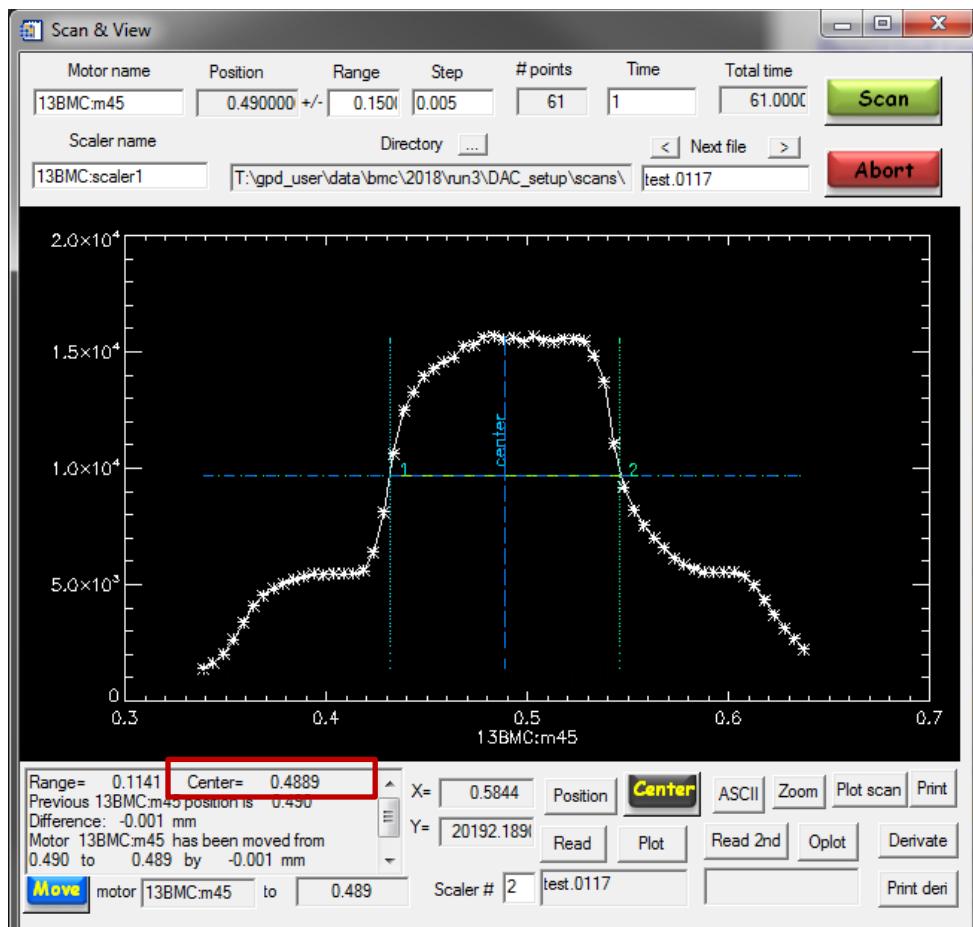




Scan #2: Scan M46, center and move



Scan #3: Scan M45 center and move, put the center value to the DAC correction program



DAC correction

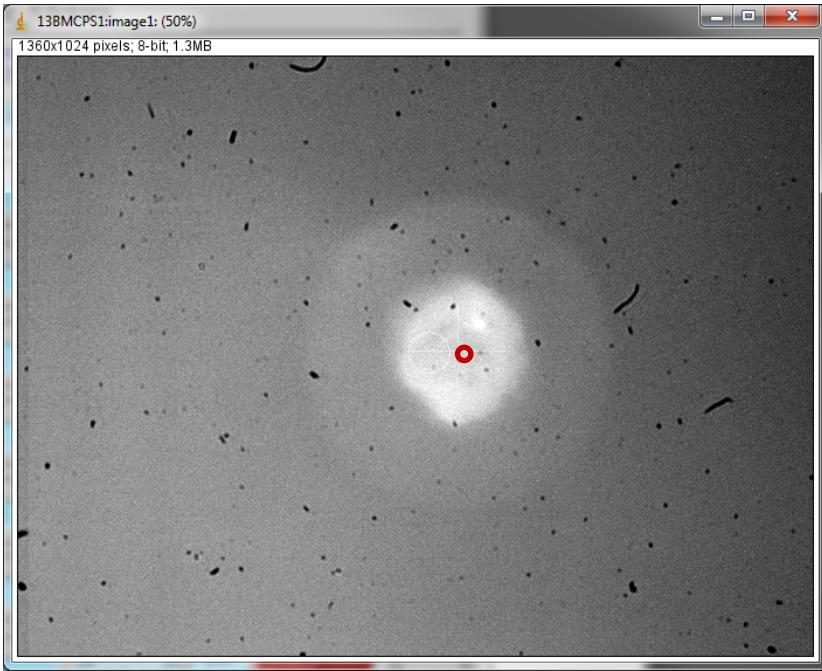
$\Delta k\phi_i$ (m33)	10
Center of Sample_Y (m45) scan at $k\phi_i = 90$	0.4889
Center of Sample_Y (m45) scan at $k\phi_i = 90 + \Delta k\phi_i$	0.725
Center of Sample_Y (m45) scan at $k\phi_i = 90 - \Delta k\phi_i$	0.675

Make correction Move sample_X from -0.2598999999999993 by -0.14396 to -0.40386

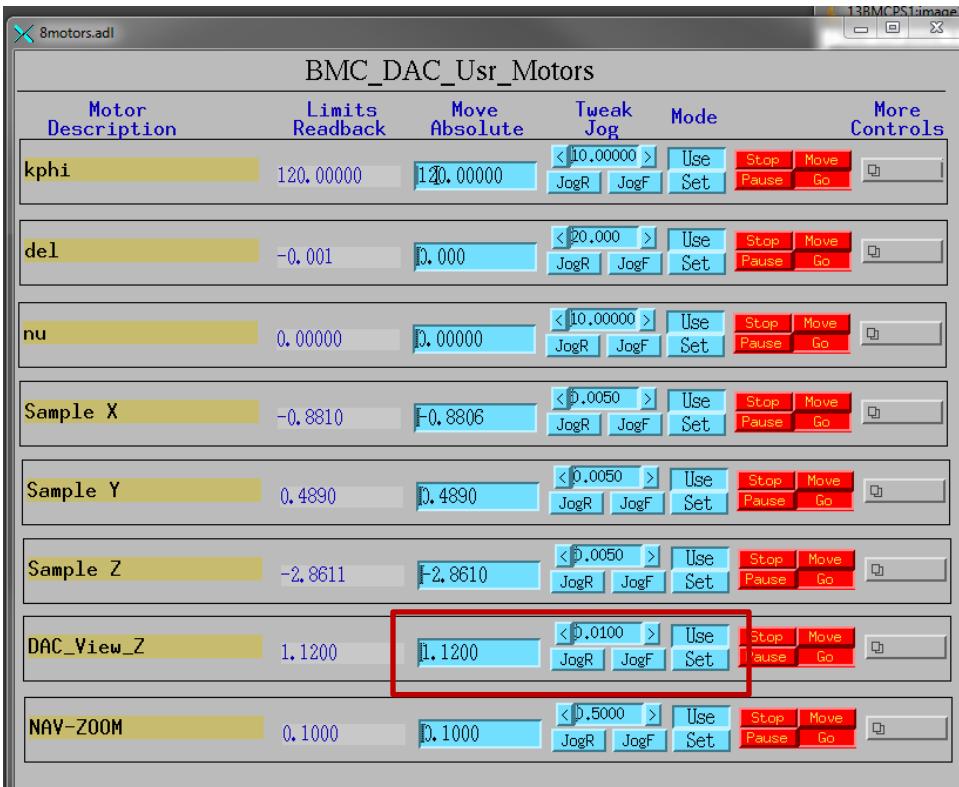
Scan #4: Rotate $k\phi_i$ to $90 + \Delta k\phi_i$, scan m45, find center **do not move**, put the center value to DAC correction box 3, $k\phi_i = 90 + \Delta k\phi_i$

Scan #5: Rotate $k\phi_i$ to $90 - \Delta k\phi_i$, scan m45, find center **do not move**, put the center value to DAC correction box 4, $k\phi_i = 90 - \Delta k\phi_i$

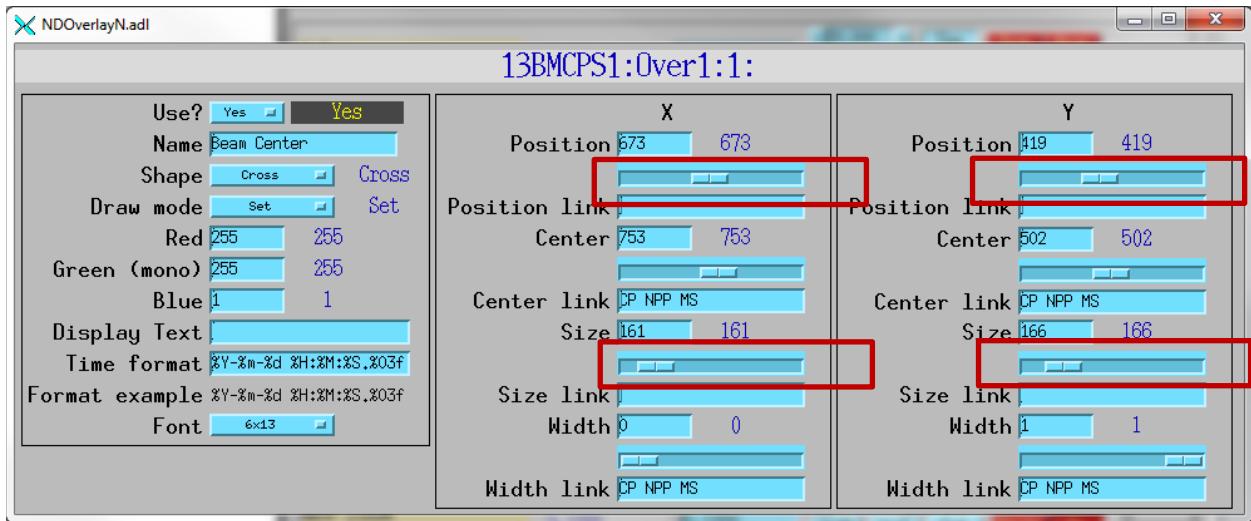
Press “**make correction**” on DAC correction program. Then the gasket hole center is aligned to X-ray.
 Rotate kphi to 120 , now X-ray should be at the center of the gasket hole (**red circle in the figure below**)



4. Now the image is blurry, move DAC_View_Z to focus the image. DAC_View_Z tweak size should be at least 0.02.

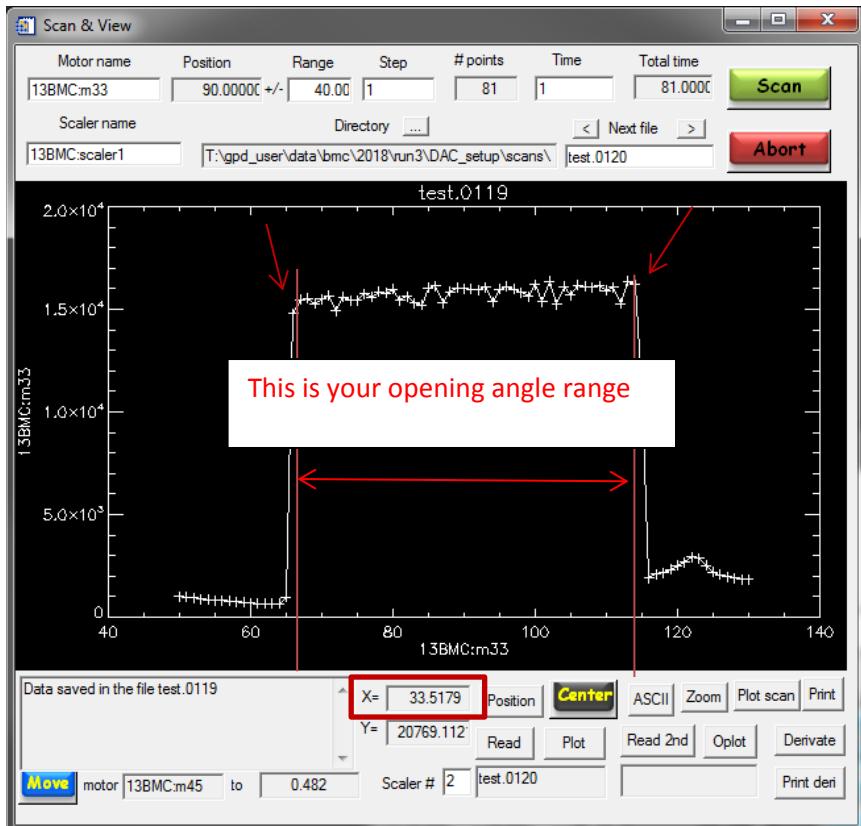


Put the crosshair to the center of the gasket hole, change the bar of position and size at **NDOverlayN.adl**, now the crosshair marks the x-ray position

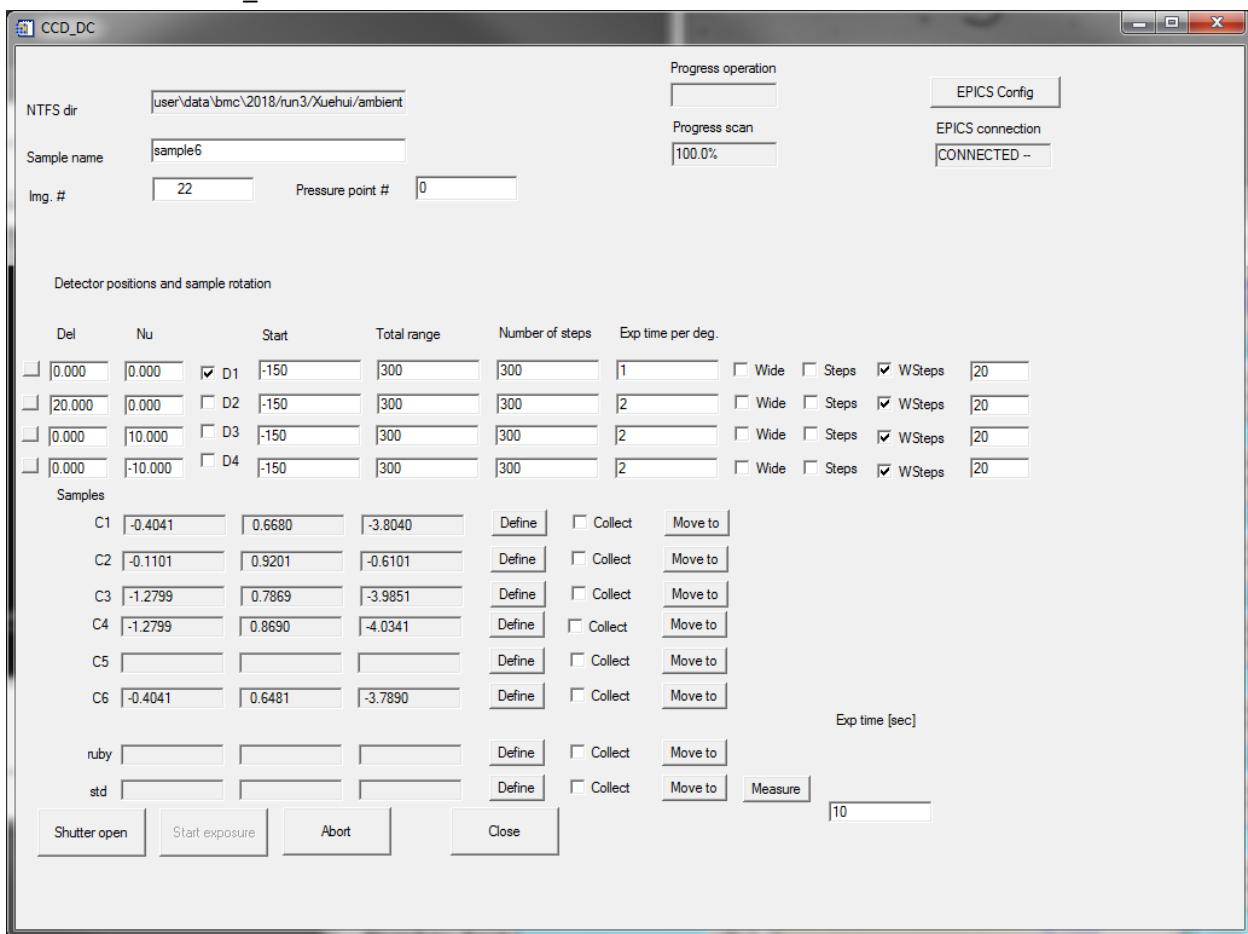


5-I. Set up single-crystal data collection. **For powder see Section 5-II**

Determine opening angle: move kphi to 90, scan m33, range 30 (symmetric DAC) or 40 (BX90 or large opening DAC), step=1. Use mouse cursor to determine the start and stop of the opening angle, in this case the start is **66**. And the stop is **114**, so the range is **114-66=48**.

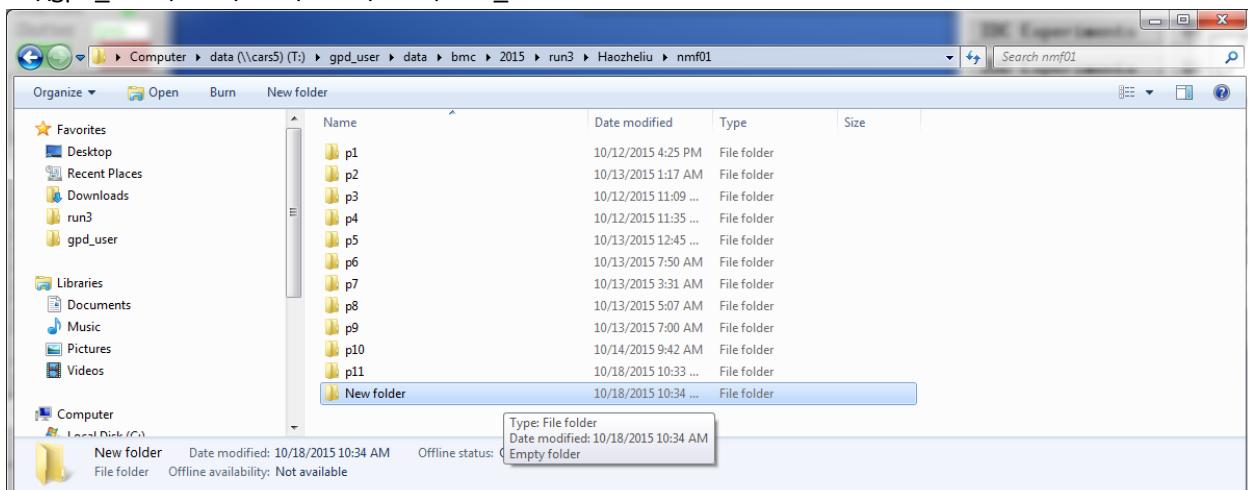


The software is CCD_DC.

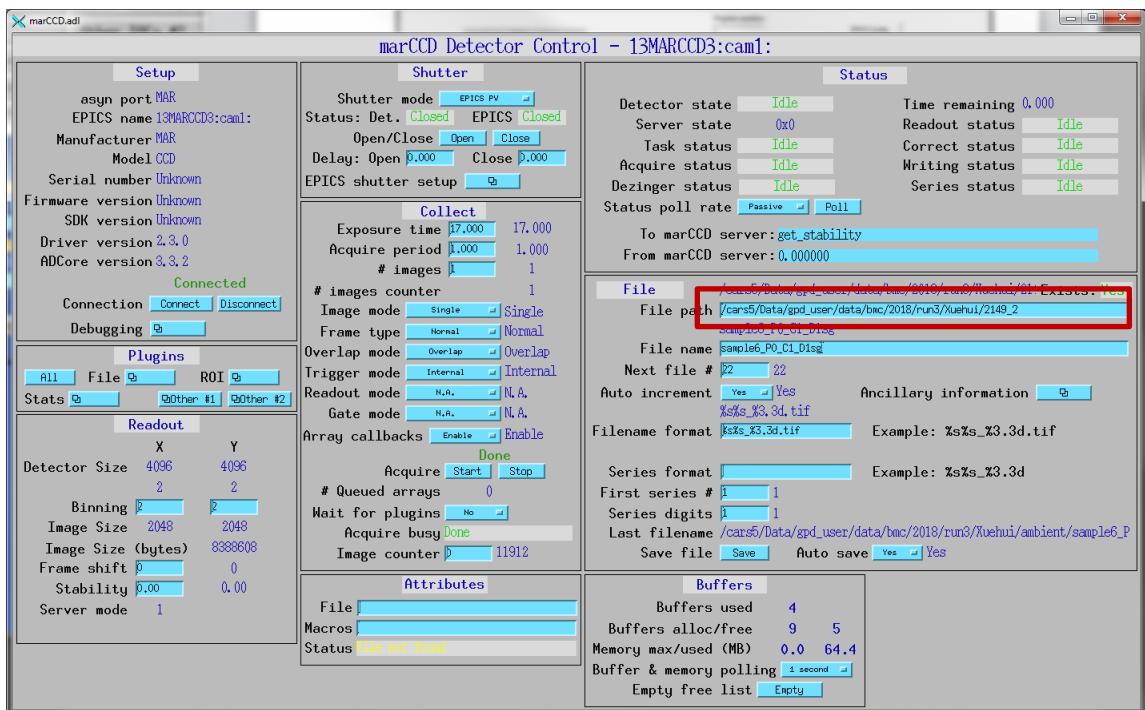


First make a new folder: Make a new folder for the data in

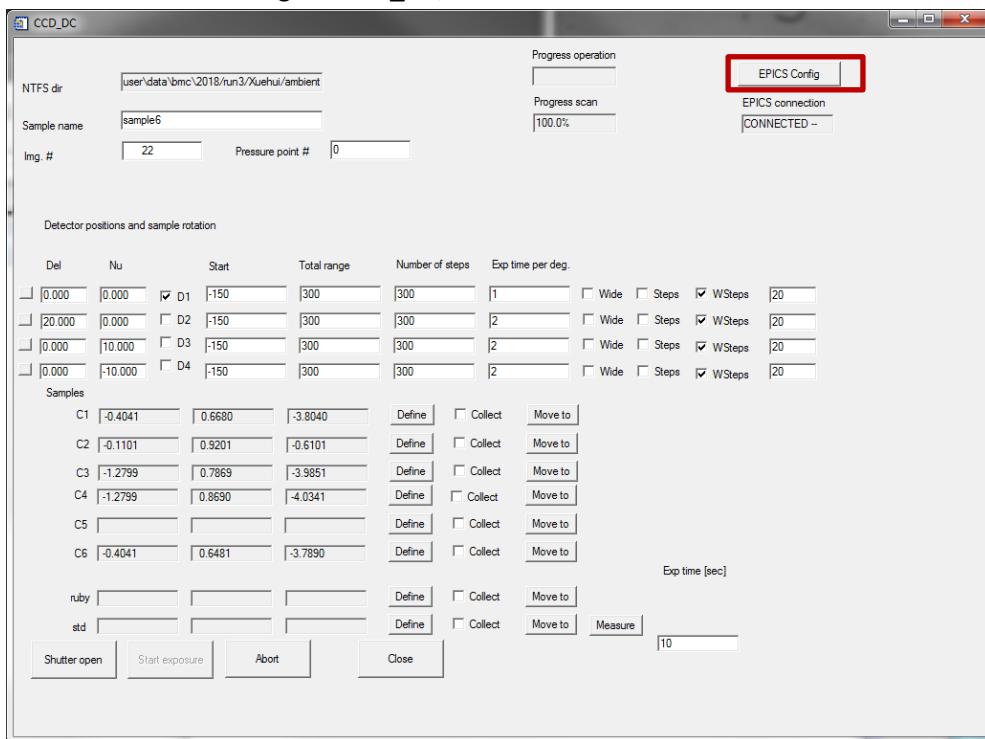
T:\gpd_user\data\bmc\20XX\runY\Your_folder



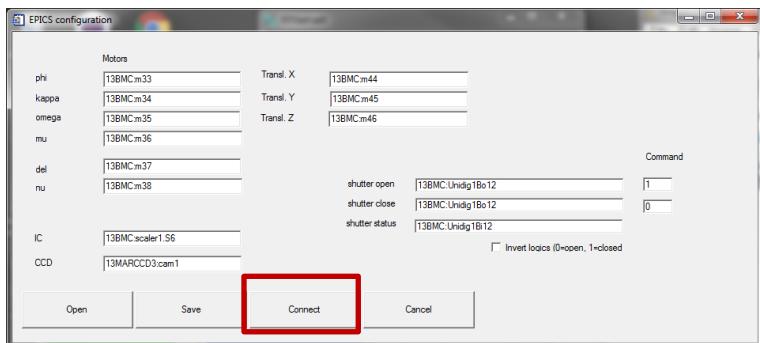
In the marccd.adl window, change the folder to your new folder.



Then click "EPICS Config" at CCD_DC,



Click connect



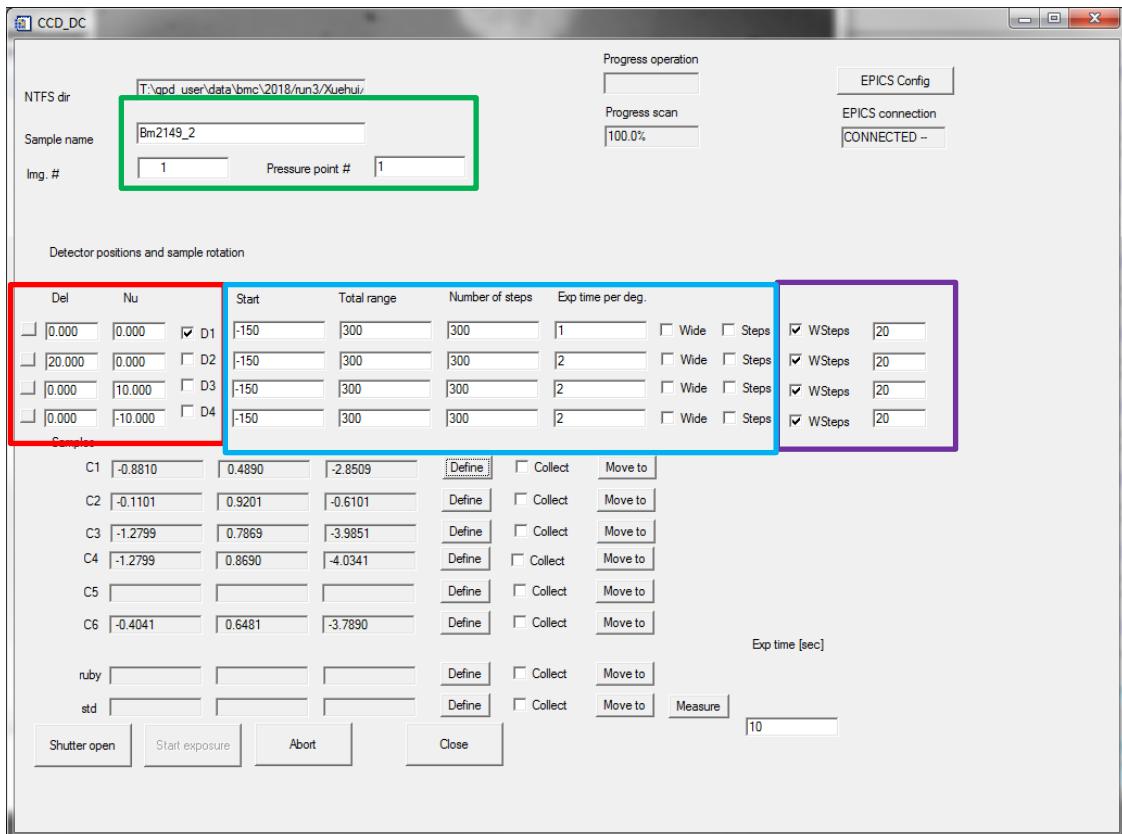
What do the boxes in CCD_DC mean?

Sample name, starting image number, pressure number

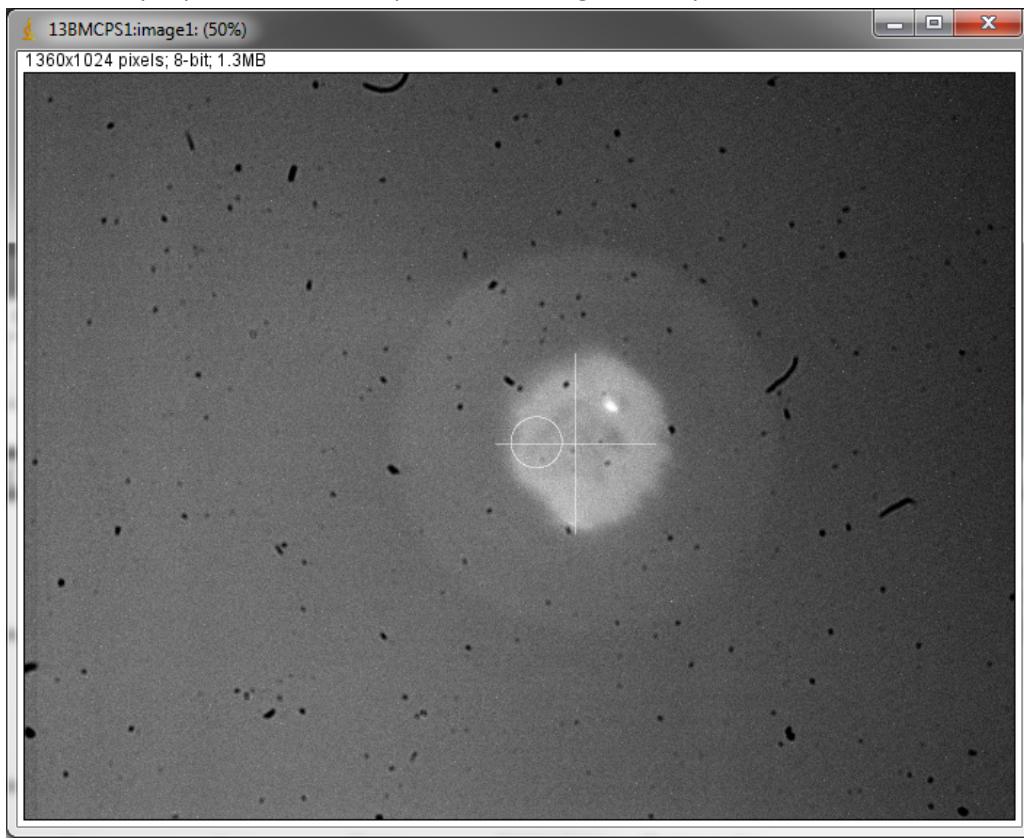
Start phi angle, rotation range, total number of steps (only affects step scans). Each step scan will cover a range of **Rotation Range/Number of steps**, experimental time per deg (Total wide angle exposure time will be **Ttot=Exp Time Per Deg * Rotation Range**, each step scan exposure time will be **Ttot/Number of steps**) Usually we need both wide and step scans

Detector position: Del moves detector up and down, and Nu moves detector left and right.

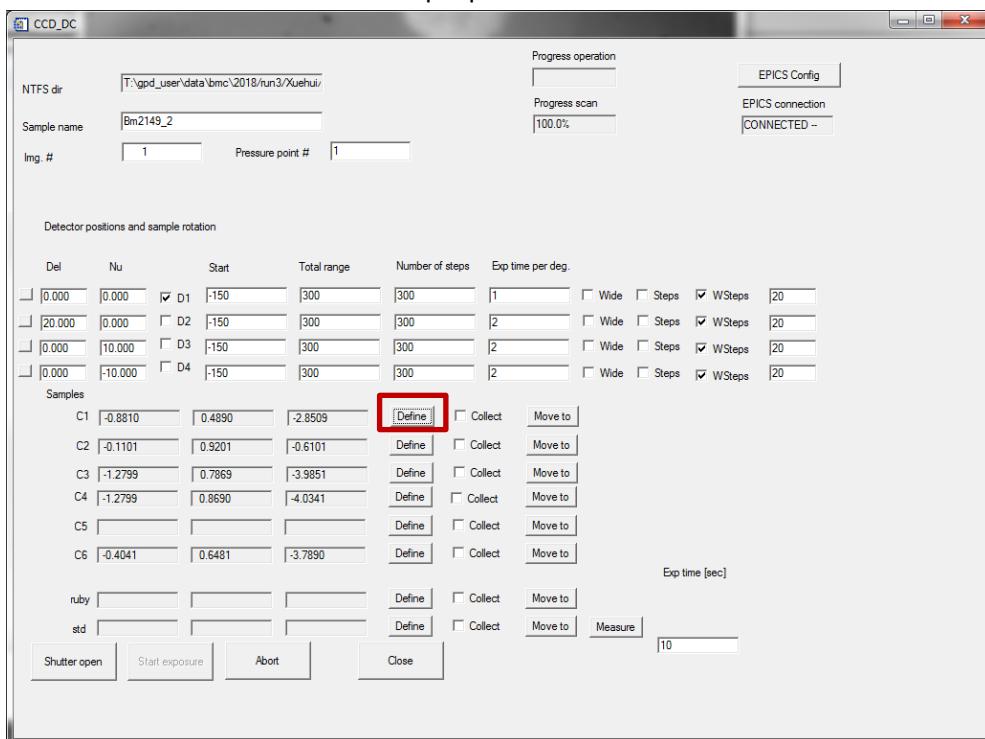
Wide segments for better peak intensity (need for structural refinement). **Segment number = round(Total range/15)**. E.g., total range = 72, then round(72/15) = 5.



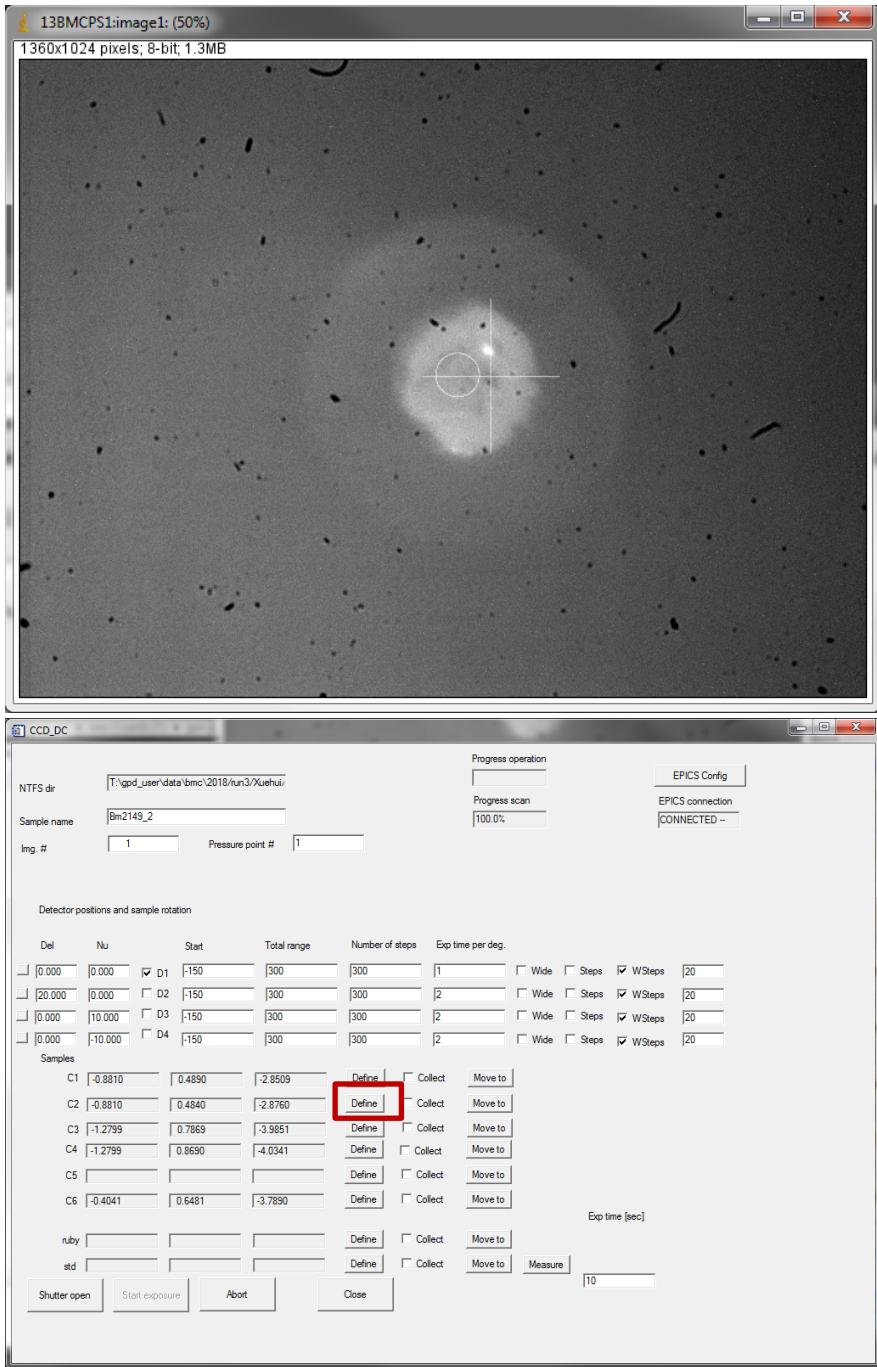
Define sample position: Rotate kphi to 120, bring the sample to cross hair



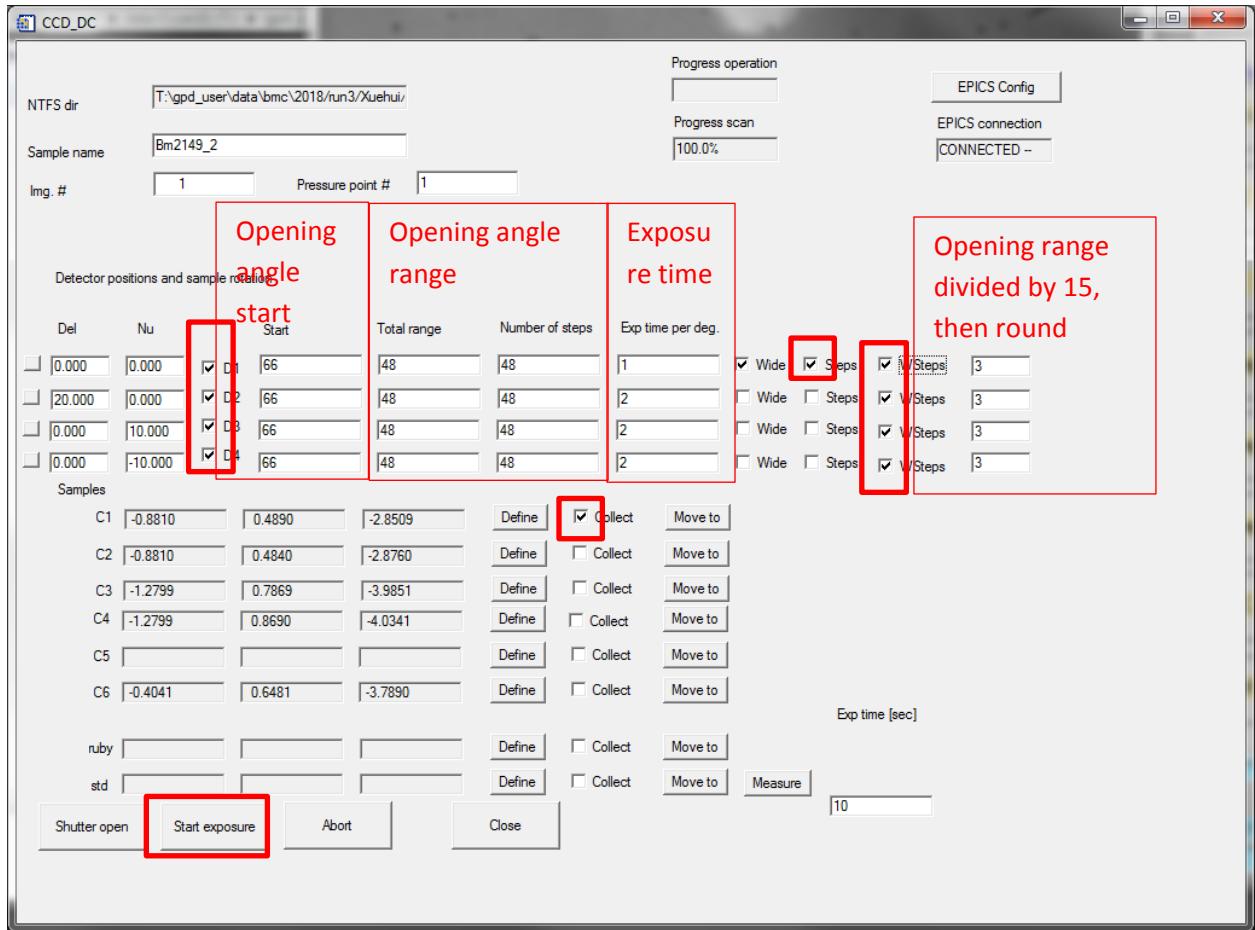
Then click define to record the sample position.



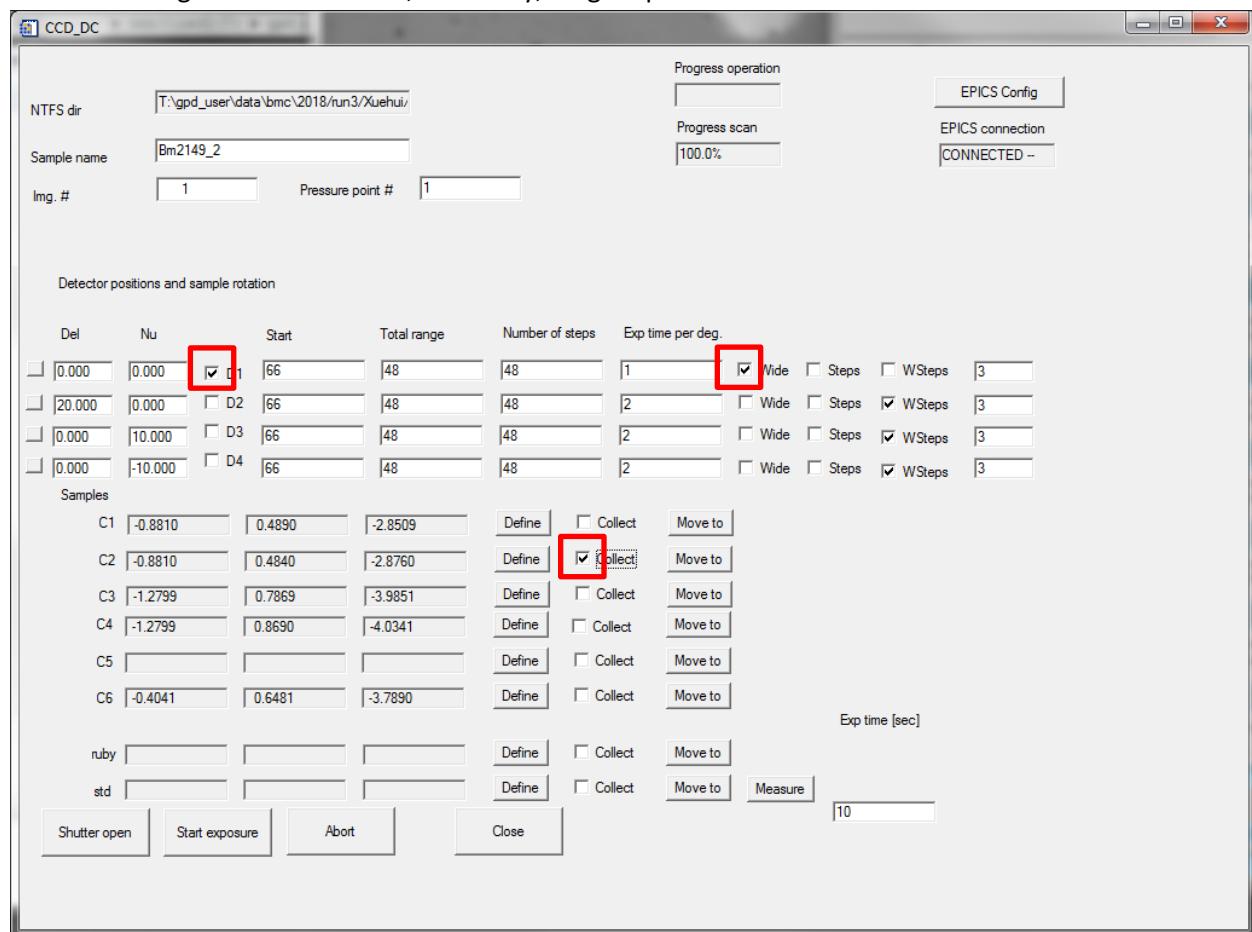
Do the same thing for gold.



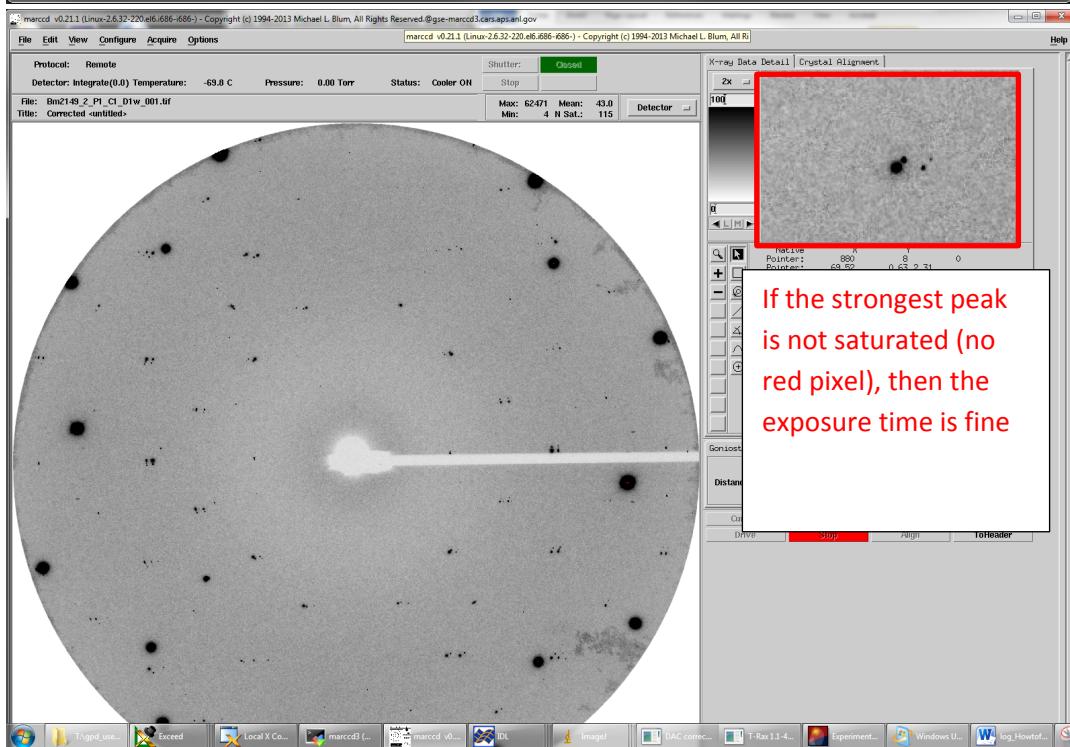
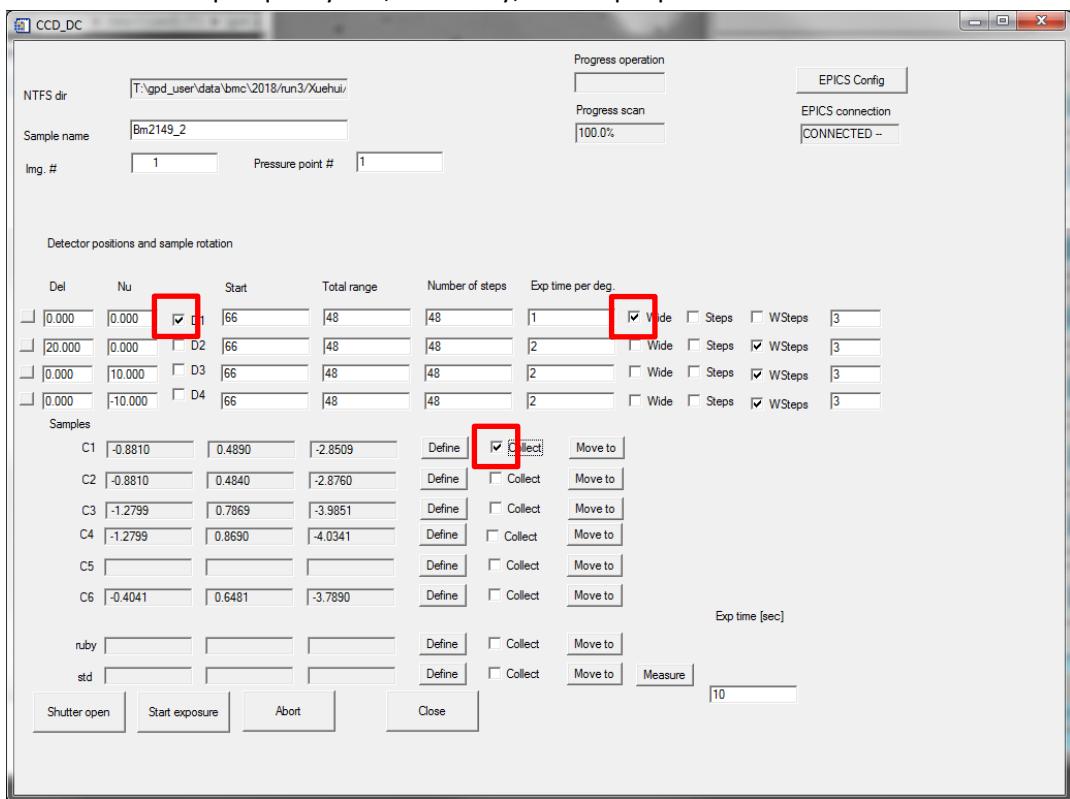
Set up collection parameters. To collect single crystal diffraction on sample and do structural refinements, we need step images for D1 position to index the peaks, reconstruct the reciprocal lattice, and calculate UB matrix, and we need the wide-segments for intensities.



To collect the gold diffraction: D1, wide only, on gold position.



To check the sample quality: D1, wide only, on sample position



5-II: Powder diffraction setup with MARCCD.

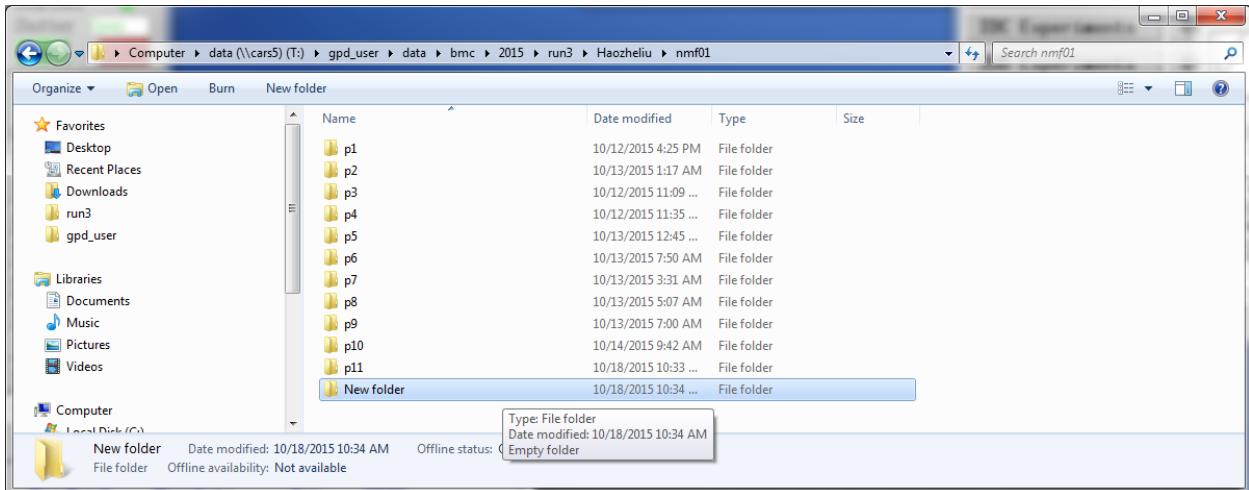
Powder diffraction is simpler than single crystal diffraction because it (usually) doesn't require DAC rotation.

Similar to single crystal diffraction, first bring your sample to the X-ray crosshair that you just defined. Record the sample position on your logbook.

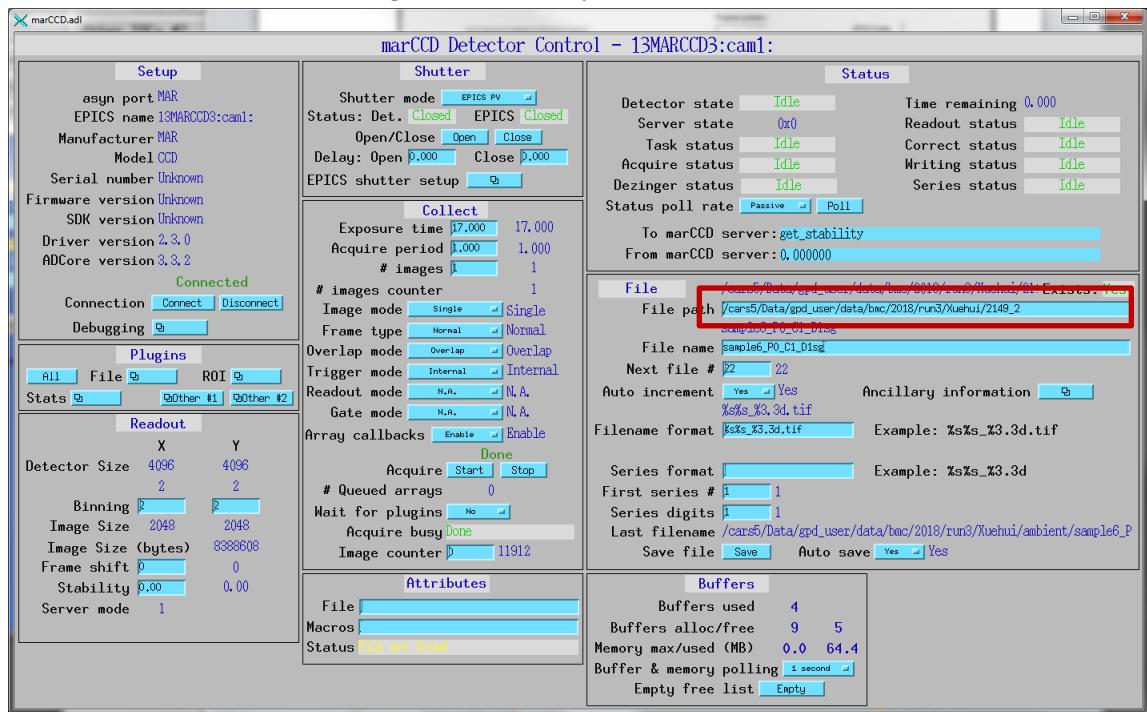


Then make a new folder: Make a new folder for the data in

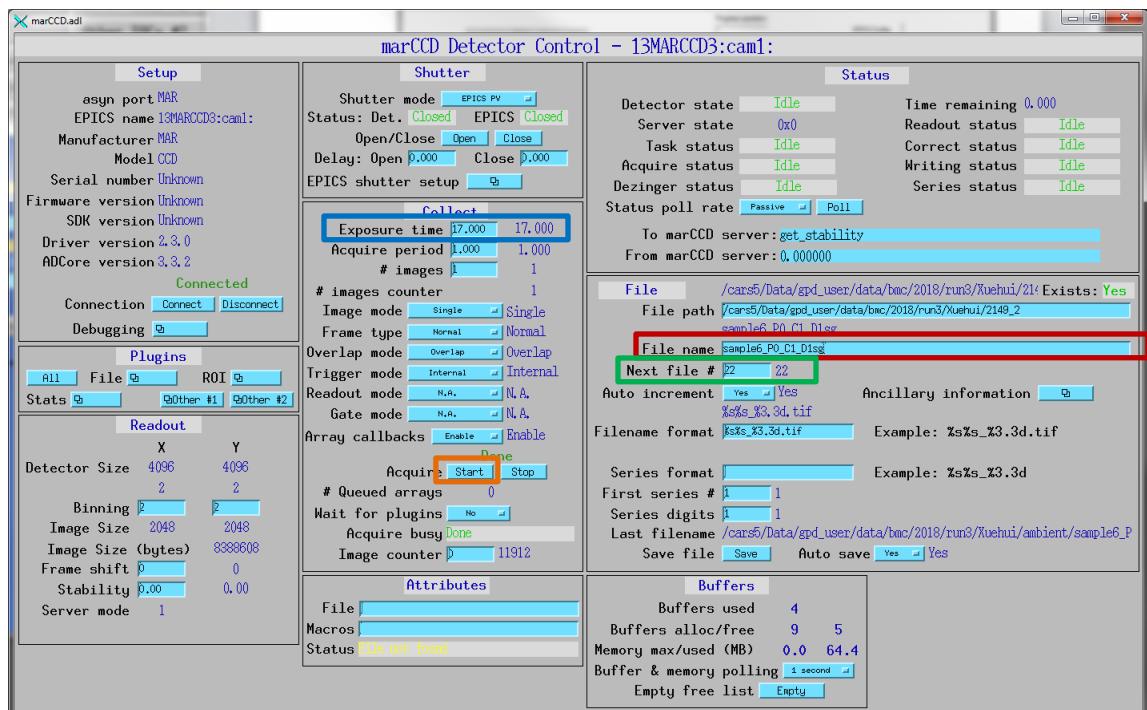
T:\gpd_user\data\bmc\20XX\runY\Your_folder



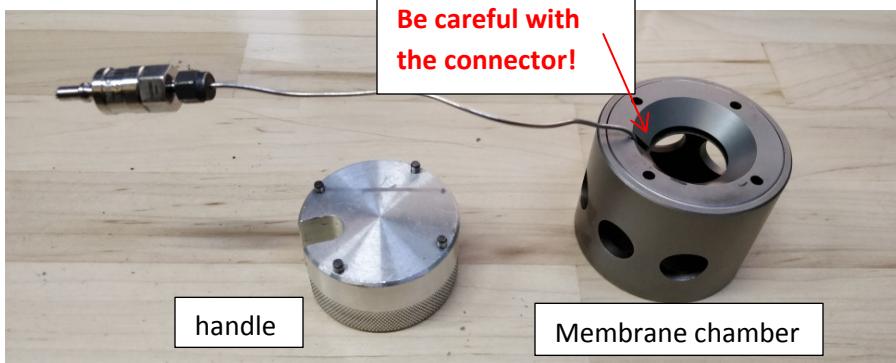
In the marccd.adl window, change the folder to your new folder.



Rotate kphi to 90 degrees. Change **file name**, **file number**. Set **Exposure time**. Press **Start**.



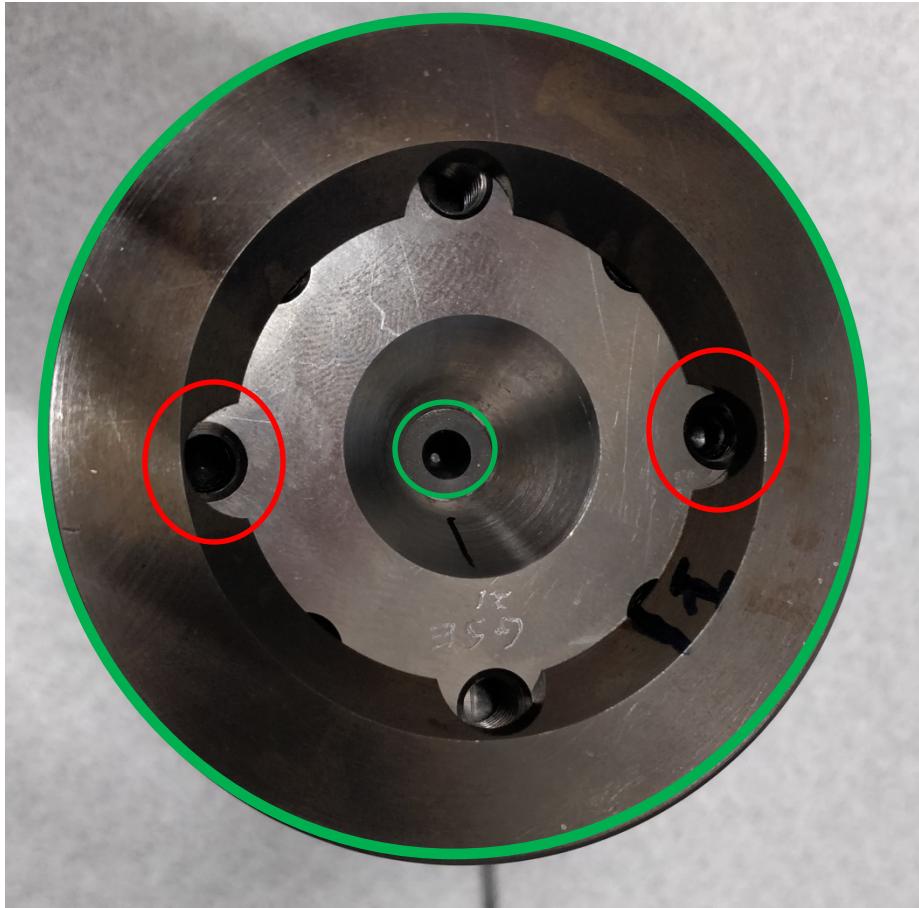
Membrane setup



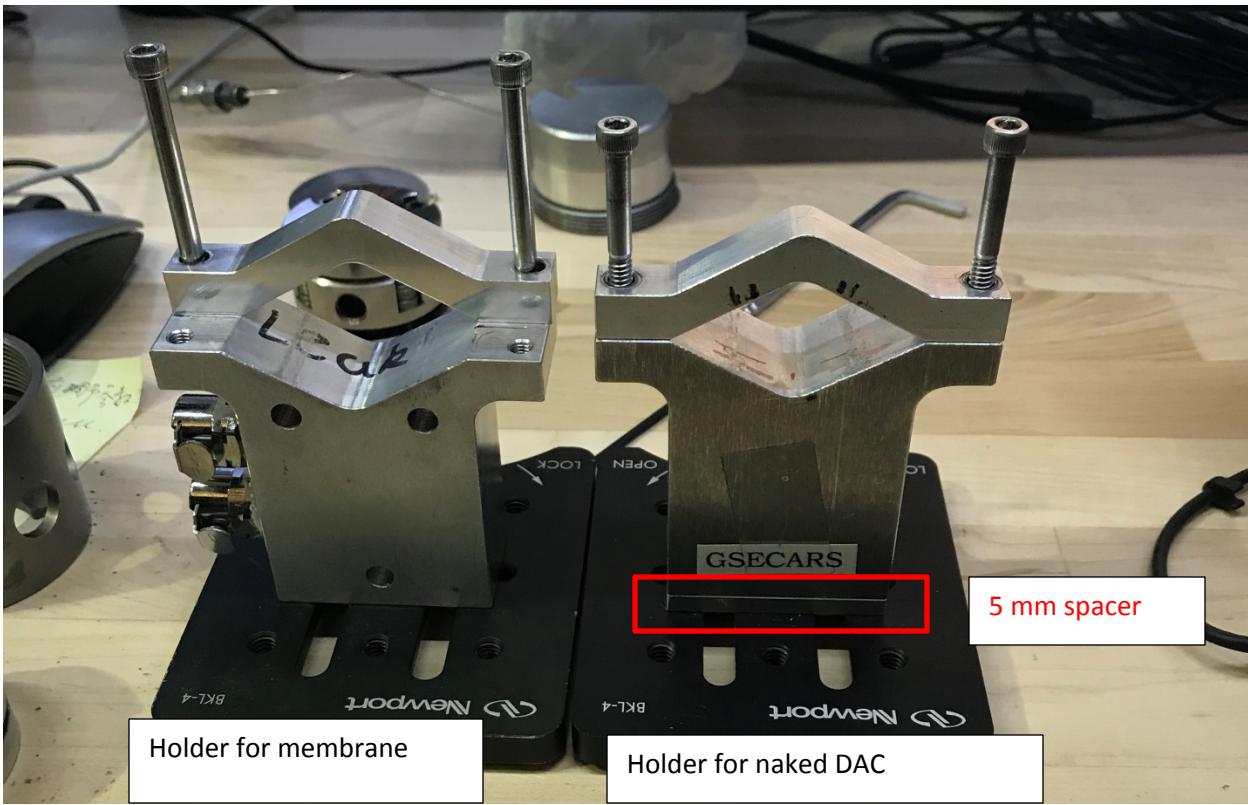
First, remember to use the handle (left) to tighten the membrane chamber (right). **Do not exert force on the connector that connects the pipe and the membrane!!!** It is very fragile! **Make sure your culet is as centered to the membrane chamber as possible**, or you will not be able to see the sample in the microscope.



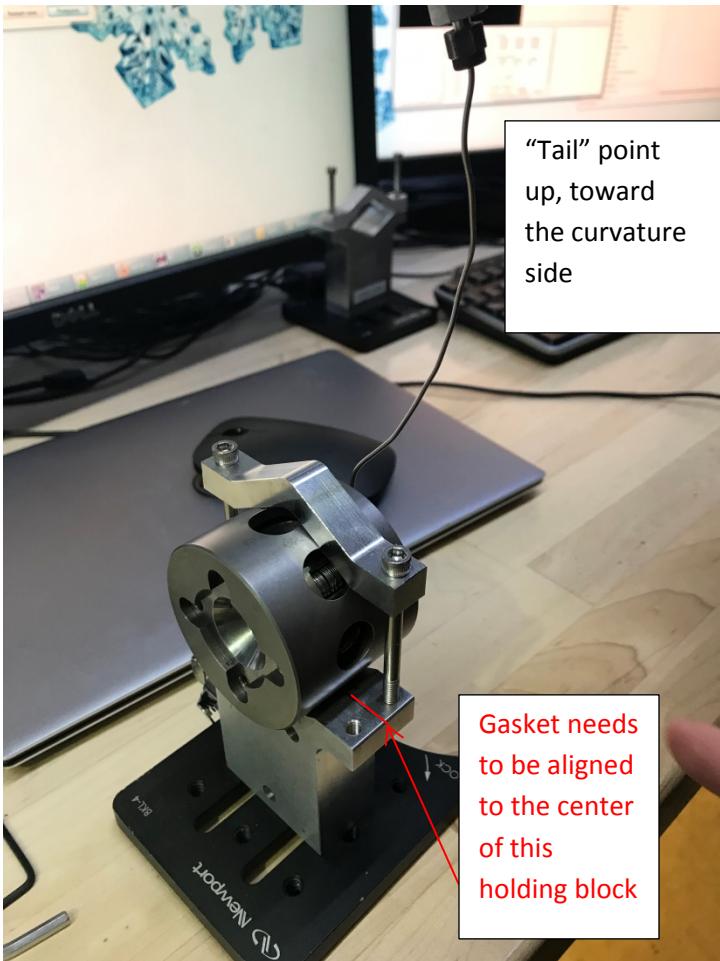
Use the handle to tighten and loose the membrane chamber.



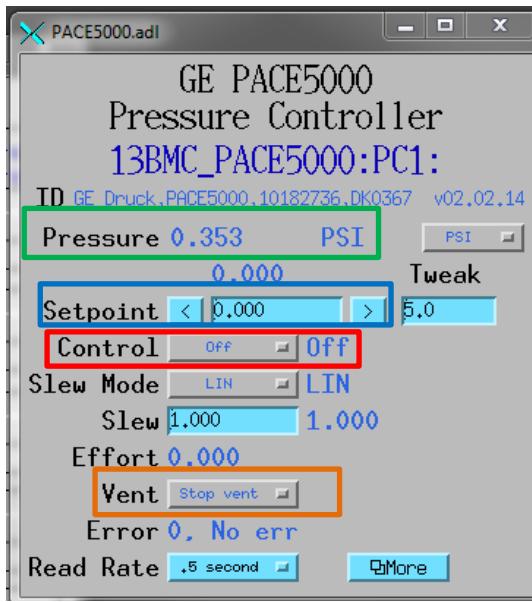
- I: **Tighten the set screws before you tighten the membrane chamber. The set screws must be accessible when the membrane chamber is tight.** The set screw secures the pressure in the DAC. If the set screw is loose, the pressure in the DAC might jump up by 40 GPa once you tighten the membrane chamber. **You need to loosen the set screw before you start pumping gas into the membrane.**
- II: **The diamond culet (small green circle) needs to be concentric with the membrane chamber (large green circle).** As concentric as possible.



We have two kinds of sample holders. The holder with a spacer is for naked DAC (not in membrane chamber). The holder without a spacer is for DAC in the membrane chamber.



1. Put the pipe "tail" toward the curved side of the holder
2. The "tail" should point up
3. The gasket should be located at the center of the holding block

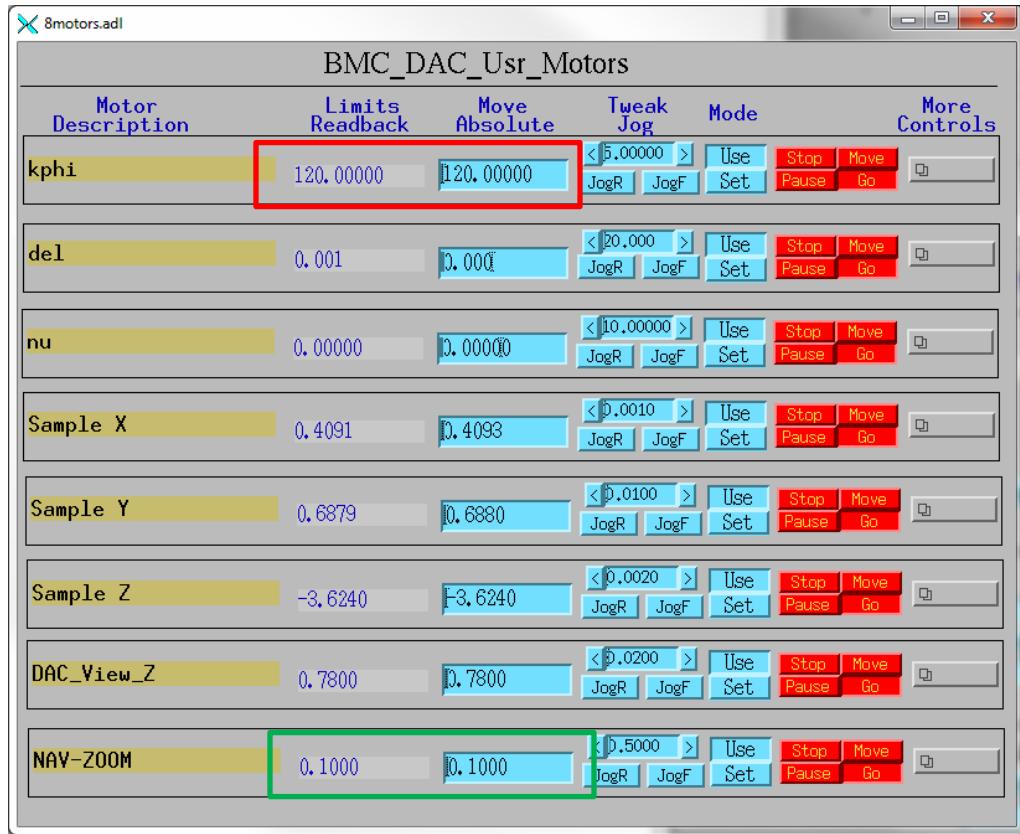


Turn **control** on to start compression. **Change setpoint** to increase **membrane pressure**, and measure the gold/ruby pressure in the DAC. When reach the target DAC pressure, decrease **setpoint pressure** by 10 Psi to keep the DAC pressure stable during single crystal diffraction.

Decompression: **change Setpoint** to 0, until the **membrane pressure** declines below 1, then turn the **control** off and **start vent**, wait 2 seconds then change to **stop vent**. Then go into the hutch to disconnect the membrane pipe.

Ruby fluorescence pressure determination

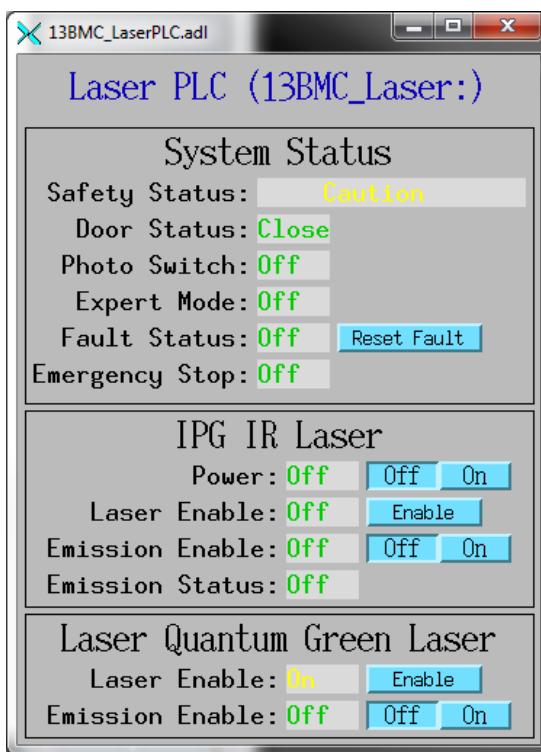
1. For ruby pressure determination, the kphi needs to be at **120 degs**. NAV-ZOOM needs to be at **0.1**.



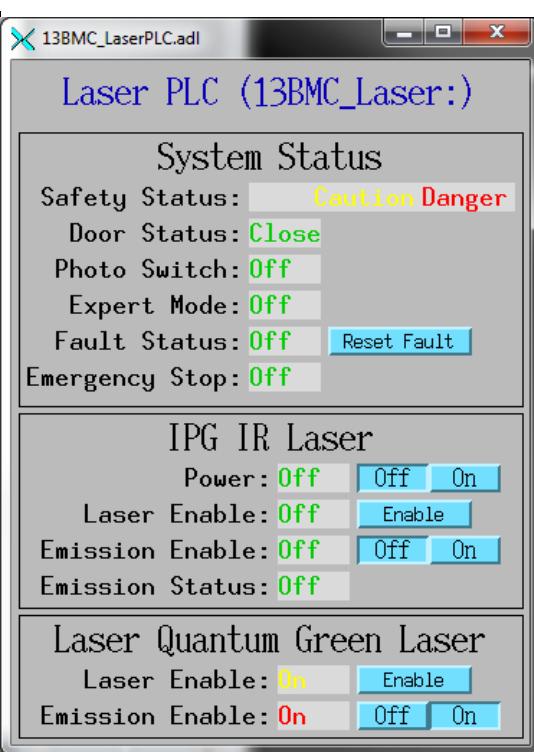
Now you can see the sample chamber in the camera image. Bring the ruby to the circle.



Turn on the laser while the hutch door is closed.

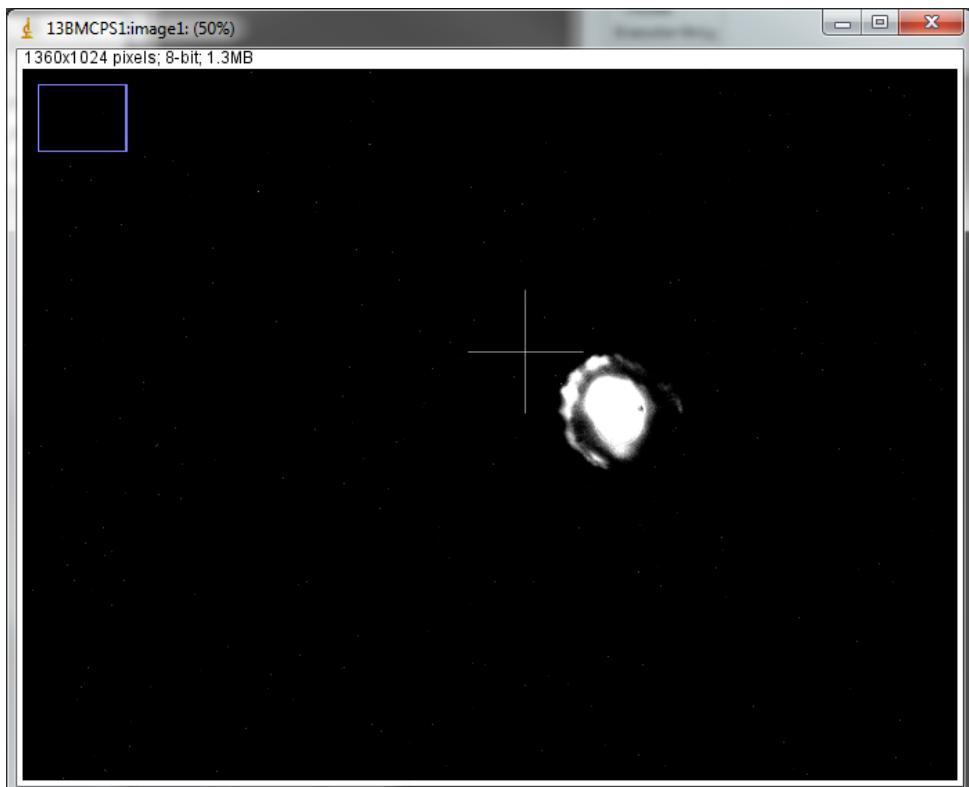


Laser off



Laser on

Now the camera image should look like this:

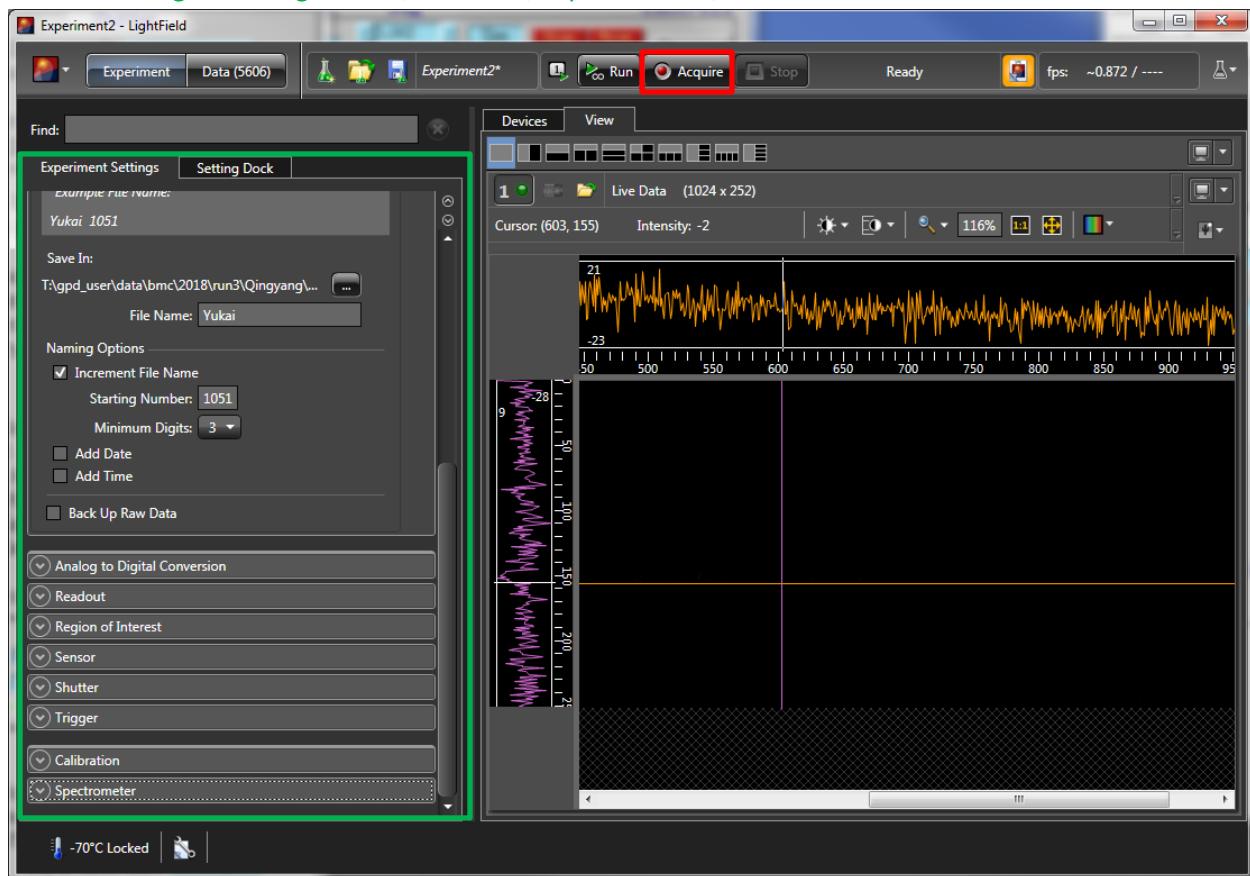


Turn off the hutch light. The switch (white in color) is behind the Crate computer box.

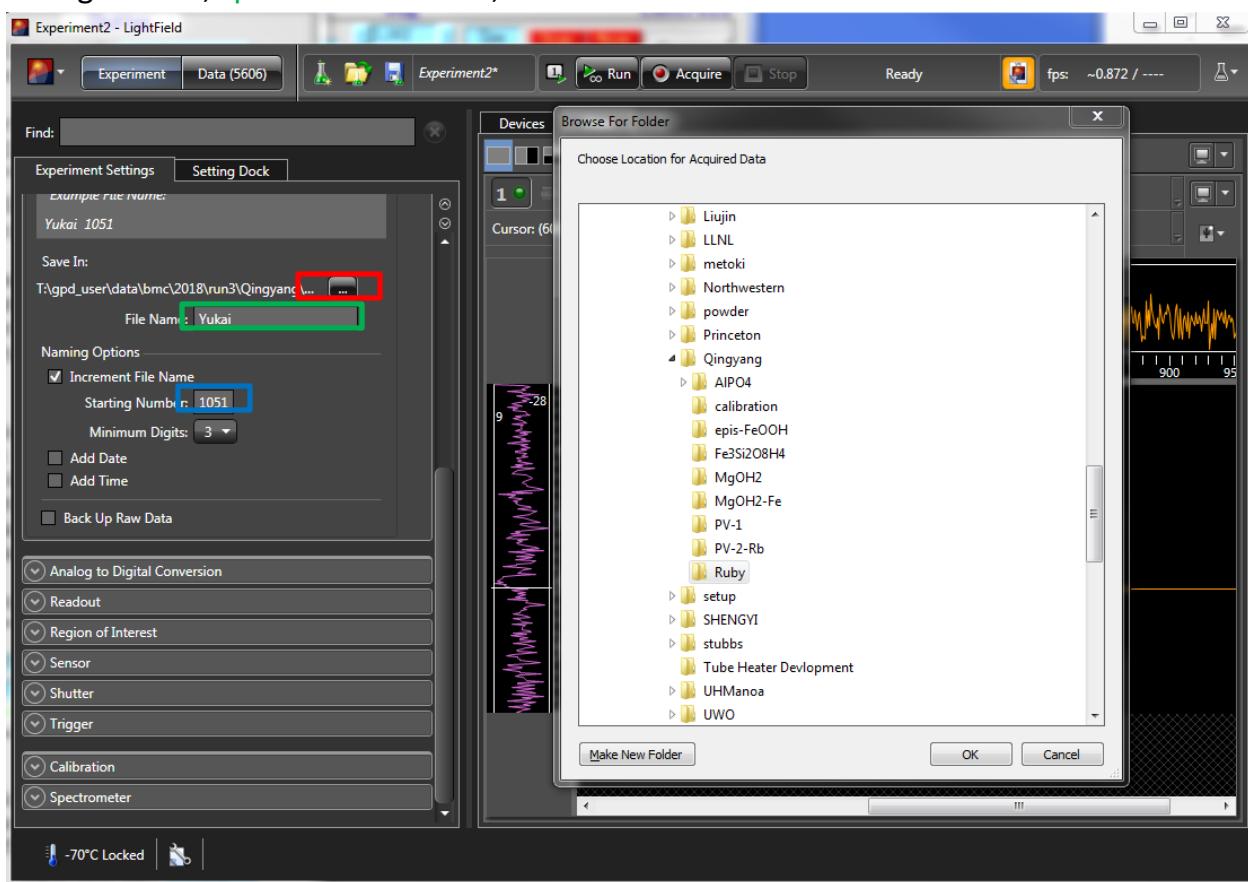


Go to the Lightfield program. The program looks like this:

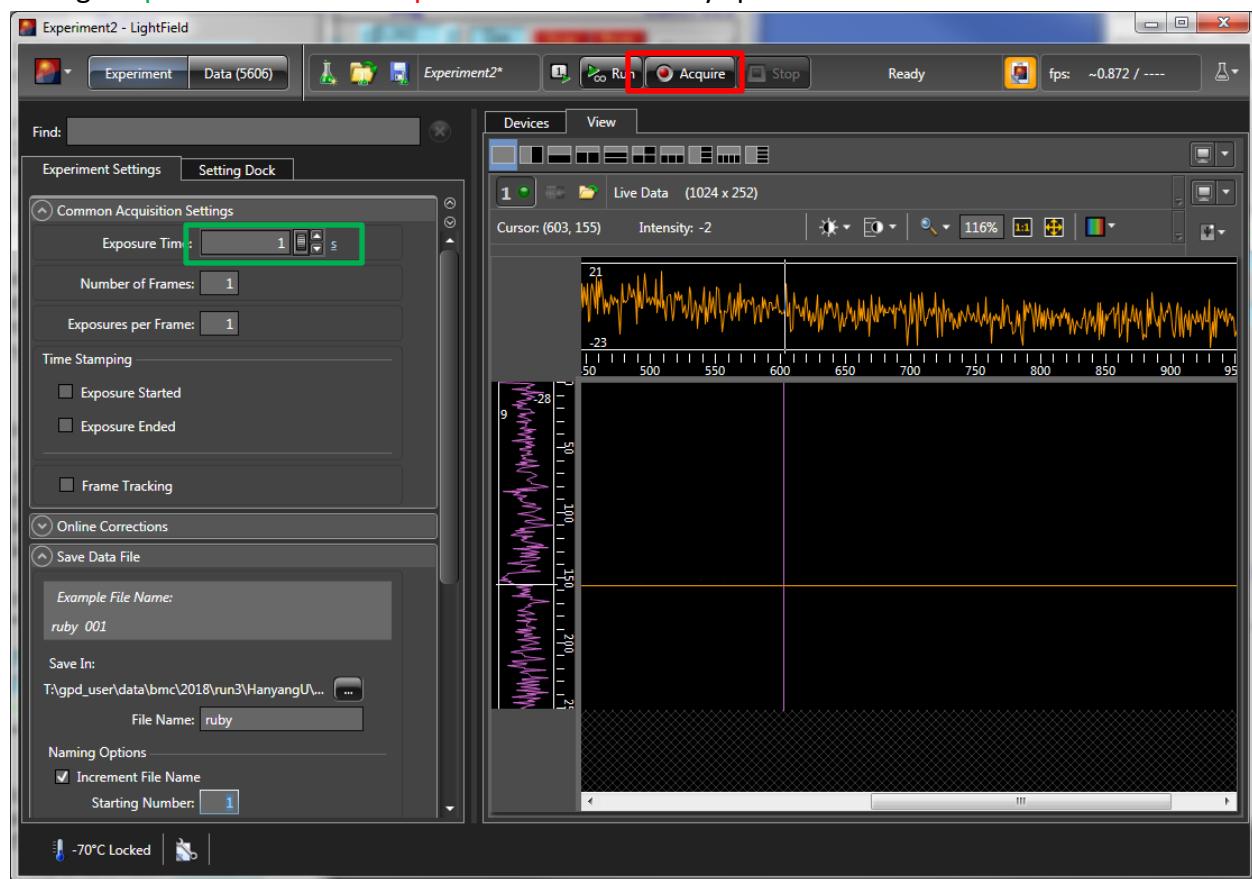
1. Acquire button: collect ruby spectrum
2. Function region: change folder, file name, exposure time, etc.



Change **folder**, **spectrum file name**, and **file number**.



Change **exposure time**. Use **Acquire** to collect the ruby spectrum.



Once you finish collecting the ruby spectrum, go to T-Rax software. **Load your spectrum, constrain the fitting range by changing the shape of the white rectangle, and press “Fit Ruby Peaks” to constrain the pressure. Record the reference ruby peak wavelength and your current ruby peak wavelength if you need to re-evaluate the pressure in the future.**

